Adhesive Compendium for Conservation

Written and Compiled by
Jane L. Down

Canadian Conservation Institute
Institut canadien de conservation
Canada

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Jane L. Down

Preface

This book has grown out of a few ideas, suggestions and needs over the years. For instance, the Canadian Conservation Institute has received numerous requests for its unpublished adhesive workshop manuals, which were only available to workshop participants. Several people have suggested that these manuals be published as a book. Also, many others have suggested that I write an adhesive book to capture my knowledge before I retire. And, too, I noticed that in answering the hundreds of adhesive-related questions over the years, I relied on many sources, not just one: my own research, published research by others, industrial polymer and adhesive handbooks, conservation handbooks such as Horie's *Materials for Conservation*, manufacturers' data sheets, material safety data sheets and online sources like CAMEO (Conservation & Art Material Encyclopedia Online). It has always been my wish to bring together much of this important information on adhesives into a single source for conservation use. Although I have tried to be as comprehensive as possible with the adhesives most used in conservation, it became obvious that, with a book this size, I could not be all encompassing. For instance, the book emphasizes adhesives more than consolidants because consolidation is complex and beyond the scope of this book to discuss in more than the basic way that it is mentioned throughout. Also, it had to be decided which adhesives could be included and which ones could not, so certain non-recommended, little-used adhesives are only briefly mentioned.

The book is organized with the theoretical information first. This gives information on liquid and solid adhesive properties, bonding and consolidation. This is followed by technical and aging information on each adhesive class: adhesive facts, characteristics and handling properties, and reviews of relevant aging studies. The book is completed by a chapter devoted to discussing how adhesives are chosen for each conservation specialty. This last chapter was written by colleagues who are experts in their fields, and I am indebted to them for agreeing to contribute their knowledge to this project.

The book is intended for practicing conservators or conservation students. If the reader wants general or theoretical information on adhesion and bonding, go to the first five chapters; specific property or aging information (e.g. solubility, aging studies, chemistry, characteristics) on an adhesive or class of adhesives, go to Chapter 6; or to know what to consider when choosing an adhesive for a specific treatment and some suggestions of frequently used products, go to Chapter 7.

This book is my legacy to conservation. It is my sincere wish that it is useful and helps conservators make wise adhesive choices in their work to preserve our cultural heritage for future generations.

Jane L. Down
Senior Conservation Scientist, Canadian Conservation Institute

About the Authors

Writer and Compiler

Jane L. Down graduated from Queen's University (Kingston, Ontario) in 1973 with an Honours Bachelor of Science in chemistry and mathematics. She joined the Canadian Conservation Institute (CCI) in 1978 and, as a Senior Conservation Scientist, was responsible for all adhesive research carried out at the Institute and for answering the numerous adhesive-related questions that CCI receives worldwide each year. Jane published many papers, presented her work at national and international conferences, supervised interns, organized and taught many adhesive workshops, including ***Adhesives for Textile and Leather Conservation: Research and Application*** delivered four times at CCI (1999–2001) and ***Adhesives Today: Exploring Current Adhesive Options and Application Techniques*** (2002) given at the Victoria and Albert Museum, and organized and chaired CCI symposia, including ***Symposium 2011 - Adhesives and Consolidants for Conservation: Research and Applications***. She carried out research on epoxy resins, poly(vinyl acetates), acrylics, vinyl acetate/ethylene copolymers and various tapes and heat-set tissues. In 2002, Jane received the Preparator's Award from the Society of Vertebrate Paleontology to investigate cyanoacrylate adhesives in the presence and absence of fossils. In 2002 and 2003, she was a co-recipient of two Department of Canadian Heritage Deputy Minister Awards for outstanding contributions. She was also a recipient of the CCI Director General's Award for outstanding contribution to Symposium 2011. She was an accredited member of the Canadian Association of Professional Conservators, a Fellow of the International Institute for Conservation of Historic and Artistic Works, and a member of the Canadian Association for Conservation. Jane retired from CCI in January 2014.

Chapter 5 and 7 Authors

Samantha Alderson is a Conservator in the Anthropology Division of the American Museum of Natural History, working with the museum's archaeological and ethnographic collections. In addition, she is a lecturer at the Conservation Center of the Institute of Fine Arts, New York University, teaching advanced courses in objects conservation. She holds a Bachelor of Arts from St. John's College (Annapolis, Maryland) and a combined Master of Arts degree in the history of art and archaeology, plus an Advanced Certificate in conservation, from the Conservation Center of the Institute of Fine Arts, New York University (New York). Her research interests include adhesives and consolidants in conservation, and the technology and conservation of Meso-American ceramics.

Wendy Baker has many years of experience in the conservation of fine and decorative art, and heritage interiors. She obtained a Master of Art Conservation degree in fine art conservation from Queen's University (Kingston, Ontario) (1977–79), after which she set up a private studio that she maintained until 2002. During her many years of private practice, Wendy undertook the conservation of paintings on multiple supports including canvas, wood, plaster and glass. Her experience in the conservation of heritage interiors extends from 1st century-AD adobe wall fragments (Meröe, Sudan) to gilded, painted and ornamented cathedral interiors. Wendy has been employed at CCI since 2002, where she is a Senior Paintings Conservator in the Fine Art, Furniture and Heritage Interiors Division. Her time is now divided among a number of duties, including treatment, training, workshop delivery and client consultation.

Lynn Curry is the Head Conservator of Books at Library and Archives Canada. She studied history at Carleton University (Ottawa, Ontario) and graduated from the Museum Technology program at Algonquin College (Ottawa, Ontario). She began her career at Library and Archives in 1984 in a 5-year in-house archival conservation training program. She has studied historical book structures, history of the book and descriptive terminology for more than 25 years. She has taught history of book structures and book conservation at the Master of Art Conservation program at Queen's University (Kingston, Ontario).

Amy Davidson is a vertebrate fossil preparator and has worked as Senior Principal Preparator for the Paleontology Division of the American Museum of Natural History since 1993. She specializes in small dinosaurs and mammals in difficult matrices, including loose sandstones, exploded mudstones and very hard volcaniclastic rock. She has collaborated with conservators since 1994 to adapt conservation materials and methods for fossil preparation, resulting in numerous presentations, workshops and publications. Prior to her current position, she apprenticed for 3 years in fossil preparation at the Museum of Comparative Zoology, Harvard University. She has a background in sculpture and model-making. Amy holds a Bachelor of Arts from Oberlin College (Oberlin, Ohio).

Carole Dignard graduated from the University of Ottawa (Ontario) with a Bachelor of Science in physics and Italian (1981) and an Honours Bachelor of Arts in classical studies (1983), and from Queen's University (Kingston, Ontario) with a Master of Art Conservation, specializing in objects (1986). She joined CCI in 1988 and is currently a Senior

Objects Conservator. She has published on various treatment case studies (Northwest coast basket, birchbark quilled box, kayak, fur-trimmed beaded cape), laser-cleaning, ultrasonic misting and mount-making. She was one of the instructors in CCI's professional development workshop ***Adhesives for Textiles and Leather Conservation: Research and Application***, delivered four times at CCI (1999–2001). She was Program Chair for CCI's ***Symposium 2007 - Preserving Aboriginal Heritage: Technical and Traditional Approaches***, was Coordinator of the International Council of Museums – Committee for Conservation Working Group on Ethnographic Collections (2005–2011), and is currently Assistant Coordinator of the International Council of Museums – Committee for Conservation Working Group on Leather and Related Materials (2014–2017). She is a Fellow of the International Institute for Conservation of Historic and Artistic Works and the American Institute for Conservation of Historic and Artistic Works, and an Honorary Member of the Canadian Association for Conservation.

Julia Fenn was born in South Africa and obtained a degree in archaeology at the University of Cape Town (South Africa). She subsequently qualified in Conservation (archaeological and ethnographic) at the Institute of Archaeology (London, United Kingdom) and was employed for several years at the British Museum Research Laboratory. Following her marriage to a Canadian, she moved to Ontario where she has worked for National Historic Sites, coordinated the Conservation Technician training program at Fleming College in Peterborough, and taught in the Museum Studies program at the University of Toronto. In 2014, she retired from her long-standing position as Senior Ethnographic Conservator at the Royal Ontario Museum but is continuing to teach and research adhesives and conservation.

Tara Grant graduated from the Master of Art Conservation program at Queen's University (Kingston, Ontario) in 1985, following which she was an Andrew W. Mellon Fellow at the Kelsey Museum of Archaeology, University of Michigan (Ann Arbor, Michigan). In 1987, she began working with Parks Canada, Prairie and Northern Region (Winnipeg), and a year later joined CCI's Archaeology Laboratory. Since coming to CCI, Tara has specialized in wet organic materials recovered from Arctic sites and has been the on-site conservator for many excavations in Nunavut, including sites on Baffin, Devon, Bathurst, Somerset, Igloolik and Ellesmere islands. She is currently working on developing treatments for cedar basketry as well as becoming specialized in the conservation of ceramics and glass. From 2011 to 2014, Tara was the Coordinator for the Wet Organic Archaeological Materials Working Group of the International Council of Museums – Committee for Conservation.

David W. Grattan, born in the United Kingdom, earned a Bachelor of Science from the University of London (United Kingdom) (1968) and a Doctor of Philosophy from the University of Keele (United Kingdom) (1973). A physical/polymer chemist, he began his career at the National Research Council Canada as a Research Fellow (1974), then as a Research Associate (1976). In 1978, he joined CCI as a Senior Conservation Scientist, where he became Manager of Conservation Research in 2005. His interests range from problems related to the conservation of wood, synthetic materials and paper to the preservation of Canada's Aboriginal tangible and intangible heritage. David is the author of numerous publications on conservation and has lectured extensively in Canada, Europe and Brazil. He was Chair of the Committee for Conservation of the International Council of Museums from 1996 to 2002. David retired in 2011 but continues to write and lecture on a number of conservation topics. He is a Fellow of the International Institute for Conservation of Historic and Artistic Works.

Sherry Guild graduated from the Art Conservation Techniques program at Sir Sandford Fleming College, now Fleming College (Peterborough, Ontario). In 1984, she started as a conservator at CCI in the Works on Paper Laboratory, specializing in the conservation of works of art on paper. She retired from CCI as a Senior Paper Conservator in 2015.

Gregory Hill is Senior Conservator of Archival and Photographic Records at CCI. Prior to joining CCI, he spent 18 years at Library and Archives Canada as a conservator, conservation manager and preservation advisor. He has presented workshops, spoken and published on a range of conservation activities, and is a former President of the Canadian Association of Professional Conservators.

Irene F. Karsten has a Master of Science (1998) and a Doctor of Philosophy (2003) in human ecology, with a specialization in textile conservation science, from the University of Alberta (Edmonton, Alberta), as well as a Diploma in art conservation techniques (1994) from Fleming College (Peterborough, Ontario). She was the Conservator for the Clothing and Textiles Collection at the University of Alberta from 2004 to 2009, and is currently a Preservation Development Advisor at CCI.

Stephen P. Koob received a Master of Arts (1976) in classical archaeology from Indiana University (Bloomington, Indiana) and a Bachelor of Science (1980) in archaeological conservation and materials science from the Institute of Archaeology, University of London (United Kingdom). He spent 5 ½ years as conservator of the Agora Excavations with the American School of Classical Studies in Athens, Greece. From 1986 to 1998, he worked as conservator, specializing in ceramics and glass, at the Freer Gallery of Art and Arthur M. Sackler Gallery, Smithsonian Institution, Washington, D.C. In 1998, he went to the Corning Museum of Glass, where he is now Chief Conservator. He also continues teaching archaeological conservation at the site of Samothrace in northern Greece, and has numerous conservation interns at Corning. He is the author of *Conservation and Care of Glass Objects*.

Lisa Kronthal Elkin joined the American Museum of Natural History in 1994 where she is currently Chief Registrar and Director of Conservation. Prior to this position, she was a Conservator in the Museum's Division of Anthropology and then Director of Conservation for the Natural Science Collections. In recent years, her focus has been on planning strategically for the preservation of the scientific collections, which involves surveying and evaluating risks to collections, planning for collections upgrades, and setting procedures and policies for collections and conservation management. She holds a Bachelor of Arts from the University of Rochester (New York) and a Master of Arts with a Certificate of Advanced Studies in conservation from Buffalo State (New York).

Anne F. Maheux received a Master of Art Conservation from Queen's University (Kingston, Ontario) and a Certificate in the conservation of works on paper at the Center for Conservation and Technical Studies, Harvard University Art Museums (Cambridge, Massachussetts). She is a recipient of the American Academy in Rome Prize in Historic Preservation and Conservation. She was Conservator of Prints and Drawings at the National Gallery of Canada for over 25 years, and is now Head Conservator of Works on Paper, Maps and Manuscripts at Library and Archives Canada. Her scholarly interests include 19th century pastel painting, particularly the work of Edgar Degas. She also enjoys the challenge of treating and mounting oversize works on paper.

Janet Mason is a conservator in the Objects Laboratory at CCI where she consults on and undertakes treatment of artifacts, and develops and delivers workshops on the care of organic materials, including CCI's professional development workshop ***Adhesives for Textile and Leather Conservation: Research and Application***. Her association with CCI began in 1979. She graduated from the Art Conservation program at Fleming College (Peterborough, Ontario) in 1980. From 1986 to 1988, Janet worked primarily with the Polynesian Collection at the Bishop Museum, Hawaii, as a Mellon Fellow.

Stefan Michalski is a Senior Conservation Scientist at CCI. He holds an Honours Bachelor of Science (1972) in physics and mathematics from Queen's University (Kingston, Ontario). Since 1979, Stefan has developed advice for both preservation and treatments. He was lead author for the humidity and temperature specifications in the "Museums, Galleries, Archives and Libraries" chapter of the *ASHRAE Applications Handbook* (editions 1999 through to 2011), and author of the CCI Technical Bulletin No. 23, *Guidelines for Humidity and Temperature for Canadian Archives*. He developed the CCI Light Damage Calculator, first as a slide rule and now as a web page. He has written reviews of light damage, the mechanics of paintings, the mechanics of gilding, physical models of suction table treatments, varnish removal by solvents, and consolidation of porous materials, especially powdery paints. In 2005, he received the Harley J. McKee Award from the Association for Preservation Technology International, given to "individuals who have made outstanding contributions to the field of preservation technology." He is currently preparing a risk management manual and collaborating with Jim Druzik of the Getty Conservation Institute on a book on the museum environment.

Ruth E. Norton, Fellow of the International Institute for Conservation of Historic and Artistic Works and Fellow of the American Institute for Conservation of Historic and Artistic Works, earned her Master of Science in Art Conservation from the Winterthur Museum/University of Delaware (Newark, Delaware) in 1978. For 4 years, she practiced and provided conservation training at the Bishop Museum (Honolulu, Hawaii) and at the National Museum of the Philippines (Manila). From 1982 to 1988, she taught Objects Conservation at the University of Canberra, Australia. Her work with Pacific and Asian material continued under UNESCO and Australian government consultancies and through workshops addressing practical approaches to conservation problems in the tropics. Following a year of field conservation in Pella, Jordan, she returned to Sydney, Australia, as Senior Conservator of the Museum of Applied Arts and Sciences (Powerhouse Museum). Between 1997 and 2001, she divided her time between New York State as guest lecturer in the Art Conservation Department, Buffalo State College, and the Netherlands as Deltaplan Project Manager, National Museum of Ethnology, Leiden. She is

now McCarter Chief Conservator at the Field Museum of Natural History in Chicago. She continues teaching workshops and conducting consultations in conservation and collection care. Her published books include *Practical Manual for the Storage and Display of Textiles for Museums in Southeast Asia*; *Bibliography of Works on the Conservation of Ethnographic Materials* (with S. Walston); and *The Conservation of Artifacts Made From Plant Materials* (with M.L. Florian and D.P. Kronkright).

Geneviève Samson is a Senior Book Conservator at Library and Archives Canada. She graduated with a Bachelor of Arts in art history from McGill University (Montréal, Quebec) in 1980 and a Master of Arts in art history from Université de Montréal (Quebec) in 1985. She also graduated from the Museum Technology program at Algonquin College (Ottawa, Ontario) in 1984. She began her career at Library and Archives Canada in 1985. For over 25 years, she has studied historical book structures, history of the book and descriptive terminology. She has also taught many workshops on preventive conservation, historical book structures and historical pigments to members of professional associations in Quebec and Ontario. She is currently collaborating on a catalogue raisonné of Books of Hours dating from the 15th and 16th centuries held in various institutions in Quebec (made possible by a Social Sciences and Humanities Research Council grant, 2014–2017).

Jan Vuori was Senior Textile Conservator at CCI where she has worked from 1981 to 2014. She graduated from Queen's University (Kingston, Ontario) with a Master of Art Conservation (1981) and pursued additional course work in textile science from North Carolina State University, College of Textiles (Raleigh, North Carolina). Within the context of conservation treatment, Jan published on a variety of subjects, including archaeological textiles, dyeing, pH measurement, digital inkjet printing, upholstery and pressure mounting. She was one of the instructors in CCI's professional development workshop ***Adhesives for Textiles and Leather Conservation: Research and Application***, and was an invited speaker at ***Adhesives Today: Exploring Current Adhesive Options and Application Techniques***, a professional development workshop for textile conservators organized in 2002 by the Victoria and Albert Museum together with the British Museum and CCI. She was accredited as a textile conservator by the Canadian Association of Professional Conservators and is a Professional Associate of the American Institute for Conservation of Historic and Artistic Works. Jan has served on the executive boards of the International Institute for Conservation – Canadian Group (now known as the Canadian Association for Conservation), the North American Textile Conservation Conference and the Canadian Association of Professional Conservators. She also served as an Assistant Coordinator of the International Council of Museums – Committee for Conservation Textile Working Group. Jan retired from CCI in 2014.

George Wheeler is the Director of Conservation in the Historic Preservation Program at Columbia University and Research Scholar at the Metropolitan Museum of Art. He is a leading expert in stone conservation and has published extensively in this area, including his book, *Alkoxysilanes and the Consolidation of Stone* (J.P. Getty Trust 2005). He received a Bachelor of Arts in art history and physics from Muhlenberg College (Allentown, Pennsylvania), a Master of Arts in art history from the City University of New York (New York), a Graduate Certificate in art conservation from New York University (New York), and a Doctor of Philosophy in chemistry, also from New York University. He is a Fellow of the American Academy in Rome, the American Institute for Conservation of Historic and Artistic Works, and the International Institute for Conservation of Historic and Artistic Works.

Donald C. Williams was a Senior Furniture Conservator at the Smithsonian Museum Conservation Institute. With over 40 years in the furniture and restoration trades, he trained as a fairly traditional craftsman in furniture restoration and foundry pattern-making and metal casting, then went on to receive his Bachelor of Arts in technology of artistic and historic artifacts from the University of Delaware (Newark, Delaware). He joined the Smithsonian in 1984. For many years he directed the Conservation Analytical Laboratory Master's Degree program in furniture conservation. He is the co-author of *Saving Stuff* (Simon & Schuster 2005). Donald is a past Chair of the American Institute for Conservation Wooden Artifacts Group. He currently serves on the United States Senate Curatorial Committee, the Executive Council of the Society of American Period Furniture Makers and the advisory boards of several museums. Donald retired from the Smithsonian in 2012.

Abbreviations

AMDES	aminopropylmethyldiethoxysilane
BA	butyl acrylate or *n*-butyl acrylate
BMA	butyl methacrylate or *n*-butyl methacrylate
BMP	2,6-di-t-butyl-4-methylphenol
c.	circa, approximately
CA	cellulose acetate
CCE	coefficient of cubical expansion
CE	cellulose ether
CED	cohesive energy density
CLE	coefficient of linear expansion
CMC	sodium carboxymethylcellulose
CN	cellulose nitrate
CTE	coefficient of thermal expansion
DBP	dibutyl phthalate
DGEBA	diglycidyl ether of bisphenol A
DMF	dimethyl formamide
DMSO	dimethyl sulfoxide
DP	degree of polymerization
DS	degree of substitution
EA	ethyl acrylate
EC	ethylcellulose
EHEC	ethylhydroxyethylcellulose
EMA	ethyl methacrylate
EO	ethylene oxide
EP	epoxy
EVA	ethylene/vinyl acetate copolymer
g	gram
h	hour
HEC	hydroxyethylcellulose
HPC	hydroxypropylcellulose
HPMC	hydroxypropylmethylcellulose
IBMA	isobutyl methacrylate
IMS	industrial methylated spirits
L	litre
m	metre
MA	methyl acrylate
MB	methyl bromide
MC	methylcellulose
MEK	methyl ethyl ketone
mfr	manufacturer
MHEC	methylhydroxyethylcellulose
MHPC	methylhydroxypropylcellulose
MMA	methyl methacrylate
mmHg	millimetres of mercury
M_n	number-average molecular weight
M_w	weight-average molecular weight
MW	molecular weight
N	newton
NMP	*n*-methyl-2-pyrrolidone
OS-EHEC	organic solvent soluble EHEC
P	poise
Pa	pascal
PAM	polyacrylamide
PAT	photographic activity test
PBA	poly(butyl acrylate) or poly(*n*-butyl acrylate)
PBMA	poly(butyl methacrylate) or poly(*n*-butyl methacrylate)
PEA	poly(ethyl acrylate)
PEG	poly(ethylene glycol)
PEMA	poly(ethyl methacrylate)
PEOX	poly(2-ethyl-2-oxazoline)
PIBMA	poly(isobutyl methacrylate)
PMA	poly(methyl acrylate)
PMMA	poly(methyl methacrylate)
ppm	parts per million
PSA	pressure-sensitive adhesive
PUR	polyurethane
PVAC	poly(vinyl acetate)
PVAL	poly(vinyl alcohol)
PVB	poly(vinyl butyral)
PVC	poly(vinyl chloride)
RH	relative humidity
RI	refractive index
RSG	rabbit skin glue
RTV	room-temperature vulcanizate
s	second
SBR	styrene-butadiene rubber
SI	silicone plastic (not to be confused with Si, the element)
T	temperature
TEOS	tetraethoxysilane
THF	tetrahydrofuran
TFEMA	2,2,2-trifluorethyl methacrylate
T_g	glass transition temperature
TLV	threshold limit value
TLV-C	threshold limit value–ceiling limit
TLV-STEL	threshold limit value–short-term exposure limit
TLV-TWA	threshold limit value–time-weighted average
T_m	melting point
T_s	softening temperature

UN	unknown
UV	ultraviolet
VAC	vinyl acetate monomer
VADBM	vinyl acetate/di-butyl maleate copolymer
VAE	vinyl acetate/ethylene copolymer
WS-EHEC	water soluble EHEC
WSP	wheat starch paste
w/v	weight to volume
w/w	weight to weight

1 Introduction

by Jane L. Down, Canadian Conservation Institute

The more you know about adhesives and consolidants both practically and technically, the more likely you are to make the best choice in conservation treatments of your artifact or object. The first five chapters of this book aim to give an overview of the technical and theoretical aspects of adhesion and bonding.

Definitions

An *adhesive* is a substance capable of joining materials by surface attachment (adhesion) (ASTM D907-05, 2006). "Adhesive" is a general term that includes cements, glues, mucilages and pastes.

A *consolidant* is a product, sometimes composed of the same materials as an adhesive, that is introduced into a crumbling or deteriorated material to make up for the loss of naturally occurring binding media.

Selecting adhesives and consolidants for conservation

Adhesives and consolidants should only be used for conservation treatments after first considering alternative non-adhesive treatments. If, after careful consideration, it is resolved to use an adhesive or consolidant, then all pros and cons must be examined. Generally for conservation, an adhesive or consolidant should:

- be compatible with the substrate, which could be the object (artifact), any part of it, or a backing, lining, patch, etc.;
- be chemically and physically stable over time, for example, showing little or no change upon aging of its pH, colour, flexibility, strength, shrinkage, etc.;
- remain removable and allow future re-treatment;
- be minimally invasive (less is best);
- be strong enough to hold yet weak enough to let go before the substrate becomes damaged;
- be non-tacky when set so as not to pick up dust;
- be easy to apply;
- be inexpensive; and
- have a good shelf life.

No adhesive or consolidant will satisfy all of these demands at once, so choice is usually a compromise that will satisfy the most important properties. Careful examination and selection of the adhesive or consolidant is necessary, which could include testing and mock-ups. Each object and adhesive or consolidant treatment must be considered separately. Do not employ the same adhesive for all applications, but examine each as unique, requiring different and appropriate adhesives or consolidants. Not only is it important to understand the properties of the object in question (substrate), but it is also important to understand the properties of each adhesive or consolidant in order to make the right choice for use in a conservation treatment.

Bonding

Bonding, the union of two materials by an adhesive, is the attachment at the interface between the adhesive and the adherend (substrate) (Ebnesajjad 2008, p. 328; Shields 1984, p. 344; Cagle 1973, p. 14). Bonding usually requires wetting of the substrate surface by the adhesive and requires the adhesive to set in order to form an attachment that prevents movement in the join. The bond should have the ability to adjust to stresses at the interface.

Bonding is achieved by one or more of the following mechanisms (SpecialChem), which are illustrated in **Figure 1**:

- *Adsorption,* which is the intermolecular contact of the adhesive and substrates due to Van der Waals forces.
- *Chemisorption,* which is the chemical bond formation between the adhesive and substrate.
- *Mechanical Interlocking,* which is a network of hardened tentacles of adhesive in the substrate formed when the adhesive seeps into pores, holes and crevices in the substrate, and sets.
- *Interdiffusion,* which is the interpenetration of chains of the adhesive and the substrate winding together around each other.
- *Electrostatic forces,* which are the attraction of positive and negative charges on the adhesive and substrate. There are positive and negative charges on the surface of all materials.

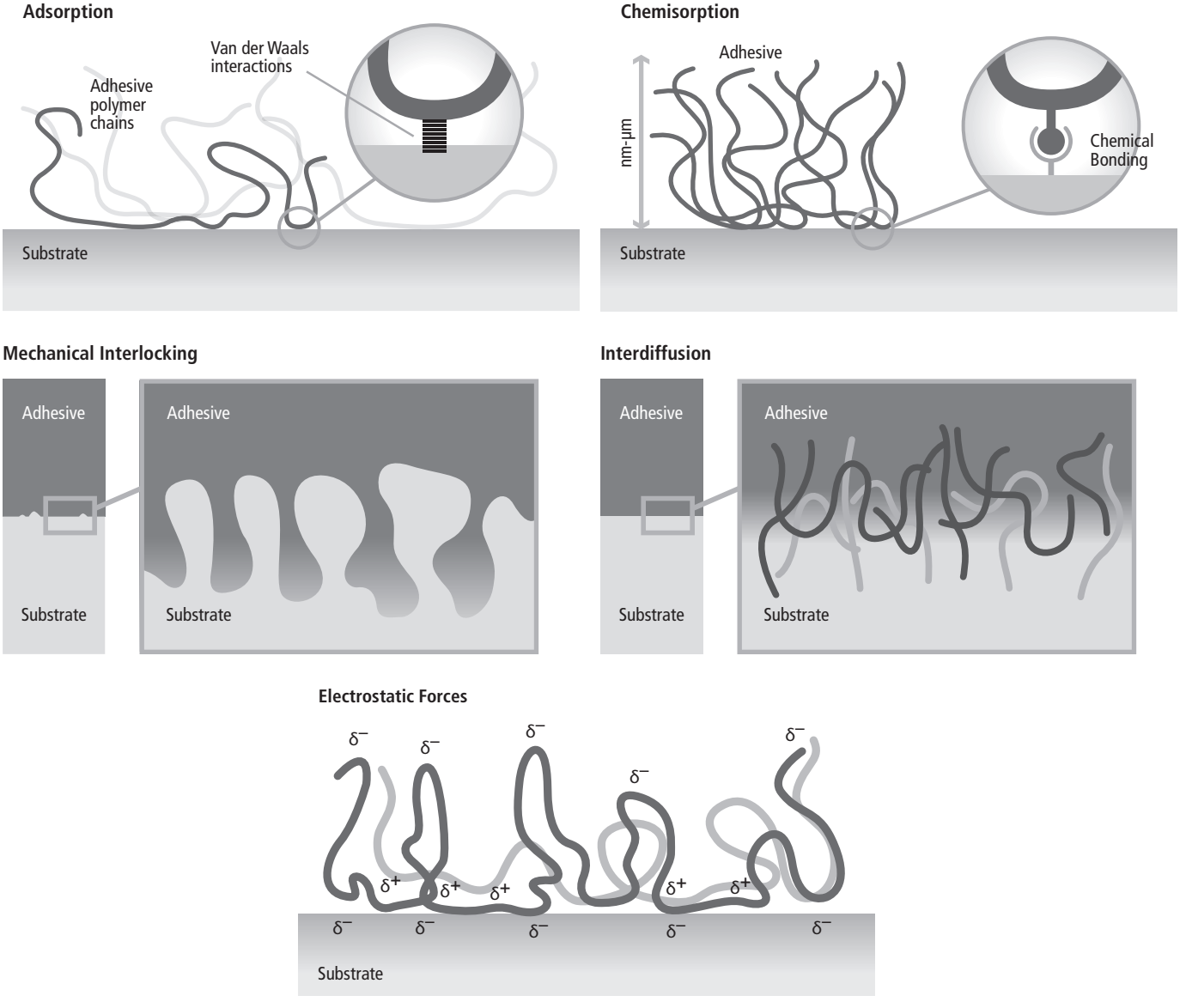


Figure 1. Examples of bonding attachment mechanisms (SpecialChem). Reprinted with permission from SpecialChem, www.specialchem4adhesives.com © SpecialChem S.A.

ADHESION AND COHESION

In bonding, there are two types of attraction that are important: adhesion and cohesion.

Adhesion is the state in which two surfaces (adhesive and substrate) are held together at an interface by adsorption, chemisorption, mechanical interlocking, interdiffusion and/or electrostatic forces (Ebnesajjad 2008, p. 323; Cagle 1973, p. 4).

Cohesion (internal strength of adhesive film) is the state in which particles of a material are held together by primary or secondary valences forces (Ebnesajjad 2008, p. 329; Cagle 1973, p. 20).

In order for the bond to be adequate, both the adhesion at the interface between the adhesive and substrate and the cohesion of the adhesive need to be strong.

Strong bonds can be formed if the adhesive is a *polymer* because polymer molecules are large enough to have numerous sites for intermolecular contact, chemical bonds, interpenetration of chains and/or positive

and negative charges, all necessary mechanisms for bonding. These sites also afford high self-attraction (cohesion) of the polymer that imparts strength to the bond (Cagle 1973, pp. 1–11).

TYPES OF BOND FAILURE

There are four types of bond failure relating to adhesion and cohesion (**Figure 2**).

Adhesion failure of the adhesive is the type of failure that would be desired in conservation if failure happens. The bond failure is totally at the interface between the adhesive and substrate and the substrate is not damaged.

Cohesion failure of the adhesive is another type of bond failure that would be somewhat desired in conservation if failure was to happen. This failure assures that the substrate is not damaged, only the internal part of the adhesive film. The adhesive is left on the substrate for later removal, if desired and possible. This points out one problem with this type of failure: residual adhesive on the substrate may not be desirable and may not be easily removable.

Adhesion and cohesion failure of the adhesive is most typical of bond failure. Some failure occurs at the adhesive and substrate interface and some occurs in the internal part of the adhesive film.

Cohesion failure of the substrate would not be desired in conservation. In this failure, the adhesive bond is so strong that the substrate fails before the adhesive.

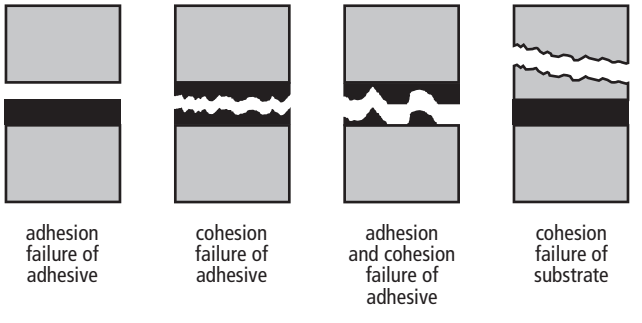


Figure 2. Adhesion and cohesion failures.

POLYMERS

The word *polymer* comes from the Greek words "polloi" meaning "many" and "meros" meaning "part" (Barber 2004, pp. 969, 1202). So, *polymer* means a molecule with "many parts". A polymer is formed from monomer units ("single" parts) that react (polymerize) to give linear or branched macro molecules (higher molecular weight molecules with monomeric repeated units). If the chain contains identical monomers, then the polymer is called a *homopolymer*. If the chain contains two or more different monomers, then the polymer is called a *copolymer*.

In a copolymer, monomers can occur randomly, alternately, in blocks or can be grafted onto the main structure (**Figure 3**) (Hoechst 1972, p. 36; Roff and Scott 1971, pp. 505–7). Block copolymers are formed when the repeated units in the main chain occur in blocks. Grafted copolymers have side chains from other monomers that are grafted onto the main chain.

Copolymers have different properties than mixtures of homopolymers containing the same type of originating monomers. For instance, the properties of copolymer (terpolymer) AAAABBCCC could be different than a mixture of homopolymers AAAA + BB + CCC.

Figure 3. Examples of different copolymers.

THERMOPLASTIC POLYMERS

In a thermoplastic polymer, the chains are not chemically bonded to one another, but may flow past one another in the molten state

or if dissolved in solvents. A thermoplastic polymer can be repeatedly melted by heat and hardened by cooling.

THERMOSETTING POLYMERS

In a thermosetting polymer, the chains are crosslinked and cannot flow past each other when heated or dissolved in a solvent, unless the molecular structure is broken down chemically. A thermosetting polymer undergoes a chemical reaction that leads to an infusible and insoluble product that cannot be softened by heat or dissolved in a solvent.

POLYMER MOLECULAR WEIGHT

The adhesive polymer is a mixture of similar molecules with a range or distribution of different molecular weights. Thus, the molecular weight of an adhesive polymer is really an average of all the polymer molecules and this average can be determined in different ways.

The *number-average molecular weight* (M_n) is the total weight of all the molecules divided by the total number of molecules (Equation 1) (Napper 1983, p. 12).

$$M_n = \frac{\sum n_i M_i}{\sum n_i} \quad (1)$$

Where n_i is the number of molecules of molecular weight M_i.

The *weight-average molecular weight* (M_w) is more complex and takes into account that bigger molecules account for more of the total mass than smaller molecules (Equation 2) (Napper 1983, p. 12).

$$M_w = \frac{\sum n_i M_i^2}{\sum n_i M_i} \quad (2)$$

M_n is the average M_i weighted according to number fractions, while M_w is weighted according to weight fractions. M_n is used when the number of molecules is important, such as in determining the number of end groups in a resin or hardener for mixing an epoxy resin adhesive (Horie 2010, p. 22). M_w is used when the weight of each molecule is important, such as in mechanical properties (Horie 2010, p. 22).

Figure 4 gives an example of polymer molecular weight distribution, M_n and M_w.

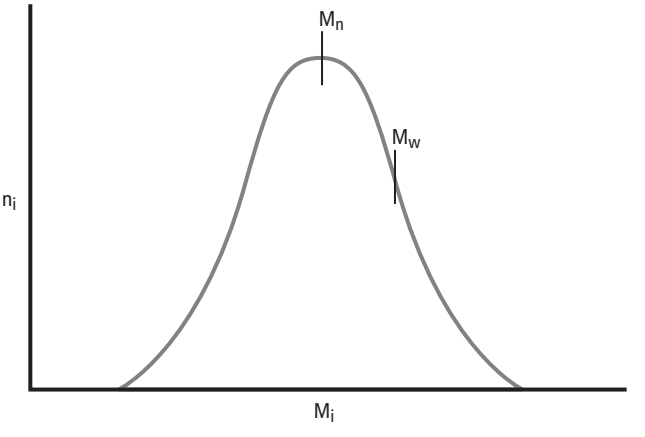


Figure 4. Example of a polymer molecular weight distribution. Adapted from Napper (1983, p. 12).

2 Liquid Adhesive Properties

by Jane L. Down, Canadian Conservation Institute

Adhesive composition

Adhesive formulations are composed not only of polymers, but may also contain solvents, diluents, stabilizers, thickeners, fillers, plasticizers, wetting agents, humectants, antioxidants, freeze-thaw stabilizers, emulsifiers and/or protective colloids. Each component is important and composition is tailored for many reasons, for example, to be compatible with the substrate, to reduce costs and/or to adjust the setting mechanism or setting time. To achieve good contact with the substrate, adhesives are often applied in solution or dispersion form (wet).

Solutions

A solution is a homogeneous mixture of two or more compounds in a single phase (Geller 2003, p. 1974; Cagle 1973, p. 55). Typically for adhesives, a solution contains an adhesive polymer(s) along with additives (not always) dissolved in a solvent(s). A polymer dissolves in a solvent if the polymer molecules are separated by solvent molecules due to dispersion or dipole forces and/or hydrogen bonding.

Dispersion forces, or Van der Waals forces, are forces of attraction that exist between molecules that have no permanent dipole and are caused from the movement of electrons around molecules. They are related to molecule surface area and are extremely weak forces (Blank and Stavroudis 1989).

Dipole forces (polar forces) are produced in unsymmetrical molecules when there is an imbalance in the electron distribution causing one area of the molecule to have a partial positive charge and another area to have a partial negative charge. The negative charge of one molecule is attracted to the positive charge of other adjacent molecules. These forces tend to be stronger than Van der Waals forces (Blank and Stavroudis 1989).

Hydrogen bonding occurs when a hydrogen atom is bonded to a strongly electronegative atom (e.g. oxygen, nitrogen, chlorine). A strong dipole force is set up and the hydrogen atom is attracted to other adjacent molecules (Blank and Stavroudis 1989).

Adhesive polymers dissolve best when attraction between polymer and solvent is greater than between polymer chains themselves. In this situation, the solvent is considered to be a *good* solvent: the polymer chains are extended and films formed after drying have interpenetrating chains and dense structure. When the attraction between polymer and solvent is less than between the polymer chains, this is considered a *poor* solvent: chains are contracted and the dried films form a porous structure (Hansen 1995; Hansen et al. 1991). Solvents therefore affect the properties of the resulting adhesive films. Large polymers are sometimes slow to dissolve because the solvent needs time to penetrate between the large molecules (Torraca 1990, p. 14).

Solubility parameters

There have been numerous attempts, notably by Hildebrand, Hansen and Teas, to explain solvent behaviour through the use of a variety of "solubility parameters" (Hansen 2007). These parameters, some of which are explained briefly below, attempt to quantify solvent behaviour as it relates to dissolving adhesives.

Hildebrand solubility parameter

Hildebrand devised a method to calculate total Van der Waals forces needed to separate solvent molecules using heat of vapourization (Burke 1984). He called this method cohesive energy density (CED) (Equation 1). His solubility parameter (δ in Equation 2) indicates the solvency power of a solvent, which is the amount of energy needed to completely remove a unit volume of molecules from their neighbours to infinite separation (an ideal gas).

$$CED = \frac{\Delta H - RT}{V_m} \quad (1)$$

$$\delta = (CED)^{1/2} \quad (2)$$

Where ΔH is heat of vapourization, R is the gas constant, T is temperature and V_m is molar volume.

According to this approach, polymers dissolve best in solvents with matching solubility parameters, and thus the term "like dissolves like". Such solutions

with similar solubility parameter values (δ) have low viscosity because the polymer molecules are evenly dispersed in the solvent. Hence, the resulting adhesive films are stronger. It is important to remember when applying from a mixture of solvents to ensure that the last solvent to evaporate is the best because then the polymer will be most spread out and produce the strongest film (Horie 2010, pp. 89–92). **Tables 1** and **2** give examples of Hildebrand solubility parameters for some solvents and polymers.

Two further facts about Hildebrand solubility parameters are worth noting:

- Sometimes the solubility parameter of a *polymer* or non-liquid material is referred to as the *cohesion parameter* rather than the solubility parameter because, in reality, δ cannot be determined from the heat of vapourization because the polymer would decompose before the heat of vapourization could be achieved (Burke 1984). The cohesion parameter of polymers is usually determined by swelling, cloud-point determinations or theoretical calculations (Burke 1984; Kok and Rudin 1983).
- If a suitable solvent (one with matching solubility parameter) for a polymer is not available or if the solvent is too toxic, solvents can be mixed by simple proportionality to obtain a match. For example, the cohesion parameter of poly(methyl methacrylate) is 18.6. To obtain a mixture match, solvents with higher and lower solubility parameters must be used and should be reasonably close to the area of solubility (Teas 1971). Toluene (δ = 18.2) and acetone (δ = 20.2) satisfy this condition. The calculation for a 10 mL volume of solution is as follows:

$$18.6 = \frac{a}{10} \times 18.2 + \frac{b}{10} \times 20.2 \quad (3)$$

$$a + b = 10 \quad (4)$$

Where a is the volume of toluene (mL) and b is the volume of acetone (mL) required.

Rearranging Equation 4 (a = 10 - b) and substituting a into Equation 3 yields:

$$18.6 = \frac{10 - b}{10} \times 18.2 + \frac{b}{10} \times 20.2$$

Thus $a = 10 - 2 = 8$

So for a 10 mL mixture of toluene and acetone that has a solubility parameter of 18.6, one would mix 8 mL of toluene and 2 mL of acetone.

HANSEN SOLUBILITY PARAMETERS

Hansen described an extension of the Hildebrand parameter to include polar and hydrogen-bonding intermolecular forces between molecules as well as Van der Waals dispersion forces. He proposed that all three forces occurred simultaneously and thus the Hildebrand solubility parameter was divided into three parts: dispersion forces (δ_d), polar forces (δ_p) and hydrogen bonding (δ_h) (Barton 1991). Hansen proposed the following equation to describe the relationship between the forces:

$$\delta_t^2 = \delta_d^2 + \delta_p^2 + \delta_h^2$$

TABLE 1. HILDEBRAND, HANSEN AND TEAS-FRACTIONAL SOLUBILITY PARAMETERS FOR VARIOUS SOLVENTS
(Hansen 2007; Barton 1991; Torraca 1990)

Solvents	Solubility parameters							
	Hildebrand δ ($MPa^{1/2}$)	Hansen δ ($MPa^{1/2}$)				Teas		
		δ_d	δ_p	δ_h	δ_t	f_d	f_p	f_h
turpentine	16.6	16.4	1.4	0.4	16.5	77	18	5
cyclohexane	16.8	16.8	0	0	16.8	94	2	4
toluene	18.2	18.0	1.4	2.0	18.2	80	7	13
ethyl acetate	18.6	15.8	5.3	7.2	18.1	51	18	31
methyl ethyl ketone	19.0	16.0	9.0	5.1	19.0	53	26	21
acetone	20.2	15.5	10.4	7.0	20.0	47	32	21
ethanol	26.0	15.8	8.8	19.4	26.5	26	18	46
methanol	29.6	15.1	12.3	22.3	29.6	30	22	48
water	47.9	15.6	16.0	42.3	47.8	18	28	54

δ_t is the total Hansen solubility parameter and should correspond to the Hildebrand parameter, although the two might not always be identical as they are determined by different methods (Barton 1991). **Tables 1** and **2** list Hansen solubility parameters for some solvents and polymers.

Hansen also described another parameter called the interaction radius (R) which, when plotted, describes a solubility sphere. Liquids that fall within the sphere for a given polymer would be good solvents for it (Burke 1984). The calculation and/or plotting of all four parameters is quite complex and not often done. There is a computer program [Hansen Solubility Parameters in Practice (HSPiP), available on the Hansen Solubility Parameters website] that plots these parameters three-dimensionally. However, sometimes

TABLE 2. HILDEBRAND, HANSEN AND TEAS-FRACTIONAL SOLUBILITY PARAMETERS (COHESION PARAMETERS) FOR VARIOUS POLYMERS
(Hansen 2007; Barton 1991; Torraca 1990)

Adhesives	Solubility parameters								
	Hildebrand δ (MPa$^{1/2}$)	Hansen δ (MPa$^{1/2}$)					Teas		
		δ_d	δ_p	δ_h	δ_t	R	f_d	f_p	f_h
ethylcellulose		17.90	4.30	3.90	—	5.90	47	28	25
		Ethocel HE10							
		20.10	6.90	5.90	—	9.90			
		Ethocel STD 20							
cellulose acetate		16.90	16.30	3.70	—	13.70			
cellulose nitrate		16.90	13.50	10.30	—	9.90	38	38	24
methyl acrylate/ethyl methacrylate		19.2	11.20	1.80	—	11.00			
		Paraloid B-72							
ethyl acrylate/methyl methacrylate		19.10	9.10	3.30	—	9.00			
		Paraloid B-82							
		19.40	11.20	4.40	—	10.50			
		Paraloid B-44							
poly(methyl methacrylate)	18.6	16.20	6.80	5.70	—	9.10	51	27	22
		Lucite 2044							
poly(ethyl acrylate)							55	32	13
							Lucite 2042		
poly(vinyl acetate)	19.2	20.93	11.27	9.66	—	13.71	51	27	22
		Mowilith 50					Mowilith 50		
		22.90	18.30	7.70	—	20.40			
		Vinylite AYAA							
poly(vinyl alcohol)		15.00	17.20	17.80	—	10.20			
ethylene/vinyl acetate		17.70	3.50	3.70	—	4.70			
		EVA 4055 SOL							
poly(vinyl butyral)		18.60	4.36	13.03	—	10.64	46	24	30
		Butvar B-76					Butvar B-76		
epoxy resin (before setting)		20.00	10.32	10.11	—	10.02	44	28	28
		Epikote 1001					Epikote 1001		

the parameters are plotted as a cross-section of the three-dimensional graph using only the hydrogen bonding and the polar parameters as axes and ignoring the dispersion parameter. Prediction of solubility using this method is said to be about 90% reliable (Burke 1984).

TEAS FRACTIONAL SOLUBILITY PARAMETERS

Plots of Hansen solubility parameters are desired so that prediction of solubility behaviour of polymers can be made without extensive testing. However, Hansen solubility parameters are difficult to plot on a two-dimensional graph. Teas took the data one step further and proposed fractional solubility contributions calculated from Hansen parameters that can be plotted on a triangle. The fractional contributions for dispersion, polar and hydrogen bonding are given by the following equations and represent the percentage contribution that each Hansen parameter contributes to the whole Hildebrand value (Burke 1984):

$$f_d = \frac{\delta_d}{\delta_d + \delta_p + \delta_h} \qquad f_p = \frac{\delta_p}{\delta_d + \delta_p + \delta_h}$$

$$f_h = \frac{\delta_h}{\delta_d + \delta_p + \delta_h}$$

$$f_d + f_p + f_h = 100$$

These Teas parameters are completely empirical and do not have the theoretical justification of Hansen solubility parameters, but they are easier to plot (Barton 1991, p. 112). **Tables 1** and **2** list Teas fractional solubility parameters for some solvents and polymers, and **Figure 1** illustrates a Teas chart. Solvents in the right lower corner of the triangle (e.g. alkanes) show almost exclusive intermolecular bonding due to dispersion forces, while in the lower left corner, the hydrogen bonding contribution becomes more prevalent (e.g. alcohols). As one moves up the triangle, solvents are more polar and show less hydrogen bonding (e.g. ketones, nitro compounds). One problem with this plot is that dispersion forces, which all molecules display, are overemphasized in comparison to the polar and hydrogen-bonding forces (Burke 1984). Some values on the Teas chart were adjusted by Teas to correspond to empirical evidence (Burke 1984). Also, non-random hydrogen bonding, lack of sufficient space around solute molecules, reaction temperature in relationship to glass transition temperature, the amorphous/crystalline ratio of the solute, and the presence of ionic materials may alter a material's solubility and prove the Teas approach inaccurate (McGlinchey 2002).

Teas (1971) suggested that the best way to determine a solvent system for a particular polymer was to:

1) plot on the Teas chart, using Teas fractional parameters, available solubility data from

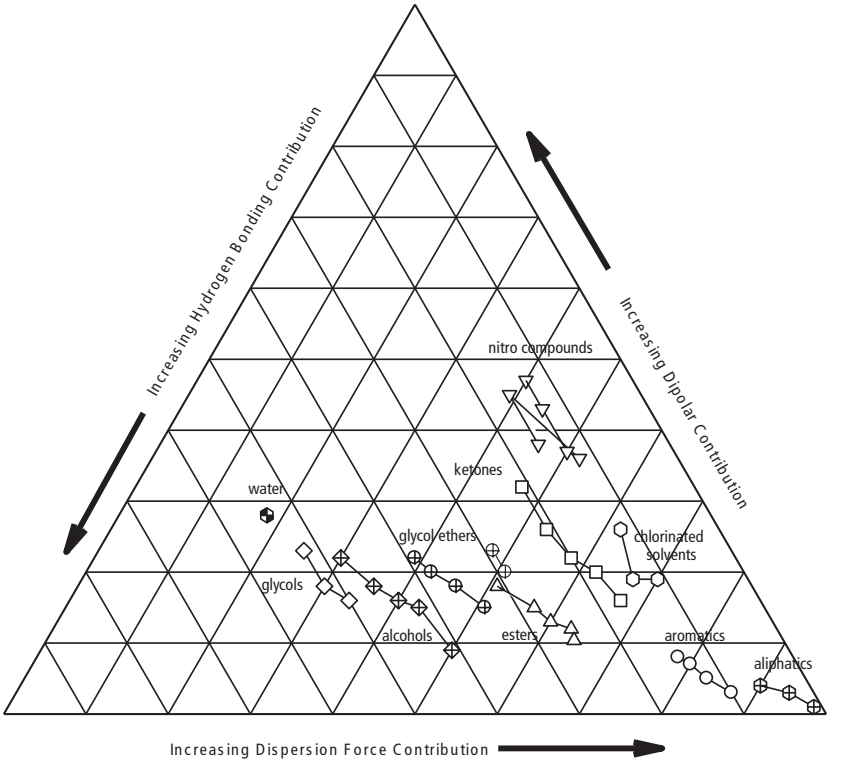


Figure 1. Example of a Teas chart. Adapted from Teas (1971, p. 3).

the polymer manufacturer indicating solubility, partial solubility and insolubility;
2) add to the data by doing a few solubility tests on the polymer with solvents, if published data is not sufficient to define an area of solubility;
3) draw a boundary line (area of solubility) on the chart that separates good solvents from poor solvents;
4) choose a solvent in the centre of the area of solubility, as this will have the best viscosity and the polymer will be spread out as much as possible in solution. If a solvent does not sit in the centre of the range or one is reluctant or unable to use that solvent due to government regulations, cost, ventilation requirements, etc., then choose two (or more) solvents located as close to the centre as possible and formulate a mixture that places it in the centre of the area of solubility; and
5) test the central singular solvent or mixture to ensure that it is compatible with the object and has the appropriate application, drying and film formation properties.

To help simplify this process, computer calculators (TeasTime or Solvent Solver) are available that easily determine the Teas fractional solubility parameter for solvent mixtures or a matching solvent mixture, given a target fractional solubility parameter (Ormsby 2006; Henry 1989).

For more information on solubility parameters see Barton (1991), Hansen (2007; 1982), Fisher (1982), Phenix (1993; 1992a; 1992b) and Eastaugh (1995; 1995b; 1994; 1994b).

Selecting safer solvents for adhesives

The threshold limit value (TLV) is the average concentration of a chemical substance (e.g. solvent) that a worker can be exposed to without injury for eight hours per day, five days per week (Geller 2003, p. 2145). There are three TLVs of interest (ACGIH 2011, pp. 3–6; Geller 2003, pp. 1922, 2155):

- *Time-weighted average* (TLV-TWA), which is the average exposure to a contaminant or condition that a worker can be exposed to without adverse effect over a period such as an 8-hour day or a 40-hour week.
- *Short-term exposure limit* (TLV-STEL), which is the maximum amount of harmful contaminant (e.g. gas or dust) that a person can be exposed to for a brief period (usually 15 minutes) without being harmed.
- *Ceiling limit* (TLV-C), which defines an absolute exposure limit (concentration) that should not be exceeded at any time during working exposure.

In the last 20 years, the level of exposure to some common adhesive solvents that a worker can safely be exposed to repeatedly day after day has been lowered, causing conservators to re-examine the solvents they use and try to find less toxic substitutes that will perform the same task. Data on 31 solvents with higher, more acceptable toxicity ratings (TLV-TWA > 200 ppm) have been published that also give vapour pressures and Teas fractional solubility parameters (**Table 3**) (Thorn 1992).

With this information, it is possible to find less toxic substitutes for dissolving adhesives. Thorn (1992) illustrated this technique by using Elvacite 2013 as an example, which is often dissolved in toluene (TLV-TWA = 20 ppm) for two reasons: toluene dissolves Elvacite 2013 quickly and toluene evaporates slowly enough to prevent cracking but fast enough to produce a non-tacky film. To find a less toxic substitute, the solubility area of Elvacite 2013 was plotted on a Teas chart with information obtained from the manufacturer's data sheet. The 31 solvents in **Table 3** were also plotted on the Teas chart, which identified a number of solvents that fell in the Elvacite 2013 solubility area. Next, the ideal vapour pressure for the solvent was chosen because this relates to the evaporation requirement. As toluene has a vapour pressure of 37 mmHg, all solvents that fell in the solubility area and had a vapour pressure, for example, between 15 and 40 mmHg were considered. Only propyl acetate and *sec*-butyl acetate satisfied these conditions. Thus, it should be possible to dissolve Elvacite 2013 with these less toxic solvents and preserve the same basic properties as a toluene solution.

Dispersions and emulsions

A *dispersion* is any system of matter in which finely divided particles are uniformly scattered throughout another medium (Cagle 1973, p. 25). The dispersed phase may be a solid, liquid or gas. If the dispersed phase is a solid and a polymer, then it may be referred to as a *latex* (Napper 1983, p. 2). If the dispersed phase is a solid and a non-polymeric material, then it is referred to as a *sol*. If the dispersion medium for a sol is aqueous, then it is called a *hydrosol*. If it is an organic liquid, then it is an *organosol* (Napper 1983, p. 2). The dispersion of a gas in a liquid is a *foam*, while an *emulsion* is a stable dispersion of one liquid in a second immiscible liquid (Napper 1983, p. 2). So an emulsion is always a dispersion, but a dispersion is not always an emulsion. Due to the scattering of light from the resin globules, these stable suspensions (emulsions) are usually opaque or milky white and are never clear-like solutions (Feller 1966).

TABLE 3. SOME SOLVENTS WITH TLV-TWA ≥ 200 PPM
(Thorn 1992)

Solvent	Vapour pressure (mmHg; 20°C)	Teas			TLV-TWA ppm
		f_d	f_p	f_h	
water	17.5	19	22	58	
pentane	439	100	0	0	600
hexane	137	100	0	0	500
heptane	37	100	0		400
octane	11	100	0	0	300
cyclohexane	82	94	2	4	300
cyclohexene		5			300
methylcyclohexane	38	94	0	6	400
bromochloromethane		65	22	13	200
bromoethane	390	64	13	23	200
1,1-dichloroethane	218	66	33	2	200
1,2-dichloroethylene	200				200
1,1,1-trichloroethane	100	70	19	11	350
ethanol	44	36	18	46	1000
methanol	96	30	22	48	200
propan-1-ol	17	40	16	44	200
propan-2-ol	35	41	17	42	400
diethyl ether	445	64	13	23	400
diisopropyl ether	61	70	10	20	250
dimethoxymethane		59	7	34	1000
tetrahydrofuran		55	19	26	200
acetone	216	47	32	21	1000
butanone	84	53	30	17	200
pentan-2-one	13	48	29	23	200
pentan-3-one	12	56	27	17	200
sec butyl acetate	18	52	29	19	200
ter butyl acetate					200
ethyl acetate	77	51	18	31	400
isopropyl acetate	50	52	22	26	250
methyl acetate	294	45	36	19	200
propyl acetate	29	57	15	28	200

Emulsions have three advantages over regular solutions: first, they can carry a higher polymer concentration than solutions; second, they can achieve relatively low viscosity with high molecular weight polymers (Feller 1966); and third, they are generally water-based, which makes them a non-toxic possibility.

An *emulsifier* is added to emulsions to help disperse the adhesive monomer in the external phase

(usually water) and is generally a water-soluble, low molecular organic compound that has hydrophilic and hydrophobic parts (Hoechst 1972, pp. 39–40). It forms micellae in aqueous solutions, which are spatial structures with all the hydrophilic and hydrophobic ends facing outwards or inwards. It is thus possible for hydrophobic molecules, such as monomers, to be taken up into the interior space of the micellae. Emulsifiers reduce interfacial surface tension of the water/monomer system and prevent the polymer from depositing out due to gravity. Emulsions of this type have particle size between 0.01 and 0.2 μm (Hoechst 1972, pp. 39–40).

Stable dispersions are also formed using *protective colloids*, but, unlike emulsifiers, they do not lower the monomer/external phase interfacial surface tension significantly and do not form micellae or solubilize the water-insoluble monomers (Hoechst 1972, pp. 39–40). They usually have a larger particle diameter (0.5–3 μm), greater viscosity and high stability to mechanical or other effects (e.g. frost, storage, pigment compatibility or electrolyte stability) (Hoechst 1972, pp. 39–40). The protective colloid of choice for vinyl acetate polymers is usually poly(vinyl alcohol) or cellulose derivatives. Sometimes mixtures of both emulsifiers and protective colloids are used to stabilize dispersions.

Dispersion adhesives harden by *coalescence* (from the word "coalesce", meaning to unite). First, water evaporates and the dispersion particles make contact, then the coalesced film hardens further as remaining moisture dissipates and the particles flow into each other.

Other additives in adhesives

Besides those already mentioned, there are other additives that could be added to adhesive formulations to change their flexibility, viscosity, rheology, pH, cost, setting properties and/or freezing properties.

Plasticizers

Plasticizers are polar compounds that are added to liquid adhesives to increase dry film flexibility. Plasticizers reduce the intermolecular attraction between sterically hindered groups in the adhesive that have a very strong attraction to each other and thus make the polymer stiff (Jaffe et al. 1990). They can increase the viscosity of some dispersions (especially vinyl types) but tend to reduce viscosity of polymer solutions because they act very similar to solvents. They help wet non-porous surfaces and lower glass transition temperature (Jaffe et al. 1990). In the dried film, they increase tack, reduce heat-seal temperature and improve water resistance (Jaffe et al. 1990). The worry about adding plasticizers to an adhesive is that they could migrate into the substrate and cause damage. Examples of plasticizers are dibutyl phthalate and polyethylene glycol dibenzoate, which are common ones, and ditridecyl phthalate, which is a heavier phthalate that might not migrate as readily as the lighter dibutyl variety (Selwitz 1988, p. 31).

Thickeners

Thickeners are added to liquid adhesives to increase viscosity and open-time. A thickened adhesive releases water or solvent more slowly than a non-thickened one (Jaffe et al. 1990). Also, a thickened adhesive reduces the likelihood of a starved joint with porous surfaces as it reduces penetration. Examples of thickeners are methylcellulose, starches, fumed silica, polyethylene oxide and neutral acrylic acid salts.

Fillers

Fillers are inexpensive additives that are added to extend the product and reduce cost (Jaffe et al. 1990). They also increase viscosity, reduce penetration into porous surfaces and change rheology (deformation and flow). In the dried film, they add stiffness and strength, and decrease the tack of the film. They also act as matting agents. Examples of some fillers are calcium carbonate, talc and china clay.

Freeze-thaw stabilizers

Freeze-thaw stabilizers are added to liquid adhesives to prevent them from freezing when shipped in cold climates. Many dispersion adhesives would separate and be destroyed if kept in freezing conditions without the addition of a freeze-thaw stabilizer. An example of a freeze-thaw stabilizer is ethylene glycol (anti-freeze).

Humectants

Humectants are added to liquid adhesives to prevent skinning on the surface of the adhesive (Jaffe et al. 1990). Humectants slow the setting of an adhesive and thus improve open time. They also help to plasticize the dried film of adhesive. Examples of humectants are glycerin, propylene glycol, sorbitol and sucrose.

Wetting

In order to achieve good contact between the adhesive and the substrate, the adhesive must wet the substrate surface well. High energy surfaces are polar, while low energy surfaces are non-polar (**Table 4**). Liquids (e.g. adhesive solutions or dispersions) will wet only surfaces of higher energy than their own (Cagle 1973, p. 1–12).

TABLE 4. RELATIVE SURFACE ENERGIES OF SOME MATERIALS
(Cagle 1973, p. 1–12)

Material	Relative surface energy
metal oxides	very high
acrylics	
poly(vinyl chloride)	
polyethylene	
silicones	
teflon	very low

To illustrate wetting, consider the effect of water on polyethylene and metal (Cagle 1973, p. 1–12). When a drop of water is placed on polyethylene, it beads. This occurs because water does not wet polyethylene, which has lower surface energy than water. Water is more attracted to itself than to the polyethylene and thus beads. However, when a drop of water is placed on metal, it spreads out. Water wets metal because metal has higher energy. Water is more attracted to the metal than to itself and thus spreads on the surface.

One can increase surface energy of a substrate by etching or sanding the surface (Cagle 1973, p. 1–13). This puts more oxygen into the surface, which makes it more polar and more wettable. Another way to increase wetting is to incorporate *wetting agents* into the composition of the liquid adhesive. This will help to eliminate "crawling" (redistribution of the adhesive that no longer evenly spreads out or wets the surface). Wetting agents can be anionic, non-ionic and cationic.

There are no common adhesives or liquids with surface energies lower than polyethylene, silicones and Teflon. Thus, no adhesive wets these materials well and bonding is very weak to these substrates. For this reason, they are often used as release materials for adhesives.

Wetting is also dependent on the s*urface tension* of a liquid, which comprises the contractive forces of the surface of the liquid film that make the liquid occupy the least volume. The unbalanced surface molecules are attracted towards the interior of the liquid (Cagle 1973, p. 57). *Critical surface tension* is defined as the surface tension of a solid equal to that of the liquid of highest surface tension that will wet the solid (Hansen 2007; Skeist and Miron 1990, p. 12).

Wetting is measured by *contact angle*, which is the angle at which the liquid meets the solid surface (Rudolph 2004). The smaller the contact angle, the better the liquid wets the surface (**Figure 2**). So, wetting is dependent on the physical and chemical properties of the two materials, liquid surface tension and solid surface energies (SunMan Engineering Inc.).

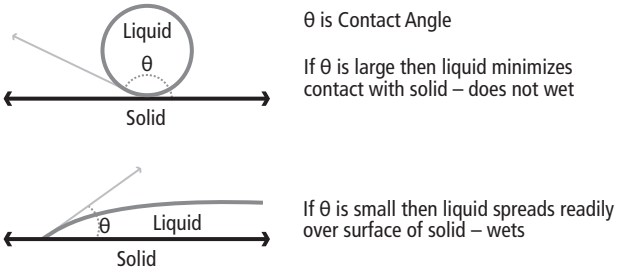


Figure 2. Contact angle.

In order to achieve the strongest bonds when bonding two substrates made of the same polymeric material, an adhesive should not only have a lower surface tension than the substrate, but also be close to the critical surface tension of the substrate (Niem et al. 1996). When bonding dissimilar polymeric materials, an adhesive should be selected that is closer to the critical surface tension of the substrate with the lower critical surface tension (Niem et al. 1996).

VISCOSITY

For good bonding, the liquid adhesive must flow over the surface of the substrate and have good contact with it. Viscosity is the ability of a liquid to resist change of shape and is measured in pascal seconds (Pa.s) or millipascal seconds (mPa.s). Old viscosity units are poise (P) or centipoise (cP) and can be converted to Pa.s or mPa.s using Equations 5 and 6 respectively.

$$10\ \text{P} = 1\ \text{Pa.s} \quad (5)$$

$$1\ \text{cP} = 1\ \text{mPa.s} \quad (6)$$

In *solutions*, viscosity increases with polymer molecular weight and concentration. In *dispersions*, viscosity is independent of polymer molecular weight and concentration. Low viscosity liquids can penetrate porous materials and are usually desired in consolidation. The viscosities of some common liquids and adhesives are given in **Table 5**.

TABLE 5. VISCOSITY OF SOME COMMON LIQUIDS AND LIQUID ADHESIVES
(Horie 2010, p. 282; Weast 1975, pp. F50–F55; product literature)

Liquid or adhesive	Viscosity (mPa.s)	Consistency
water	1	watery
antifreeze (ethylene glycol)	20	
olive oil	84	
cyanoacrylates (typical, unfilled)	~100	
glycerin	1490	syrupy
poly(vinyl acetate) dispersion (typical)	~2000–2500	
silicone rubbers	10,000–150,000	pasty

pH

pH is a measure of the acidity or alkalinity of an aqueous solution or dispersion. It is a measure of the concentration of hydrogen ions and is defined by Equation 7.

$$pH = -\log_{10}[H^+] \quad (7)$$

pH values are in the range of 1 to 14. If the pH is 7, then the solution is neutral. If the pH is less than 7, then the solution is acidic. If the pH is greater than 7, it is alkaline.

An acceptable pH range for conservation adhesives would be one that is tolerated by objects without damage. Since objects are composed of many different materials that possess varying tolerances to acid and alkali, it is difficult to set universal pH ranges. However, a pH range of 5.5–8.0 (**Figure 3**) has been chosen by some researchers as the one that could be tolerated without significant damage to most materials (Down et al. 1996). One should remember, however, that some objects are more stable in acid or alkaline conditions, and thus the acceptable pH range varies with the object.

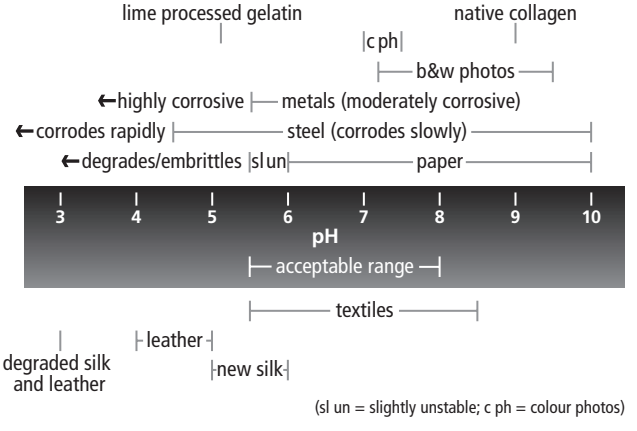


Figure 3. The pH stability range of various substrates.

There are a few standard test methods for measuring adhesive pH (BS EN 1245:2011; ASTM D1583-01 2006). Also, some standard methods for measuring paper pH have been adapted for adhesives (TAPPI T 509 om-11; TAPPI T 435 om-11; TAPPI T 529 om-09). A guide for measuring pH in conservation is also available (Tse 2007).

The easiest method to measure aqueous adhesive pH is to directly measure the wet solution or dispersion using a pH electrode and meter. A technique for measuring the pH of a dried adhesive involves extracting the dry film in distilled water (Down et al. 1996). Yet other methods involve taking surface pH measurements of dried films or micro-extracting very small amounts of adhesive (1–10 mg) in distilled water (50 µL) (Tse 2007). Less accurate techniques use pH indicator strips, pencils or pens, which only give a rough idea of the pH.

Adhesive pH given on data sheets is for the wet aqueous solution or dispersion, and is not a dry-film extracted pH.

SHELF LIFE

Shelf life, or storage life, is the period of time that the adhesive remains usable or the period that the manufacturer guarantees that the adhesive stored at a specified temperature will produce specified mechanical properties (Geller 2003, p. 1915; Cagle 1973, p. 53). It is frequently given on data sheets (e.g. six months at 15°C or one year at 22°C).

The shelf life of dispersions is determined by the hydrolysis of the polymer, biodegradation of components and/or physical stability of stabilizers. The shelf life of reactive adhesives is determined by reaction with the atmosphere (e.g. oxygen, water), thermal instability and/or cross-reaction with contaminants.

Usually, refrigerating adhesives will extend their storage life (Ebnesajjad 2008, p. 341).

3 Bonding

by Jane L. Down, Canadian Conservation Institute

Good bonding involves not only good wetting and coverage of the substrates with the liquid adhesive, if liquid adhesive is used, but also includes appropriate adhesive selection for the substrate (see Chapter 7 for more detailed discussions on adhesive recommendations for specific materials) and many other considerations, such as clean surfaces, good fit between the substrates, good contact between the adhesive and substrate, relatively thin bonds, and other joint design requirements to minimize stresses in the bond.

Clean surfaces and surface preparation

Good bonding occurs with clean surfaces, which are possible when there is proper surface preparation prior to bonding. The bond areas must be free from powder, dirt, grease, fingerprints and oxides, otherwise, the adhesive will bond to the powder, dirt, etc. and not to the substrate itself. As powder, dirt, etc. exhibit very weak bonds to the substrate, bonding to these contaminants leads to very weak bonds if not removed. While some metal oxides (e.g. on aluminum) are fairly stable, are well adhered to the metal and might not pose a great problem if bonding occurred to the oxide layer, others (e.g. rust on iron) are not so stable, are only loosely attached to the metal and would result in unstable bonds.

Generally, conservation bonding needs are not as stringent as industrial needs. Before proceeding to prepare a surface for adhesive bonding, carefully consider what surface preparation is adequate, appropriate, possible and ethical for the object. Quite often in conservation we do not feel comfortable preparing the surface for bonding as it would be prepared for an industrial application, so we weigh the loss of adhesion that could result. The following are examples of possible surface preparations for industrial applications, some may be suitable for conservation and some may not—please exercise caution.

Surface preparation of *metals* to remove grease, dust, oil and/or oxides can include chemical alteration of the surface if high-quality bonding is desired (removal of oxides), solvent cleaning and/or abrasive blasting (Adhesives Digest 1997). The amount of cleaning required is dependent, of course, on the object and amount of preparation that is ethical. It is also dependent on the metal, strength of the oxide layer and ultimate bond strength desired. Surface preparation of *wood* to remove resins, waxes, paints and oxidation products can include sanding or planing, as appropriate for the situation, but is often not an ethical option in conservation (Adhesives Digest 1997). Surface preparation of *non-metal surfaces* to remove similar contaminants can include solvent cleaning, followed by chemical alteration of the surface, abrasion and another solvent cleaning (Adhesives Digest 1997). The number of steps is dependent on the surface, desired bond strength and ethical considerations.

The four possible types of surface cleaning include the use of a detergent, use of a solvent, chemical alteration and mechanical cleaning (Adhesives Digest 1997).

The use of *detergent* solutions to remove dirt and oil is the easiest, least expensive and most non-toxic of all methods. However, some detergents can contaminate metals and glass. The detergent must be rinsed off thoroughly and the surface dried before bonding. Sometimes, the detergent can never be completely removed and this can pose problems.

The use of *solvents* to remove grease and oil involves wiping the surface with a cloth that is lightly saturated with solvent. Do not totally saturate the cloth because contaminants could be left on the surface with the solvent, and this contaminated solvent could be pushed into a porous substrate with the cloth. The cloth should be replaced as it becomes soiled. Sometimes in conservation, the surface is flushed with solvent rather than wiped with a cloth.

Chemical cleaning, rarely used in conservation because of its aggressive nature, can involve any or all of the following steps: degreasing, soaking in an alkaline bath, pickling and/or chemically altering the surface. The first step is to degrease with a solvent that is compatible with the surface (e.g. methyl ethyl ketone, toluene, naphtha, acetone or trichloroethane) to remove organic materials, wax, oil or grease. This is done by wiping, spraying or immersing, as dictated by the object and what is appropriate and ethical. An alkaline bath is always followed by a good rinse to remove metal chips, dirt, oil and other contaminants.

Pickling is done to loosen the oxide film from ferrous metals and involves baths of acidic solutions, the content of which depends on the metal in question. Chemical alteration of the surface involves either dissolving part of the surface (e.g. etching) or making the surface more chemically active by adding chemical functionality or coupling agents (e.g. silanes).

Mechanical cleaning is the simplest method to remove rust, scale, paint, etc. and to roughen the surface to bring more energy into it. It involves abrasion with sandpaper, emery cloth, wire brushes and/or scrapers, or by sandblasting, tumbling and/or sanding with power tools, followed by degreasing to remove the contaminants and loose particles. As an example of the effect of surface abrasion on bond strength, Niem et al. (1996) noted that an abraded polymeric substrate could increase the joint strength by as much as 40%, which was attributed to the increase in interfacial bond area and wettability of the substrate. However, the direction of sanding had no effect on the strength (Niem et al. 1996).

Water molecules on the substrate surface can sometimes cause problems with bonding. This contaminant can be reduced by working in a dry, warm environment (Horie 2010, p. 101).

Cleaning techniques should be done in a clean room and not in the same area where sanding or blasting is done. Also, once the surface is cleaned, it should be handled as little as possible and bonded as soon as possible, especially if metal, since re-oxidation and re-contamination could occur.

Specific surface preparation suggestions for individual substrates are often given in manufacturer data sheets and in various adhesive handbooks (Ebnesajjad 2008; Adhesives Digest 1997; Mahoney 1990*). However, it must be emphasized that the suggestions above and those in handbooks are for industrial uses and some can be toxic and hazardous to the health of the object and the worker, and must be assessed carefully for use in any conservation application.*

Substrate fit

Good bonding occurs when there is good fit between the substrates. Weak bonds are formed when the bonding area has gaps and spaces. So, it is necessary to eliminate as many gaps and spaces as possible. Unfortunately in conservation, joins are frequently not tight because of the age of the object and nature of the materials or the ethical constraint not to modify original material.

Adhesive and substrate contact

Good bonding follows when there is good contact between the adhesive and substrates. This is one reason why adhesives are applied as liquids; best contact occurs when the adhesive is a liquid and wets the substrate effectively. Also, good contact is achieved through clamping while the adhesive sets.

Bond thickness

There is a decrease in joint strength as bond-line thickness increases (Tomblin et al. 2002). Adhesive forces at the interface between the adhesive and substrate are larger than cohesive forces within the adhesive. So, for strong bonds, the adhesive should be thin to minimize the weaker cohesive forces as much as possible. Also, thicker bonds have higher void content (bubbles), which makes them weaker than thinner bonds with fewer voids. Suggested optimum bond thickness for non-porous materials is 0.1–0.15 mm (Adhesives Digest 1997). In conservation, where "strongest" is not always desired, bond thickness can be adjusted so that the adhesive fails cohesively before the substrate is damaged.

Joint design

There are four types of *bond stresses* that are important in joint design: shear, tension, peel and cleavage (**Figure 1**) (Ebnesajjad 2008, pp. 159–161).

Shear stress is imposed on the whole bond area, uses the joint area to the best advantage, and is the most resistant to joint failure. Generally, most bonds are formed in the shear mode or undergo some form of shear stress.

Tensile stress is also imposed on the whole bond area and is comparable in strength to shear. It is important for the substrates to be thick when bonded

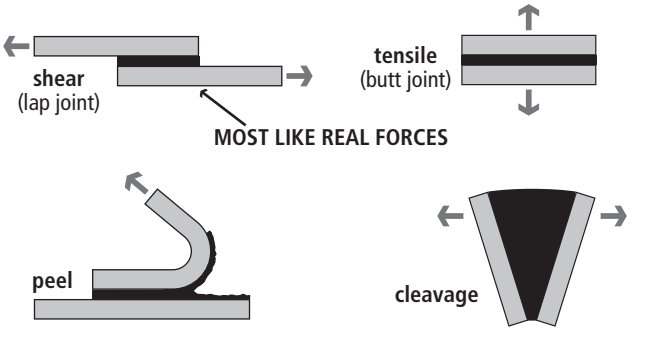


Figure 1. Examples of some bond stresses.

in the tensile mode, otherwise deflection occurs that involves other stresses (e.g. cleavage).

Peel stress only occurs when one or both substrates are flexible. The stress is placed on the boundary line of the joint and, unless the bond is wide and the load small, failure occurs.

Cleavage stresses occur if a tensile join deflects. It is not uniform across the join and a large bond area or length is needed to combat this stress.

There are stresses in a stationary join (no load applied), which are not uniform and cannot be totally eliminated but can be reduced through proper joint design (Petrie 2005; Adhesives Digest 1997). The average stress across the join is always lower than the maximum stress, but failure always starts at the maximum stress points, so it is advantageous to reduce maximum stress levels as much as possible. In a simple lap shear join, the maximum shear stress occurs at the edges (**Figure 2**).

"Tough flexible adhesives provide joints with a more uniform stress distribution and less of a difference between average and maximum stress. If high stress distributions are expected in service either because of external loading, such as peel or cleavage, or from internal stress, such as from thermal expansion or shrinkage, then tough, flexible adhesives and sealants are usually preferred over more brittle ones" (Petrie 2005). However, tough flexible adhesives have lower shear and cohesive strength, so are usually used on substrates of similar physical properties (tough and flexible) in less extreme environments, while rigid, cohesively strong adhesives are used in structural applications and extreme environmental conditions (Petrie 2005).

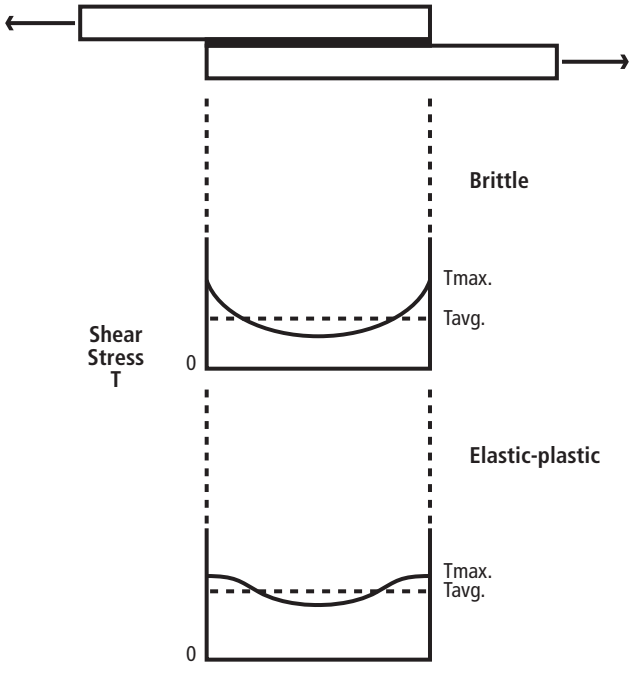


Figure 2. Shear stress distribution in lap shear join. Adapted from Adhesives Digest (1997, p. A-6).

For good joint design, the bonded area should be as large as possible, however, increasing joint size is not always possible in conservation. Strength is calculated from the area of the bond, which is its width times its length. Bond strength is directly proportional to its width, but is non-proportional to its length (Adhesives Digest 1997). After a certain length, further overlap does not improve the strength of the bond (**Figure 3**).

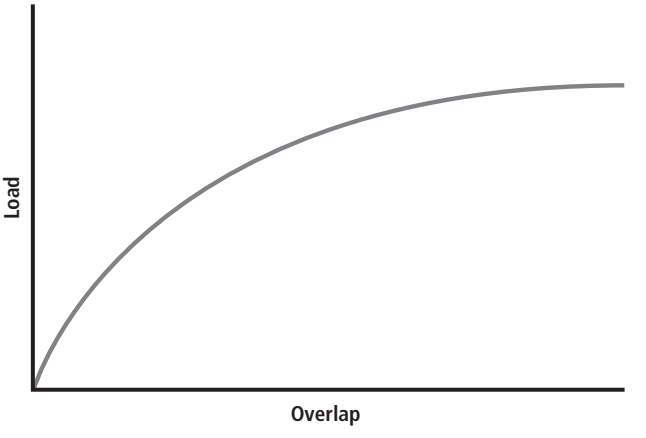


Figure 3. Effect of length of join (overlap) on bond failure load. Adapted from Adhesives Digest (1997, p. A-7).

The stiffness of the substrates is also a major factor in shear stress distribution across the bond. As the product of Young's modulus and thickness of the substrate becomes large (stiffer), the shear-stress distribution becomes more uniform (Adhesives Digest 1997).

Finally, shear stress distribution across the join approaches uniformity when adherends are tapered or stepped across the length of the bond, or when adherends are thick and a short overlap (length) is used (**Figure 4**) (Adhesives Digest 1997). If joint design and size can be modified and one wants to improve bond strength, see sections on joint design in Ebnesajjad (2008), Shields (1984) or Cagle (1973).

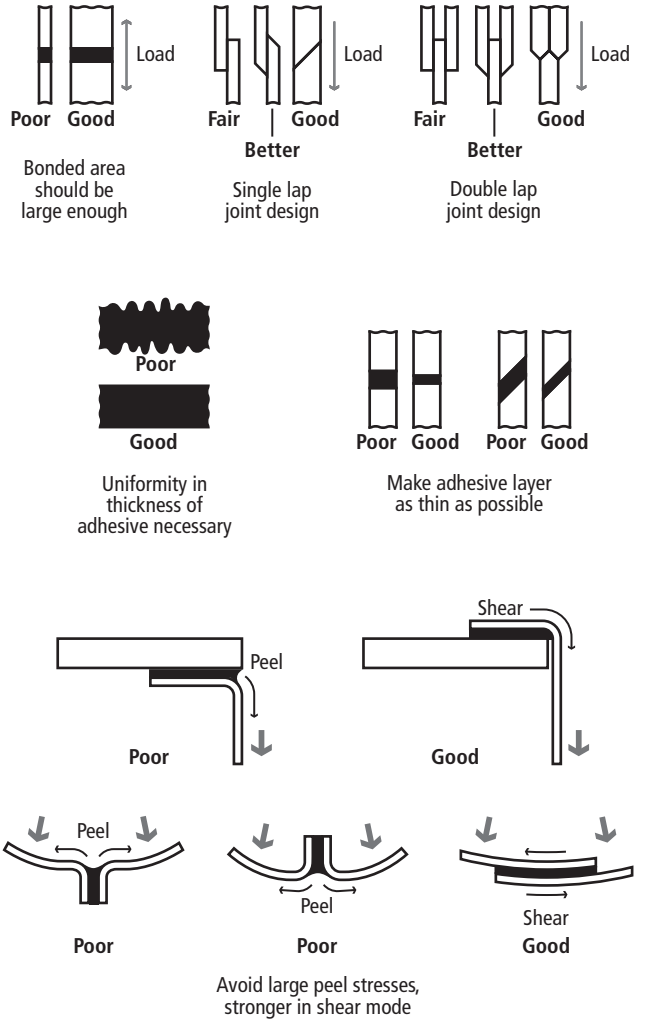


Figure 4. Joint design tips that will improve adhesive bond strength (US Department of Defense 1987, p. 35; Cagle 1973, pp. 6–20). Reproduced with permission from the McGraw-Hill companies.

Sometimes, joint design can be modified so that a weaker, more removable adhesive can be used by altering the way that forces act on the join. An example of this is given in the redesign of the V-hinge for resin-coated photographs (McGlinchey and Yuan 2005). The classic V-hinge requires adhesives with high peel strength, which are difficult to remove. In the redesigned V-hinge (**Figure 5**), the forces are in the shear mode, which are stronger than the peel mode, allowing for weaker, more peelable and removable adhesives to be used. This modified V-hinge has the same distribution of forces as a pendulum hinge used by paper conservators, but is concealed by the object so it permits objects to appear to 'float' against a wall or within an oversized matt.

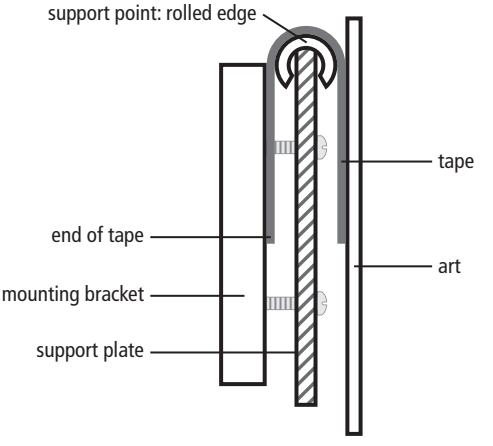


Figure 5. Redesigned V-hinge for resin-coated photographs (McGlinchey and Yuan 2005).

Setting mechanisms

Before discussing mechanisms, it is important to understand the difference between the terms *set* and *cure*.

"*Set* - to convert an adhesive into a fixed or hardened state by chemical or physical action, such as condensation, polymerization, oxidation, vulcanization, gelation, hydration, or evaporation of volatile constituents" (ASTM D907-05 2006, p. 42).

"*Cure* - to develop the strength properties of an adhesive by chemical reaction" (ASTM D907-05 2006, p. 39).

So, *set* is the all-inclusive term for any chemical or physical conversion, while *cure* only relates to adhesives that set by chemical reaction.

There are three basic mechanisms for adhesive setting: liquid solidification, evaporation and chemical cure.

Liquid solidification

Liquid solidification is the melt-freeze setting type whereby adhesive cools from a molten state to a hardened state (a.k.a. heat-seal or heat-set). Adhesives that set by this mechanism would be thermoplastic (e.g. ethylene/vinyl acetate copolymers and waxes). Some adhesives work via two setting mechanisms (e.g. animal glues), which are hot solutions that cool to a gel and then the solvent, usually water, evaporates.

The *advantages* of liquid solidification setting mechanisms include easy placement of adhesive, less mess, quick setting, fairly easy removal usually by reheating, no moisture evaporation to worry about—except in the case of some animal glues,

as mentioned above—and less shrinkage on setting than evaporation types.

The *disadvantages* include the use of heat on the object, loss of substrate moisture, internal stresses in the bond, and possible object and substrate colour changes due to heat.

EVAPORATION

Evaporation setting occurs when a solvent evaporates from the adhesive mixture. The adhesive polymer can be in solution, dissolved in a solvent (e.g. Paraloid F-10, HMG), or can be in a dispersion (e.g. Jade 403). In the latter case, the dispersant evaporates, leaving coalesced solid. Contact and pressure-sensitive adhesives (PSAs) are initially applied as solutions or dispersions to one or both substrates and are allowed to dry, and then the substrates are pressed together.

The *advantages* of the evaporation setting mechanism include more easily spreadable adhesives that are generally more removable than reaction adhesives, and non-toxic properties, if aqueous.

The *disadvantages* include shrinkage in the bond area due to the solvent evaporating—except in the case of PSAs where the bond occurs after evaporation has happened—adhesive embrittlement, and toxicity, if the adhesive is prepared in organic solvents (e.g. toluene).

CHEMICAL CURE

Chemical cure setting is produced in-situ by chemical reactions within the adhesive. These types of adhesives are usually thermosetting (e.g. epoxy resins).

The *advantages* include less shrinkage in the bond area upon setting as no solvents evaporate, fast setting (usually), and strong bonds.

The *disadvantages* include a sometimes exothermic cure (produces heat)—which might damage the object—irreversibility or difficulty with removal, and less flexibility.

SETTING TIME

Setting time is the period of time required for an adhesive to set in a bonded assembly. This bonded assembly can be subjected to heat and/or pressure during setting (Ebnesajjad 2008, p. 346). In creating a bond, one needs enough time to allow the adhesive to flow and wet the surfaces.

SHRINKAGE ON SETTING

Shrinkage is the volume reduction that occurs when adhesives set. It is expressed as percentage of volume or as percentage of linear shrinkage (Ebnesajjad 2008, p. 339). The cause of shrinkage on setting is usually due to solvent loss. **Table 1** gives examples of typical shrinkage for various adhesives.

Croll (1979) developed a theory to explain the development of internal stresses in films of solvent-based thermoplastic polymers after drying (Moran and Whitmore 1995). This theory, which suggested that higher shrinkage stresses occur when films of polymer have a high elastic modulus and/or a high glass transition temperature (T_g) (see Chapter 4) and are cast from a poor solvent, was tested on poly(vinyl acetate) and methacrylate films and was shown to be valid (Moran and Whitmore 1995). In a further study, coatings formulated with polymers of high T_g or elastic modulus dried with substantial shrinkage stresses even larger than cohesive or adhesive strength of the coating, which left the coating at high risk for further damage from handling or environmental change (Whitmore et al. 1999). Therefore, use of softer (low T_g) polymers and good solvents that reduce shrinkage stresses are recommended.

TABLE 1. EXAMPLES OF ADHESIVE SHRINKAGE ON SETTING
(Petrie 2007, p. 71; Horie 2005)

Setting mechanism	Adhesive	Volumetric shrinkage (%)
pressure-sensitive (after solvents evaporated)	adhesive tape	0
chemical cure	epoxy resin	4–5
liquid solidification	wax	13
evaporation (dispersion)	poly(vinyl acetate)	50
evaporation (solution)	cellulose nitrate	60
liquid solidification/ evaporation	hot-melt animal glue	65

4 Solid Adhesive Properties

by Jane L. Down, Canadian Conservation Institute

Several properties of the solid adhesive are important for conservation. The following will be discussed in this chapter: glass transition temperature (T_g), mechanical properties, coefficients of expansion, optical properties, aging/testing and removal.

Glass transition temperature

T_g is the temperature at which a solid changes from a glassy state to a rubbery state upon heating (Schilling 1989). It is a second-order transition, as opposed to a first-order transition (melting), because "energy is not liberated in reversing through T_g" (Schilling 1989, p. 110). Below the T_g, the solid (e.g. adhesive) is hard and glassy. Above the T_g, it is rubbery. T_g gives an indication of the ability of polymer chains to move past one another. If forces between the chains are high, then T_g is high. Raising the temperature allows chains to move past each other more easily. T_g is affected by the composition and structure of the polymer, hydrogen bonding, secondary bonding, crosslinking, scission of the polymer chains, length of the side chains, molecular weight and plasticizers (Schilling 1989). In conservation applications, it is important to know the value of T_g because, if the set adhesive is soft or tacky, it will pick up dust, while if it is hard and brittle, it could crack when stressed (Horie 2010, p. 23). Also, the adhesive T_g should not be close to ambient, as dramatic dimensional changes could occur if temperature fluctuates around the T_g, especially if the adhesive and substrate expand and contract by different amounts (Schilling 1989).

Glass transition temperature and setting

T_g indicates the flexibility and/or hardness of the polymer upon setting. **Figure 1** illustrates regions of liquid, rubbery or glassy behaviour related to setting time and temperature for hypothetical thermosetting adhesives. In **Figure 1**, if one follows a liquid adhesive setting at room temperature and if the polymer has a T_g **below** room temperature (top – Adhesive A), then the adhesive will move from a liquid state to a rubbery state, but will never reach the glassy state unless cooled. On the other hand, if the polymer's T_g is **above** room temperature (bottom – Adhesive B) and the adhesive is set at room temperature, the adhesive will move from a liquid to a rubbery state and finally to a glassy state. Depending on the adhesive, it could be made rubbery again if the temperature was raised. T_g values for various adhesives are given in **Table 1**.

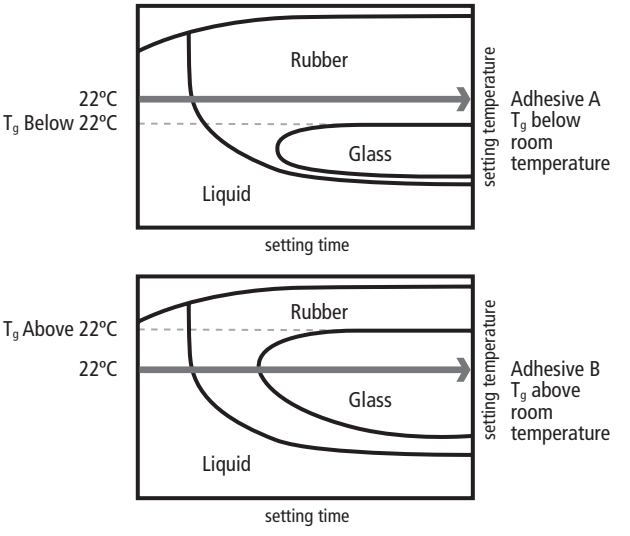


Figure 1. Setting time/temperature phase diagrams for model thermosetting adhesives. Adapted from Adhesives Digest (1997, p. A-3).

Table 1. T_G for various adhesives
(Roff and Scott 1971, pp. 69, 84, 92, 463; Horie 2010, p. 139; Talas d)

Adhesive	T_g (°C)	Set state at 22°C
silicone rubber	-112–125	rubbery
Lascaux 360 HV	-28	rubbery
Mowilith DMC2	10	rubbery
poly(vinyl acetate)	28	semi-glassy, semi-flexible
Paraloid B-72	40	semi-glassy, semi-flexible
poly(vinyl butyral)	49	semi-glassy, semi-flexible
poly(methyl methacrylate)	105	glassy/rigid
epoxy resin (aliphatic amine hardeners)	100–150	glassy/rigid

LOWERING THE GLASS TRANSITION TEMPERATURE

The higher the molecular weight of the adhesive, the greater the T_g "because the chains have less freedom of movement" (Horie 2010, p. 25). This freedom of movement can be increased by separating the chains using low molecular weight additives such as plasticizers, solvents, water or adhesives with lower T_g values (Horie 2010, p. 25). The presence of such additives decreases the average molecular weight and hence the T_g value, while increasing the flexibility of the adhesive. As the solvents and/or water evaporate, the T_g will increase, but less volatile plasticizers and low T_g additives remain to keep the adhesive flexible. Sometimes, solvents remain in the film long after setting, which creates a more flexible film. But upon aging, these solvents dissipate and the film becomes less flexible. Solvent is retained in films that possess T_g greater than ambient temperature (Schilling 1989). Therefore, it is desirable to have an adhesive with a T_g lower than ambient temperature so that all of the solvent is eliminated. Complete solvent elimination results in less change in T_g upon aging.

GLASS TRANSITION TEMPERATURE AND AGING

An adhesive film exposed to temperatures below its T_g remains glassy and relatively uncontaminated by dust as it ages. However, an adhesive film exposed to temperatures higher than its T_g as it ages will remain largely rubbery and can pick up dirt and contaminants that can catalyze degradation (Tímár-Balázsy and Eastop 1998, p. 318). Although a glassy adhesive might be better for aging purposes, it is not always preferred in conservation treatment as it might not "give" in the join.

MECHANICAL PROPERTIES

For most polymer-based adhesives, flexibility and cohesive film strength are inversely related: the higher the film strength, the lower the flexibility (Petrie 2005). Generally, adhesives should mimic the flexibility and strength of the substrate—except when the substrates are rigid.

For example:

- For *textiles* that are flexible, it is best if the adhesive is also flexible. The lower strength that comes with increased flexibility should not be a problem for flexible light textiles.
- For *wood* that is semi-rigid, it is best if the adhesive is also semi-rigid. The higher strength is beneficial for these heavier objects.
- However, for *ceramics* that are rigid, it is better if the adhesive is semi-flexible so that it "gives" in the join. Stiff adhesives could produce stresses in the join, could fail and could damage the ceramic.

So, generally in conservation, flexible to semi-flexible adhesives are desired.

STRESS-STRAIN CURVE

Stress is the force, per unit cross-sectional area, required to stretch a specimen to a stated elongation, while strain is the ratio of the extension to the original length of the specimen (Cagle 1973, p. 56). Stress-strain data is measured by instruments known as "strength testers" (e.g. Instron tensile tester). They can show the relationship between load and deformation of a specimen graphically from zero loading to rupture of the film. The cohesive strength, stiffness (flexibility) and elongation (extensibility) of an adhesive can be determined from a stress-strain curve (**Figure 2**). The first part of the curve describes the adhesive's elastic behaviour (modulus of elasticity). As a specimen is extended in a strength test, it passes through various stages: from its elastic state to the yield point to the final ultimate strength and elongation of the film as it extends to its breaking point.

Modulus of elasticity is a measure of a material's stiffness. High modulus adhesives are characterized by stiffness, while low modulus ones are characterized by flexibility.

Yield point is the lowest stress at which strain increases without an increase in stress (Geller 2003, p. 2314). It is the stress at which a material starts to deform plastically. Before this point, all stress is elastic and the material will return to its original shape when the stress is removed. Once stressed beyond the yield point, the adhesive film is deformed permanently.

BOND STRESSES

The stresses that an adhesive bond can undergo may be singular or combined but, in the simplest deconstructed form, constitute these various types: tensile, shear, peel, cleavage, impact, creep, fatigue, flexural or compressive, of which some are shown in **Figure 3**. More than any other type, shear stress is thought to be most like the forces experienced in a real application in a bond. The extent to which an adhesive can withstand these types of stresses under controlled conditions is given as the strength of the adhesive. Estimates of the type and magnitude of stresses in a particular bond may be made and compared to adhesive strength information given on data sheets. However, it is wise to remember that strength information on data sheets relates to a particular substrate and may not be accurate for your substrate.

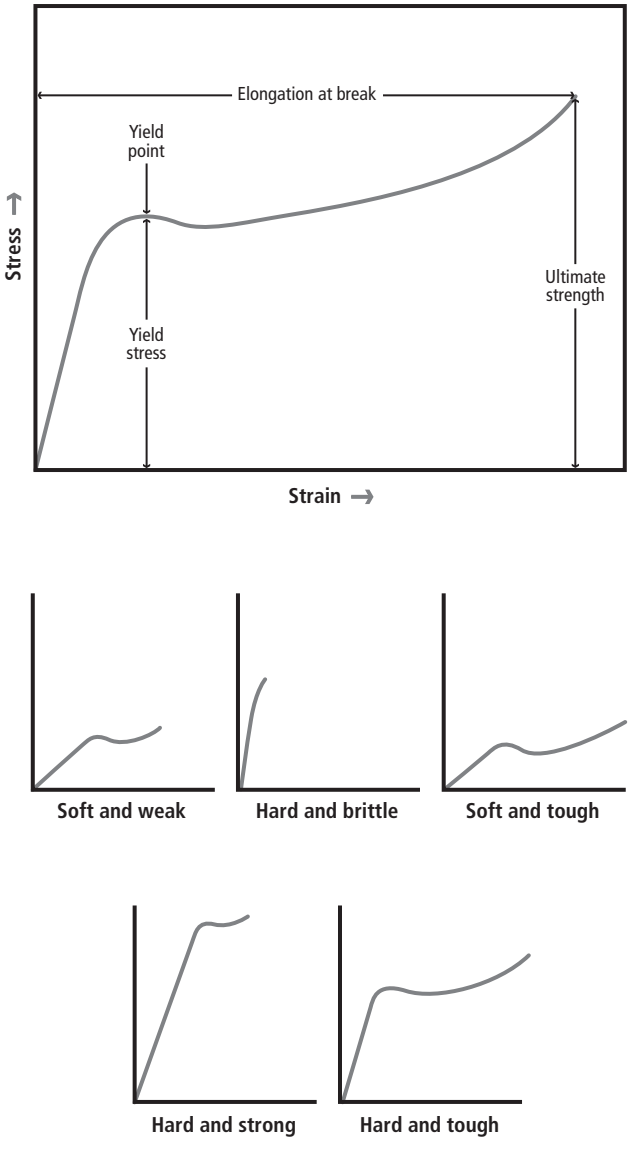


Figure 2. Typical stress-strain curve for thermoplastic materials (top) with examples of various types (bottom). Adapted from Winding and Hiatt (1961, p. 66).

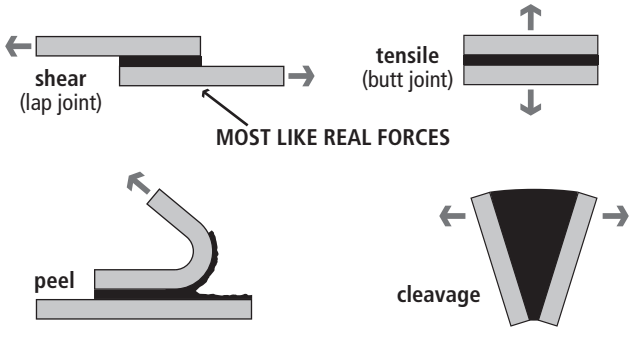


Figure 3. Bond stresses.

Also, quite often, the strength of many adhesives far exceeds that which is needed in many conservation applications (Horie 2010, p. 30).

Mechanical properties are influenced by the composition of the adhesive and substrate; the molecular weight and T_g of the adhesive; thickness and area of the bond; speed, humidity and temperature of testing; means of preparation; and age of the specimen (Horie 1987, p. 23).

There are generally two types of mechanical tests for adhesives that are performed for conservation applications: a film cohesive strength test or a bond strength test. Cohesive strength testing is performed on dry adhesive films, involves no substrates and gives information on cohesive strength, extensibility and flexibility of the adhesive film. Adhesive bond strength testing involves gluing substrates together in various orientations and then pulling, peeling or compressing them to determine the degree of stress at the breaking point (e.g. tensile, shear or peel strength).

HARDNESS

Hardness of an adhesive can indicate its resistance to scratching (if applied to an open surface like a coating) and day-to-day forces associated with handling and packing (Shashoua 1993b). It is the resistance of the surface to indentation and is measured in various ways, such as scratch resistance using abrasives, resistance to indentation using pencil hardness, pendulum damping with a Sward hardness rocker or flexibility with a bend test (Horie 2010, p. 31).

COEFFICIENTS OF EXPANSION

All materials contract or expand with temperature change and this dimensional change is described by a coefficient of expansion. The coefficient of thermal expansion (CTE) is also known as the coefficient of cubical expansion (CCE) (the increment in volume of a unit volume of solid, liquid or gas for a rise in temperature of 1° at constant pressure), as opposed to the coefficient of linear expansion (CLE) (the increment of length of a solid in a unit of length for a rise in temperature of 1° at constant pressure) (Geller 2003, pp. 421–422). In order to eliminate thermal stresses in the bond, the CTE (or CLE) of the adhesive and the substrate should be similar. Most adhesive polymers, however, have CTE (or CLE) values considerably higher than the materials they bond (**Table 2**). These large differences can cause bond failure, so use of low modulus (flexible) adhesives can help reduce thermal stresses in the bond.

TABLE 2. CLE OF VARIOUS ADHESIVES AND SUBSTRATES
(Borch et al. 2002, p. 430; Engineering Toolbox; Shields 1976, p. 299; Cagle 1973, p. 18-3)

Adhesive/Polymer	CLE $\times 10^{-6}$/°C	Substrate
	<10	glass, poly(methyl methacrylate), steel, iron, brick, marble, porcelain
	20	aluminum, brass, copper, leather, paper
	40	wood-fir
	60	wood-oak, polystyrene
poly(vinyl alcohol) (70–100) epoxy (70–120) cyanoacrylate (80–100)	80	cotton, rayon
shellac (91) poly(vinyl acetate) (85–130) cellulose nitrate (70–160) polyamide (100–118)	100	
cellulose acetate (70–180)	120	
poly(vinyl butyral) (80–220) ethylcellulose (90–200) cellulose acetate butyrate (70–370)	140	polypropylene
silicone (50–400)	160	
ethylene/vinyl acetate copolymer vinyl acetate/ethylene copolymer (160–200)	180	
	200	polyethylene

OPTICAL PROPERTIES

REFRACTIVE INDEX

Refractive index (RI) refers to the distortion that occurs when light travels through different transparent media and can be expressed by the following equation:

$$\text{RI} = \frac{\text{speed of light in a vacuum}}{\text{speed of light in a medium}}$$

Since light travels faster through a vacuum than through any other medium, RI is always >1. A common example of refraction is a rod placed in a glass of water. The rod appears bent at the interface between the water and the air. Light travels faster through air than it does through water and bends, or refracts, at the interface. The image below the water appears distorted.

RI is important when gluing transparent materials. If the RI of the adhesive and substrate are quite different, distortion occurs and the glue-line is visible; if, however, RI values are similar, then an almost invisible glue-line is possible. **Table 3** lists RIs of various polymers and glasses.

COLOUR

Generally in conservation applications, adhesives should be clear and colourless and remain so upon aging. However, adhesives often tend to yellow. This effect can be measured by the yellowness index, the CIE L*a*b* system or the change in absorbance at 380 nm (ASTM D1729-96 2006; ASTM D2244-05 2006; ASTM E313-05 2006; Down et al. 1996). The amount of perceptible or acceptable yellowing is different for each application and depends on the nature of the object (e.g. transparent, light- or dark-coloured) and bond location (e.g. exposed or hidden).

Table 3. RIs of some polymers and glasses
(Lide 2004; Tennent and Townsend 1984c; Cowan 1977; Roff and Scott 1971)

Polymers and other materials	RI	Glasses
water	1.333	
ethanol	1.360	
silicones	1.405–1.415	
paraffin	1.433	
poly(vinyl acetal)	1.45	
poly(vinyl acetate)	1.455–1.47	
cellulose ether	1.4–1.5	
	1.459	quartz (fused)
acrylic resin	1.475–1.50	
poly(vinyl butyral)	1.49	
polypropylene	1.49	
	1.495–1.54	Roman and Egyptian
cellulose acetate	~1.5	
cellulose nitrate	1.50–1.51	
	1.51–1.53	post-medieval
poly(vinyl alcohol)	1.50–1.53	
polyethylene	1.51–1.52	
	1.517	zinc crown
gelatin (various)	1.519–1.534	
	1.520	high dispersion crown
natural rubber	1.526	
starch	1.53	
epoxy resin	1.545–1.585	
	1.55–1.57	leaded glass
	1.575	light flint crystal
	1.650	heavy flint crystal
	1.890	heaviest flint crystal

Aging and testing adhesives

Chemical mechanisms of polymer aging can include crosslinking or chain scission, and the effects of these processes may be embrittlement, discolouration and shrinkage, to name a few. Adhesion can fail due to deterioration of the adhesive, the object or the whole bond assembly. Determination of the stability of an adhesive is accomplished by studying its aging under natural or artificial conditions. Aging can include thermal, photochemical, humidity cycling or pollution. Tests that measure these effects are performed before and after aging and can include measuring yellowing, solubility, strength, pH, etc. The most stable adhesives are identified according to pre-determined criteria. In some cases, aging can be correlated to conditions in museums or archives, and in other cases it is difficult to achieve a correlation.

Sometimes the Arrhenius equation is used to estimate the rates of aging processes in the dark at ambient temperatures by measuring the rates of processes at higher temperatures. Since higher temperatures speed up processes, this allows acceleration of the measurement of aging and can theoretically provide an answer about aging effects quicker than would be possible if waiting for room temperature degradation to happen (**Figure 4**).

The Arrhenius equation predicts a linear relationship between the natural logarithm of the rate of a chemical reaction and the reciprocal of absolute temperature given by the following equation (Feller 1994, pp. 144–147; Bégin and Kaminska 2002).

$$\ln(k) = \ln(A) - \frac{E_a}{RT}$$

Where A, Ea and R are constant terms, k is the rate of a reaction or process, and T is the absolute temperature.

The Arrhenius equation was developed for simple bimolecular gas phase reactions, but it can apply to other more complex processes. It is useful when straight-line plots are obtained, but this is often not the case.

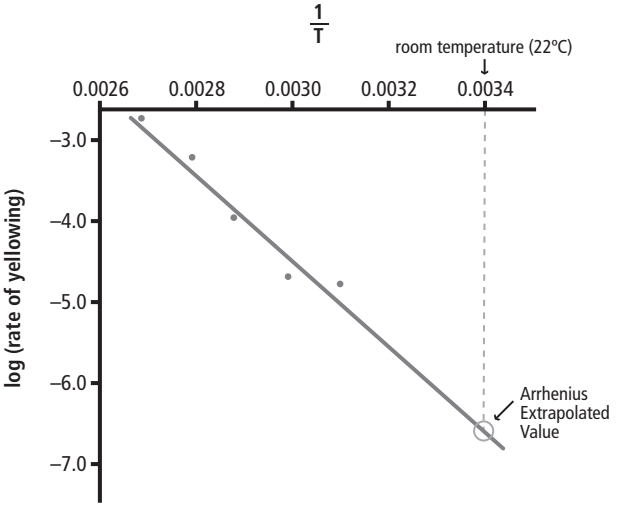


Figure 4. Accelerated thermal aging and the Arrhenius equation.

Using the Arrhenius equation in practice involves aging materials at several elevated temperatures, measuring degradation rates, and extrapolating plots of reaction rate versus reciprocal absolute temperature to ambient conditions. Using this technique to predict ambient temperature reaction rates is not always effective or accurate due to the following difficulties:

- Sometimes what happens at elevated temperature is not what occurs at room temperature. Physical changes and other chemical processes may become dominant at elevated temperatures that make the Arrhenius equation problematic to interpret at ambient temperatures.
- Several mechanisms of deterioration may be operating for different components of the adhesive, so it becomes difficult to fit a straight-line extrapolation that the Arrhenius equation requires.
- Arrhenius extrapolations crossing the T_g of the components may be unreliable (Celina et al. 2005; Edge 1996).

For light aging (measuring photochemical processes), it is generally difficult to compare the results of aging experiments from different laboratories because no standard exposure conditions have been agreed upon. Each laboratory tends to use different lamps with different spectral power distributions, exposure intensities/times, temperatures and relative humidity (Saunders and Kirby 2001). Also, if one ages a material under high light intensity and extrapolates results to lower intensities that might be found in a museum, it is necessary to prove that there is a simple linear relationship between the changes that take place at high intensity and those which occur at lower intensities (prove reciprocity). It is important to note that, from a chemical viewpoint, reciprocity is not always followed, which could be due to practical considerations such as high light intensities raising sample temperatures or lowering moisture contents, discontinuous exposure (periods of light and dark), or certain photochemical reactions that do not follow the reciprocity principle but instead have a different proportionality (e.g. square root of intensity) (Feller 1994, pp. 53–54). Proving reciprocity is a time-consuming process that depends on the spectral distribution of the lamps, absorbed intensity and nature of the material.

Feller (1977; Whitmore 2002, pp. 353–369) has defined various stages of deterioration for organic materials (inception, induction, auto-catalytic, steady state and declining rate). The type of deterioration and stages that an adhesive can undergo are dependent on the nature of the adhesive, its chemical components and the type of aging employed. Aging can give an indication of useful lifetimes of adhesives and Feller (1975; Whitmore 2002, pp. 107–119) has suggested the following classification for conservation (**Table 4**): excellent stability (A), where materials would last more than 100 years; intermediate stability (B), where properties would change sufficiently in 20 to 100 years to warrant removal; unstable class (C), where properties would last from 6 months to 20 years before removal was required; and the temporary class (T), where contact with the object should only be in the order of a few hours to 6 months.

TABLE 4. CLASSIFICATION OF USEFUL LIFE OF MATERIALS
(Feller 1975)

Class	Years to deterioration	Descriptor
A1	>500	excellent
A2	>100	excellent
B	20–100	intermediate
C	<20	unstable
T	<0.5	temporary

Other types of aging measure physical processes rather than the chemical processes discussed above. One example is dynamic mechanical analysis, which has been used for time/temperature superpositioning of curves (e.g. creep compliance or flexural modulus) to project polymer performance properties up to 20 years (Sichina 1988). This time/temperature superpositioning technique has also been used to extrapolate to room temperature for tensile elongation (Gillen et al. 2005).

Sometimes, technical data sheets include information on adhesive stability. This data may, however, refer only to the storage conditions required for the adhesive, the capacity of an adhesive to undergo a freeze-thaw cycle, or the service temperature range that the adhesive can withstand without losing its properties. Weatherability of an adhesive is almost never given on data sheets. It refers to the ability of an adhesive to resist deterioration due to exposure to weather such as to heat, light, humidity, precipitation, etc. The resistance of an adhesive to deterioration is an important aspect of adhesive selection. To reverse or undo adhesive joints that have yellowed or deteriorated is a delicate process and often dangerous for the artifact. The longer this task can be postponed, the better. Using adhesives with good resistance to deterioration prolongs the object's life. Since most information on stability will not be given on a manufacturer's data sheet, it is wise to refer to conservation research aging studies on adhesives. Many of these studies have been summarized in Chapter 6.

For the practicing conservator undertaking adhesive treatments, it is advisable to make sample films of adhesives used in the treatment that are well labelled, dated and stored so that blatant evidence of aging failure can be easily seen, and so that samples for future removability testing are available.

REMOVING ADHESIVES

The term "reversibility" once meant that any material added to an object should be easily removed (Down 1999). Now, it seems to extend to all conservation processes done to an object (Keyserlingk 1992; Appelbaum 1987). Reversibility is considered a property of the whole treatment and removal method, and not a term applied to a polymer in isolation (Appelbaum 1987; Horie 1982). People debating reversibility seem to agree that reversibility in the *strictest* sense does not exist; as soon as an object is touched, it is altered and the added material can never be completely removed (Oddy 1995; Caldararo 1990). Several studies illustrate the truth of this statement. Residues remain even after extensive and invasive removal techniques (Schniewind 1988; Charola et al. 1986; Horie 1982).

Horie (1982) has suggested that there are four levels of reversibility:
- not reversible;
- basic level, which takes an object back to a state that *looks* like the pre-treatment state;
- higher level, where the treatment, when reversed, does not affect any subsequent treatment; and
- the highest level, which leaves no trace whatsoever of contamination and no alteration of the structure of the object.

Whatever the debate, it remains a major goal in conservation treatment to be able to remove an adhesive from an object, even after many years, and this should be built into every treatment design. There are many ways to remove adhesives from objects and to separate bonds. Usually, but not always, one starts with the least intrusive technique (e.g. mechanical and heat techniques) and moves to more aggressive solvent solutions (e.g. gels, vapours or immersion), if required. The removal technique is dependent on the object and the adhesive bond, and must be considered carefully.

ADHESIVE REMOVAL TECHNIQUES

Adhesive removal techniques used in conservation and discussed below include mechanical scraping, heat, solvents, enzymes and microemulsions.

Mechanical

The adhesive is scraped away using an implement, such as a stainless steel spatula, a scalpel and/or an eraser.

Heat

The removal of the adhesive is aided by the application of heat, either by placing a tacking iron directly on the object or elevating it above the adhesive bond; by using a heated spatula directly on the object; or by putting the object on a warming tray.

Solvents

Determining best solvent for removal

There are several removal techniques that involve solvents; however, the first issue is to determine effective solvents to remove the adhesive. If the adhesive is known, then one could go immediately to the appropriate adhesive class in Chapter 6 and find possible solvents in the related "Solubility and/or Removability" section. Another method would be to match solubility parameters of the generic polymer in the adhesive to possible solvents (see Chapter 2). If the adhesive is unknown, a microscopic solubility test to determine the best solvent for removal may be performed. For this technique, enough adhesive from the object (microscopic samples) must be available. There are three possible microscopic tests that can be performed:
- The *McCrone's solubility test* uses a microscopic sample of adhesive that is placed on a microscope cover-slip which is inverted onto a glass ring on a glass microscope slide (McCrone 1965). A drop of solvent is placed outside the ring. Vapours seep under the ring and dissolve the adhesive particle. The dissolution or non-dissolution is observed with a microscope. The whole procedure takes about two minutes and more than one sample at a time can be run.
- A *microscopic swelling test* is useful when the adhesive does not dissolve [e.g. cured epoxy resins or dried poly(vinyl acetate) dispersions] (Down et al. 2002). A microscopic adhesive sample is placed in a "well" on a microscope slide. After measuring the length of the adhesive with a microscope micrometer attachment, solvent is added to the well, covered and the swelling is observed. The greater the swelling, the better the removability of the adhesive in that solvent. The ability of a polymer to swell is determined by the chemical nature of the polymer and the solvent, the molecular mass and flexibility of the polymer chain, the packing density and phase state of the polymer molecules, the inhomogeneity of the chemical composition of the chain, the presence or absence of crosslinks, and the temperature (Mencer and Gomzi 1994).

- A *microscopic weight-loss test* is also useful for relatively insoluble adhesives (Down et al. 2002). A microscopic sample of adhesive is weighed, immersed in solvent, removed from the solvent after a fixed time interval, allowed to dry and then reweighed. The percentage weight change is determined. The higher the percentage of weight loss, the more removable is the adhesive.

After completing a microscopic test, the results can be plotted on the Hansen's solubility parameter sphere or the Teas chart to determine the most appropriate solvent or solvent mixture (see Chapter 2). Before using any solvent technique, remember that:

- **the object must be tested first to make sure it is not affected by the selected solvent or mixture**, and
- rapidly penetrating solvents may move quickly between the adhesive polymer chains and cause rapid swelling that might crack and damage the object (Horie 2010, p. 88).

Solvent removal techniques

Once the appropriate solvent or mixture is selected, the following removal techniques are available, depending on the adhesive accessibility, solvent selected and object requirements:

Vapours

Two ways of using solvent vapours to remove an adhesive or to separate bonds employ a solvent chamber to localize vapours either by using:

- an inverted wide-mouthed bottle (or beaker) with solvent-saturated blotter paper tucked inside, or
- various layers of barrier material placed on the adhesive bond on the object followed by solvent-saturated blotter paper (cotton wipes or Gore-Tex) covered with Mylar and a weight (Navarro 2010).

Solvent gels

Solvent gels can be made with materials such as methylcellulose to which a small amount of solvent is added to produce a slurry, which then forms a gel upon a drop-wise addition of water (Morse 1998). The gel is then applied to the adhesive with or without barrier material, depending on whether the object needs to be protected from direct application of the gel or not. Solvent gels are useful because they can be applied on vertical or problematic surfaces. They may also help to lengthen solvent retention time, control the depth of penetration and minimize human exposure to toxic organic solvents (Stulik et al. 2004).

Poultices

Poultices have also been used with solvents or enzymes to remove adhesives, to separate joins and to remove adhesive stains. One frequently used method is to create a doughnut-shaped ring of dry poultice material around the adhesive to be removed, followed by mixing separate poultice material with the solvent. This mixture is placed in the centre of the doughnut, tamped down and covered with Mylar to ensure slow drying. Use of a barrier material between the poultice and adhesive is sometimes used to protect the object from poultice residue, but this can decrease the effectiveness of the poultice. The amount of solvent added is critical, too much can cause the adhesive to spread into and on the substrate, too little will not dissolve the adhesive. As the solvent evaporates, capillary action draws the solvent and the dissolved adhesive up into the poultice material. To function effectively, the poultice must have strong capillary action; otherwise, the solvent will diffuse into the substrate.

The following guidelines have been suggested by Lemiski (1998) for selecting poultice material: it should not harm the substrate; be easily removable, leaving no residue; absorb the solvent, but allow enough to escape to solvate the adhesive; be non-hazardous to the worker; be easy to handle; and have good working properties.

Table 5 lists some poultice materials that have been used to remove adhesives from textiles. Note that a poultice may not be suitable for some objects (e.g. certain textiles and leathers) because the poultice material might be difficult to remove later (Morse 1998).

Swabbing, brushing or spraying solvent

Swabbing, brushing or spraying solvent onto the bond can help to remove the adhesive layer-by-layer as the top layer forms a gel or swells from the solvent. Also, the solvent could be applied to a backing, which is then peeled away.

Immersion

Immersion of the object in a solvent usually delivers the most solvent to the bond, providing that the object is porous. It can also be the most dangerous technique for the object, so consider it carefully before use. The object must be impervious to the solvent chosen.

Effect of original adhesive solvent and aging on later removal

As a final note on solvents, the quality of the original solvent used to make an adhesive may have an

Table 5. Some poultice materials used to remove adhesives from textiles
(Lemiski 1998; Goetz 1989 p. 98, 1989b p. 641; Heuman and Garland 1987)

Name	Chemistry	Adhesives removed	Comments
sepiolite	hydrated magnesium silicate	shellac cellulose nitrate poly(vinyl acetate) animal glue	works well barrier required
attapulgite	magnesium aluminum silicate	limited use	good absorption of solvents
Laponite	magnesium silicate	shellac starch paste hide glue	works well with water and ethanol non-polar solvents do not work well barrier required not as effective as Sepiolite or paper poultice gives tidelines
methylcellulose	methylcellulose	starch paste	works well
Arbocel 1000	natural leafwood cellulose	ink stains	good absorbing powers easy to handle removal of fibres and residue good

effect on its later removability. In one investigation, films made with "good" solvents took longer to dissolve after light aging than films made with "poor" solvents (Lawrence 1990). Also, it should be mentioned that, as organic materials (e.g. adhesives) age, they become more polar and thus may require more polar solvents to remove them (Blank and Stavroudis 1989).

Enzymes

Enzymes are proteins that may help to remove certain adhesives. They are generally only used when all else fails. They catalyze degradation (removal of the adhesive) without being consumed in the process. Amylase is used to remove starch-based adhesives, while proteases can be used to break down protein adhesives (e.g. hide glue, gelatin) (Decoux 2002; Tse and Guild 1996). Lipase has been used successfully to remove acrylic adhesives (Belluci et al. 1999). Enzymes with the highest purity and activity function best. They must be used at specified concentrations, temperature and pH for optimal effect, or treatment times will be lengthened and residues in the substrate increased (Decoux 2002). They can be applied by brushing, swabbing, immersion or poultice techniques. If the enzyme is not thoroughly rinsed out of the substrate after treatment, discolouration can occur. Caution using enzymes should be exercised on protein-based substrates such as parchment and gelatin emulsion photographs, and other substrates that may contain starch or protein sizing or binders. Enzyme powders should be stored frozen in a desiccated container when not in use to avoid activity loss. For more information on enzymes, Decoux (2002) has written an excellent review of enzymes for paper conservation.

Microemulsions

One of the newer techniques to remove acrylic and vinyl polymers is to use microemulsions and micellar solutions. This technique successfully removed acrylic polymer resins from frescos by the application of microemulsions containing sodium dodecyl sulfate, pentanol, p-xylene, nitrodiluents and water, and vinyl resins using sodium dodecyl sulfate, pentanol, polycarbonate and water (Carretti et al. 2003). These resins were not soluble in normal solvents, using normal removal techniques.

5 A Brief Introduction to Consolidation Issues

by George Wheeler, Columbia University, and
Stefan Michalski, Canadian Conservation Institute

Consolidation is a method of strengthening deteriorated, weak, friable, crumbling or disintegrating objects, such as powdery paint, sugaring stone or deteriorated wood. It is not the purpose of this chapter to give a detailed account of consolidation, but it will review certain issues and define basic terms. The reader is referred to the consolidation literature on specific objects (e.g. stone, wood or paintings) for a more comprehensive discussion.

Consolidants

In general, consolidants should have the following properties: be able to wet and penetrate the substrate, not alter the appearance of the object, allow subsequent treatments to be carried out, and have a long service life (Koob 1981). Often, consolidants are the same organic polymers used as adhesives. In this case, the introduced material both fills voids and binds poorly consolidated fragments (**Figure 1**). In other cases, consolidants undergo chemical reactions upon introduction to the substrate. These reactions can produce solids that fill voids, or that both fill voids and bond to, or react directly with, the substrate. In all applications, a primary goal of consolidation treatments is to achieve full penetration of the deteriorated substrate (Newey et al. 1992). It is also necessary that, once adequate penetration has been reached, the consolidating material remains in place (Newey et al. 1992). Achieving these collective goals may depend on the porosity, permeability and surface properties of the substrate being consolidated; the molecular weight and solubility parameters—and, in some cases, the vapour pressure—of the consolidating material; the solubility parameters and vapour pressure of any solvent employed; and the application technique.

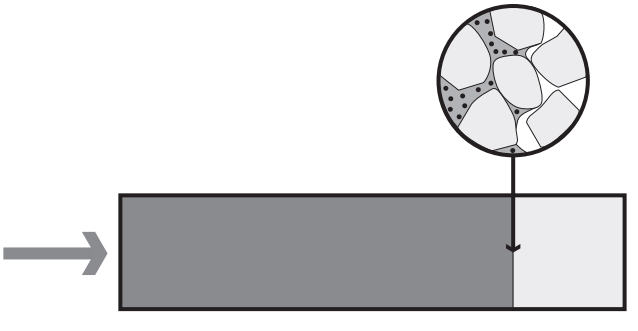


Figure 1. Cross-section of consolidant moving through the object. Left (at arrow) is the surface of the object where consolidant is applied. Circle indicates microscopic view of capillary front.

Achieving penetration

The materials or objects requiring consolidation are described as having "pores", which are the spaces between the particles or fibres that comprise the object (Michalski 2008). The effective porosity of an object refers to those pores that are interconnected and allow flow. Good consolidation requires sufficient effective porosity (Newey et al. 1992). The permeability of a given substrate is a measure of its ability to transmit fluids through its porous network and depends primarily on the size and length of its pores. Porosity and permeability are inherent properties of the substrate and cannot be altered. However, deteriorated objects generally have higher effective porosities and permeabilities that make the object more receptive to consolidation and to obtaining adequate depth of penetration of the consolidant.

Low viscosity is also important for good penetration of the consolidant into an object. One can alter the viscosity of a consolidating solution, which is controlled by molecular weight of the consolidant, choice of solvent, concentration, and, in the case of dispersions, "particle size, presence of stabilizers, emulsifiers, or surfactants" (Koob 1981, p. 86).

Of course, a consolidating system must first be able to wet the target substrate for any penetration to occur. Wettability depends on the adhesive forces between the consolidant solution and substrate overcoming the cohesive forces in the consolidant solution. Thus, wetting is controlled by the selection of the consolidant and its solvent by taking into account the surface properties of the substrate, the solubility parameters of the consolidants and the solvent, and the surface tension of the solvent.

AVOIDING REVERSE MIGRATION

Reverse migration refers to the situation during drying of the consolidated object when solution carrying consolidant flows by capillarity back towards the surface and deposits the consolidant there (**Figure 2**). This is undesirable in most cases, with the exception of perhaps wood with surface deterioration, which might actually benefit from reverse migration. Reverse migration is dependent on the structure and material of the object, solvent selection, solution viscosity and molecular weight, and drying conditions (Domaslowski 1988). The use of in-situ polymerization or precipitation techniques, and surface reactive consolidants can prevent reverse migration.

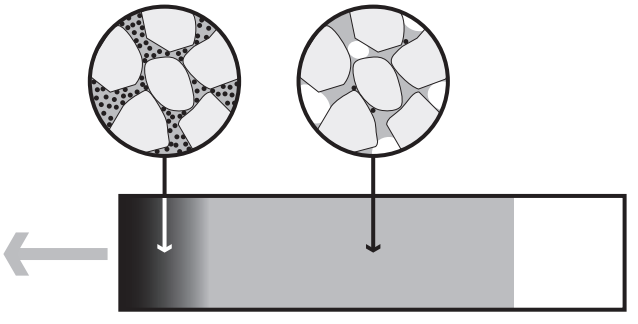


Figure 2. Cross-sectional representation of reverse migration of consolidant during drying. Left is the surface of the object where consolidant accumulates after reverse migration (darkened area). Arrow indicates direction of reverse migration. Circles indicate microscopic view.

SOLVENT SELECTION AND THE CHROMATOGRAPHIC EFFECT

As a consolidant solution advances through a porous material, some solvents will allow a layer of consolidant molecules to attach immediately to the pore walls, while others will not. Solvents that do not deposit this layer are called strongly eluting solvents for that combination of material and consolidant. When using dilute solutions for delicate consolidation of fine powders, weakly eluting solvents can be advantageous since at least some consolidant is deposited over most of the region wetted by solution, and this deposit is not subject to reverse migration during drying (Michalski 2008). Studies of the consolidation of powdery paints suggest that polarity can also be a useful guide to this effect (Michalski and Dignard 1997).

SOLUTION VISCOSITY

If one uses a solution that is viscous, but not so viscous that it cannot penetrate to the depth desired in the time allocated, and if one prevents evaporation until this slow penetration has been achieved, then reverse migration is prevented since the solution becomes too viscous to move after a small amount of drying, and the remainder of the drying can proceed with negligible migration. This technique is not feasible if one is trying to limit colour change, since a large volume of dry consolidant will deposit.

MOLECULAR WEIGHT AND DRYING RATE

With dilute solutions and slow drying, low molecular weight polymers will reverse-migrate less because they can diffuse against the capillary flow to the surface. This has been called reverse-reverse migration (Michalski 2008).

IN-SITU POLYMERIZATION OR PRECIPITATION

An in-situ polymerization technique involves the application of monomers or oligomers that polymerize once in place in the object. Thermosetting resins, such as epoxy resins, have also been used to produce an in-situ polymerization that remains in place after the two-component systems have reacted. Alkoxysilane stone consolidants contain low viscosity monomers and oligomers that polymerize through hydrolysis-condensation reactions initiated by contact with water vapour. Catalyst concentrations are chosen to produce solids in a time frame to prevent reverse migration.

Precipitation techniques involve using one solvent that the monomer is soluble in to carry the monomer into the object to be consolidated and drying the object in vapours of another solvent in which the monomer is insoluble. The second solvent eventually exchanges for the first solvent in the object and forces a precipitation of the monomer (consolidant). The monomer is not pulled out of the object by the second solvent when it evaporates (Domaslowski 1988).

Another technique is to use a solvent that dissolves the monomer but not the created polymer and thus, upon evaporation, does not pull the polymer with it (Phillips 1986). The precipitation technique is useful because high molecular weight polymer can be deposited with deep uniform non-staining penetration, but open pores can be maintained (Phillips 1986). These open pores are important because they allow the object to breathe, expand or contract with thermal changes, and allow possible future removal of the polymer with solvents (Phillips 1986).

Surface reactive consolidants

Consolidation is sometimes achieved by the application of surface reactive solutions. These solutions consume part of the substrate and, in the process, generate a well-adhered and bulky conversion product that can provide some consolidation to fragile substrates. Two common examples are pH-balanced solutions of ammonium tartrate or of ammonium oxalate, in contact with calcite in marble or limestone.

Consolidation techniques

Various consolidation techniques have been used and the choice of method depends on the nature of the object, availability of equipment and amount of consolidation desired or achievable. Methods include application by brush, syringes, tubes, sprayers and misters, impregnation techniques—such as soaking, at times with agitation or vacuum—and gravity feed.

Some of these techniques affect the viscosity of the solution, which affects the ability of the solution to reach the consolidation site. For instance:

- when applying a solution by brush, viscosity of the consolidant increases, if time is long between dipping and applying it;
- syringes and tube applicators can deliver consolidant at the right viscosity but can cause damage at the injection sites (e.g. in paintings);
- sprayers can deliver large quantities of consolidant to the object, but droplets can sometimes be larger than particles being consolidated and can increase in viscosity by the time they reach the object;
- misting surfaces with consolidant can delicately deliver small quantities of consolidant in small droplet size to fragile surfaces (e.g. powdery paint) with similar viscosity to the original solution; and
- when using solvent vapour bags, evaporation of the solvent during application and drying can be reduced, which helps to keep viscosity low as well (Michalski 2008).

One may also wish to control the drying or evaporation process. For good consolidation, slow and long drying is desired, as this allows time for the consolidant to penetrate and set before reverse migration can take place (Domaslowski 1988). Penetration without reverse migration is achieved by using a barrier film or drying in the presence of solvent vapours.

Removability of consolidant

One of the major concerns with consolidating any material is whether the consolidant can be removed from the object without damage. With consolidation, removability is much more problematic than reversing an adhesive bond because the consolidant, if properly applied, permeates the entire deteriorated zone of the object. Complete, safe removal is near impossible, especially as consolidation is usually performed on objects of questionable stability to begin with. A more realistic goal is re-treatability, in other words, that future consolidation treatments are not prevented by the current application. While we may desire a long service life for consolidants, in practice, objects often require additional consolidation even within a few years.

6 Technical and Aging Information on Adhesive Classes

by Jane L. Down, Canadian Conservation Institute

Introduction

This chapter aims to describe the technical properties of the *major* adhesive classes of interest to conservation applications. As well, current and historical conservation studies conducted by researchers to elucidate chemical, physical and aging properties are summarized in order to give an overall picture of known properties of each class of adhesive.

There are many adhesive classes and products that are available now and have been available in the past, but in a book this size it is not possible to explore all of them in detail. The adhesive classes that were chosen for inclusion in this book are listed below and are organized in this chapter in the following way:

Organic - Natural
proteins, polysaccharides, cellulose ethers, cellulose esters, shellac

Organic - Synthetic
poly(vinyl acetates), poly(vinyl alcohols), poly(vinyl butyrals), acrylics, cyanoacrylates, poly(2-ethyl-2-oxazoline), epoxy resins, soluble nylons

Partially Inorganic
silicones (polysiloxanes) and silanes

These adhesives represent ones that are frequently used in conservation applications and ones [shellac, soluble nylon, poly(vinyl alcohol) and cellulose nitrate] that are not necessarily recommended for use now but were used in the past, can be found in collections, and may pose problems. Due to space limitations, little information is provided in this chapter on inorganic materials (e.g. plaster), natural rubber, polyester, polyurethane (PUR), styrene-butadiene rubber (SBR), neoprene or formaldehyde resins because these materials are not used or are little used in conservation either because they oxidize, de-adhere and stain substrates (e.g. rubber); embrittle (e.g. SBR); yellow (e.g. PUR, polyester); contain chlorinated components (e.g. neoprene); or are highly crosslinked and thus insoluble (e.g. formaldehyde resins).

For each adhesive class in this chapter, the opening table gives, for easy reference, what is known about the setting mechanism (e.g. liquid solidification, evaporation, chemical cure), category (e.g. thermoplastic, thermosetting), water absorption, refractive index, glass transition temperature, viscosity, pH and coefficient of linear expansion. Following the table, information is given on abbreviations; types, synonyms and/or trade names; chemistry and/or preparation; history and uses; characteristics, limitations and/or aging studies; and solubility and/or removability. For the characteristics, limitations and/or aging studies section, the information is arranged with the general properties appearing first followed by information on the various subdisciplines in conservation, starting with subdisciplines whose substrates are rather flexible (e.g. paper, textiles), proceeding to subdisciplines whose substrates are semi-flexible, and concluding with subdisciplines whose substrates are more rigid (e.g. stone, fossils).

Adhesive components may vary within a class, so please note that, unless specified, properties mentioned are general for typical adhesives within that class.

Finally, please note that in all cases it is strongly recommended to take the necessary health and safety precautions when using any adhesive or consolidant, especially where solvents are involved. Some adhesives come with special warnings, which are given, if known, under *Health Issues* in the related adhesive class. Nevertheless, one should always carefully read the manufacturer data sheets and material safety data sheets and implement the necessary precautions recommended before use. As a general rule, it is always wise to avoid skin contact with adhesives (e.g. wear appropriate protective gloves) and work in well-ventilated areas (with fume extraction).

Proteins

Setting mechanism	Category	Water absorption (%)	Refractive index
evaporation (liquid types) liquid solidification and evaporation (gel and hot-melt types)		6–8 times its weight, films heated above 100°C become water-resistant	gelatin 1.516–1.534
Glass transition temperature (°C)	**Viscosity (mPa.s)**		**pH**
for glue: 220 (no water present) 45 (at 50% RH) 22 (at 80% RH)	Can depend on molecular weight distribution, concentration, temperature, intermolecular bonds, denaturation and/or if alkaline or acid pre-treated. bone glue – low to medium hide glue – medium to high rabbit skin glue – high mammalian gelatin – medium to high isinglass – highest fish gelatin – medium to high liquid fish glue – high (4000–6000) cold liquid hide glue – high (4000)		glue (general) 5.5–8.0 bone glue 5–7 hide glue 6.5–7.4 rabbit skin glue 5.0–7.5 mammalian gelatin 5.0–6.5 isinglass 6.0–7.5 fish gelatin 3.5–5.0 liquid fish glue 4.0–6.0 cold liquid hide glue 6.5

(Horie 2010, p. 232; Schellman 2007; Paper Conservation Catalog 1994, pp. 42–46; Brandis 1990)

Abbreviation

RSG rabbit skin glue

Types, synonyms and/or trade names

albumin, Alfabond, animal glue, bone glue, Canada Standard, casein, chrome glue, cold hide glue, Comet, deer skin glue, extracted bone glue, Fezandie, fish glue, Fisher, Franklin's, Fredrix, Ganes, gelatin (fish or mammalian), glue, glue-paste, Grumbacher, hide glue, Holbein, isinglass, Knox, LePage, liquid fish glue, liquid hide glue or cold liquid hide glue, mouth glue, mussel adhesive protein, Norland, packers' glue, parchment glue, parchment size, rabbit skin glue (RSG), renderers' glue, Scotch glue, Seccotine, sturgeon glue

Chemistry and/or preparation

The parent substance for animal glue, hide glue, bone glue, RSG, fish glue, isinglass, gelatin or any protein glue is collagen. It is the main protein of connective tissue, skin and bones, and is composed of amino acids linked through peptide bonds in a triple helix structure (Nguyen 2006). *Hide glue* is formed from trimmings of cattle hides or calf, pig or sheep skins (not nails, hoofs, horn or hair), while *bone glue* is made from the bones of cattle and pigs. *RSG* is made from rabbit pelts once the fur has been stripped off (Adams 1980), but may contain other collagenous glue or waste (Schellman 2007). *Fish glue* is made from skins or swim bladders of non-oily fish (sturgeon swim bladders refined as isinglass) (Schellman 2007). *Glue and gelatin* are fundamentally the same material, but gelatin is a more purified and refined (filtered and bleached) version of hide or bone glue, and is generally made from fresher raw materials (Adams 1980). Glue is made in a quicker, cheaper process.

Glue or gelatin is formed by heating the hide, skin, bone, bladders, etc. in water. During this process, collagen, which is insoluble, becomes soluble by denaturation. The denaturation temperature depends on the source. Mammalian collagen denatures at 40–41°C, while fish collagen denatures at lower temperatures of 15–36°C (Schellman 2007). Generally, to produce suitable glue, vigorous processing (e.g. extreme pH, longer time,

higher temperatures) is required for older mammals and a more gentle process for young mammals and fish (Schellman 2007). The highest grades of glue or gelatin are taken from the first extraction and are usually colourless, transparent and amorphous. The medium grades come from further extraction and are generally yellow to light brown. The lowest grades come from the final extractions and are brown.

The processes for making bone glue and hide glue are summarized as follows (Adams 1980; Perry 1944):

- For *bone glue*, the bones are crushed and then degreased to remove fat. The meat and gristle are then removed and the bones are soaked and rinsed in water to remove dirt, blood and impurities. They are then extracted in hot water and steam, and this hot water extract, which is the glue, is drained off. This extraction is repeated several times. The by-products of bone glue go to make bone china and fertilizer. *Packers' glue* is made from green stock (fresher bones); *renderers' glue* is made from bones from butcher shops, restaurants, etc. (less fresh); and *extracted bone glue* is made from all kinds of bones, such as green stock from abattoirs and slightly decomposed bones from shops (Perry 1944).
- For *hide glue*, the hides are washed in cold water to remove dirt, blood, etc. They are put in a lime water soak for several months, which removes the hair, and then rinsed and neutralized to remove the lime. They are then extracted in hot water, and this extract, which is the glue, is drained off. As with bone glue, the extraction is repeated several times. If the hide is chrome-tanned, then salts are removed and the glue made from it is called *chrome glue* (Adams 1980).
- After this, the extracts for both bone and hide glues are treated the same. They are filtered, combined with preservative, and sometimes bleached, and then the water is evaporated as they are made into cakes, pearls, grains or powders. *Scotch glue* is an old-fashioned name for any glue made in a thick cake (Adams 1980).

Liquid hide glues contain additives (a gel depressant, e.g. thiourea) to keep them liquid at room temperature, to prevent microbial growth, and to reduce moisture sensitivity of the dry films (Schellman 2007). Schellman (2007) states that most animal glues contain a preservative (e.g. sulfur dioxide), but may also contain other additives. She suggests that the "safest option for conservation" is glue that has been "minimally modified" (p. 56).

Old texts in the 14th to 18th centuries describe a *mouth glue*, which was made from "proteinaceous glue (mainly sturgeon or parchment glue), gum tragacanth, sugar and water" (Beentjes 2005, p. 13). Mouth glue was activated by placing a piece of the glue in the mouth until softened and then rubbing it on the parchment to be repaired. The sugar sweetened the glue, increased wetting and softened it.

Casein glue is derived from the protein in milk, while *albumin* is derived from mammalian serum (blood) or egg white.

Another type of proteinaceous glue is called *mussel adhesive protein*, which is secreted by the glands in the feet of mussels. This glue contains lysine (up to 20%) and hydroxy-containing amino acids (up to 50%), and has a molecular weight of approximately 130,000 (Sigma-Aldrich).

Identification

Schultz and Petersen (2011) applied an antibody technique called indirect enzyme-linked immunosorbent assay to the identification of proteinaceous binding media and adhesives. They showed that this technique can identify sturgeon collagen from other fish collagen, and distinguish between ovalbumin, casein and animal glue collagen.

Another technique for protein identification is peptide mass fingerprinting, which has been used to distinguish species origin of animal glues (Kirby et al. 2011). This technique requires a protein extraction and digestion protocol followed by a matrix-assisted laser desorption ionization (MALDI) mass spectroscopy analysis. Ions in standards are used for the identification.

History and uses

The origin of animal glue is unknown, but probably goes back long before recorded history (Adams 1980). The first recorded example of animal glue use, veneering, can be seen in Egyptian paintings during the reign of Thothmes III (1479–1425 BC) in the tomb of Rekh-mi-re, as can be seen on the cover of this book (Gardner Wilkinson 1854). It is likely that animal glue was in constant use in ancient Egypt. Animal and fish glues were used by the Romans and Greeks in the 1st century and by the Chinese in the 7th to 9th centuries (Petrohova 2000; Adams 1980). Mouth glue was used in the 14th to 18th centuries for paper and parchment (Beentjes 2005). Bone glue is a more modern development (Adams 1980). The industrial manufacture of glue in Europe started in the 17th century and in England in the 18th century (Adams 1980). In America, the manufacture of animal glue started in 1808 (Gostling 1989). Since the

1950s, production of animal glue has declined and modern-made animal glues may not be of the same quality or strength as earlier ones (Brandis 1990; Gostling 1989).

Glues and/or gelatins have been used for furniture and woodworking (Hurd 1959, pp. 19–20), gummed tapes (Hurd 1959, pp. 19–20), sizing and coatings for textile and paper (Sweatt 1945), binders and sizing of traditional East Asian inks and paintings (Winter 1984), bookbinding (Hurd 1959, pp. 19–20), photographic emulsions (Von Endt 1984) and various food stuffs. Fish glues adhere well to glass, ceramics, metal, wood, cork, paper and leather (Norland 1977).

In conservation, glue has been used for furniture, facing paintings, consolidation of friable paint and ground, and in glue-paste linings for paintings. Gelatin has been used to consolidate ivory, bone, wood, shale, pottery and porcelain (Horie 2010, p. 234). Gelatin and parchment size have been used as consolidants, fixatives and sizes for parchment (Paper Conservation Catalog 1994, pp. 42–46). Gelatin has also been used in geology and palaeontology collections as a surface adhesive, consolidant and label adhesive (Elder et al. 1997).

Sturgeon glue, which is now increasingly difficult to obtain and expensive due to the decline in fish stock, was used extensively in the former Soviet Union and many Western countries for the consolidation of flaking paint and has been used for the lining of paintings on paper and canvas (Rickman and McAusland 1988). Fish glue has been used in painting media, coatings, chalk grounds, gilding of illuminated manuscripts and pastel fixatives (Petukhova 2000).

Casein has been used as a pigment and ground binder (Von Endt 1984). Albumin has been used to manufacture plywood and to suspend silver halide in the coating of photographic prints (Von Endt 1984).

Characteristics, limitations and/or aging studies

General

Glues made as grains or powders are cheaper to make than glues in cakes, are easier to handle, and require less soaking when made up (Adams 1980). Some advantages of animal glues are that they will "give" in the join, afford the ability to correct blisters, and are non-staining, non-toxic, easy to clean up, durable and strong (Adams 1980). When dry, glues can be sanded and have excellent creep, solvent and shock resistance, and rigidity, but may become brittle with age (Young 1999). Generally, hide glues are stronger than bone glues because the process to make hide glues is gentler (lower extraction temperature) (Adams 1980). Hide glues usually exceed the strength of wood and fibrous substrates (Shields 1984, p. 37). High strength bonds are obtained under dry conditions and can be structural as long as they remain indoors (Shields 1984, p. 37).

An early study by Rosser (1939) examined various woodworking animal glues and suggested the following properties:

- gelatin from bone has different viscosity and jelly strength than gelatin from hides;
- animal glue breaks down when temperature rises above 60°C and continued heating causes glue to become very brittle;
- cold-water soaking before heating is best for making glue, otherwise, clumping occurs, requiring higher temperatures for longer times to bring into solution;
- joint starvation resulting in low bond strengths occurs if glue penetrates too much;
- mechanical adhesion is important for joint strength, but glue concentration must be adequate to ensure no shrinkage;
- pressure and clamping are advantageous for good bond strength and to demobilize the join while strength develops; and
- alkaline glue favours bacterial growth while slightly acidic glue does not.

Schellman (2007) has written an excellent review on the properties of animal and fish glues, which is summarized here:

- *Gel temperature* of gelatin depends on the collagen source. For example, mammalian gelatin gels at 30–35°C, while cold water fish gelatin gels at approximately 8°C.
- *Gel strength* or *Bloom strength* [gel rigidity or force necessary to depress a gel at a certain concentration and temperature to a certain depth by a standard plunger (Rousselot)] depends on the average molecular weight and the amount of denaturation (molecular weight decreases when there is vigorous pre-treatment and prolonged excessive heat). The molecular weights of various glues and gelatins are given in **Table 1**. Bloom strength correlates directly with bond strength, water-sorption capacity, viscosity and gelling temperature. Gel strength can vary from 500 g for strong hide glues to 30 g for weak bone glues, while fish glues are even lower. Open time is dependent on gel temperature and gel strength, while drying time is dependent on ambient temperature and relative humidity (RH). Slow drying allows the molecular structure to order

Table 1. Molecular weights of various glues and gelatins
(Schellman 2007)

Glue or gelatin	Molecular weight
bone	low–medium
hide	high
mammalian gelatin	11,000–168,000
rabbit skin glue	high
isinglass	150,000–300,000
liquid fish	60,000
fish gelatin	96,000–196,000

better and thus increases strength. Glue with higher Bloom strength develops tack faster than glue with lower Bloom strength.

- Glue solution *viscosity* is dependent on molecular weight distribution, with higher molecular weight giving higher viscosity. Isinglass, which has high viscosity and molecular weight, has a lower gel temperature than mammalian glues that have similar high molecular weight but that gel more quickly. This lower gel temperature of isinglass allows more time for penetration at room temperature when consolidating than mammalian glues afford. If water will damage a substrate, it is best to use glue with lower gel strength when lower viscosity is desired rather than diluting a glue with higher gel strength because less water can be used.
- The *pH* of glues and gelatins varies and is important for the substrate and for the glue or gelatin. When the pH of the glue or gelatin shifts from its isoelectric point—which is the point at which charges within the molecule are balanced—viscosity, gel temperature, water absorption and swelling increase, while gel strength decreases. Acids and bases can uncoil the helical collagen molecule and thus increase viscosity, while salts neutralize the charge and decrease viscosity (Von Endt 1984).
- *Mechanical strength* of dry films increases with gel strength. The tensile strength of hide glue is approximately 39 MPa, and cold water fish glues and bone gelatin about 22 MPa. RSG, although having a high gel strength, has a lower cohesion and bonding strength than hide glue, likely due to its high fat content. Stiffness is dependent on the molecular weight and other factors, with mammalian gelatin demonstrating higher modulus and greater stiffness than fish gelatin. Excessive dehydration leads to insolubility in water, brittleness and poor impact strength. Additives (e.g. sugar, alcohols, fats and polysaccharides) are often added to increase elasticity and toughness. Creep can occur when there is high water content.
- As far as *environmental conditions* are concerned, hot hide glues are not as sensitive to fluctuating RH and temperature as cold liquid hide glues, and liquid fish glues are even less stable. RSG has better stability to fluctuating conditions, likely due to the fat content. Hardness and water resistance is improved with the addition of tanning agents.
- The *colour* of the glue or gelatin is affected by Bloom. The higher the Bloom, the less yellow the gelatin, and gelatin and isinglass have shown no discolouration during aging studies.

Sionkowska et al. (2009) have suggested that the mixing of animal glues with poly(vinyl acetate) (PVAC) dispersions should be carefully considered because the addition of collagen to poly(vinyl alcohol) (PVAL), a frequently used protective colloid for PVAC dispersions, enhances PVAL photooxidation when irradiated with ultraviolet (UV) light. The collagen contains chromophores that absorb UV and energy, which is transferred to the PVAL molecule.

Chrome glue is very pale, and has low odour and gel strength (Bloom strength) (Adams 1980).

Fish glues are generally liquid at room temperature and have high tack, but when dry they are not gummy, can be sanded easily, and have good strength, rigidity, heat and creep resistance, but may become brittle with age (Young 1999; Norland 1977).

Glue made from pike and carp fish bladders has been suggested in a study by Geissinger and Krekel (2009) as a substitute for glue made from sturgeon bladders, which are increasingly difficult to obtain because of declining fish stock. Pike and carp are more plentiful and easier to obtain. This study indicated that sturgeon bladders gave the best product yield (95.5–98%) in comparison to carp (62–81%) and pike (31–60%), and that carp and pike could give a more brittle film than the sturgeon glue. The study also looked at viscosity, gluing power and aging, and concluded that carp might be a good substitute for sturgeon glue.

Casein glues are generally alkaline and their films are insoluble (Horie 2010, p. 234). They cure to a hard finish, can be sanded, have good strength, rigidity, durability and creep resistance, but may dull tools in woodworking applications, stain high-tannin woods such as oak and mahogany, and suffer bio-degradation (Young 1999). High grades have good moisture and solvent resistance,

while low grades do not have good moisture resistance (Young 1999).

Mussel adhesive protein glue is similar in strength to cyanoacrylates and epoxy resins. It is non-toxic, biocompatible and waterproof, and it attaches to many surfaces (e.g. glass, metal, biological tissues, plastics, Teflon) (Sigma-Aldrich).

MICROBIAL RESISTANCE

As gelatin and glue solutions are susceptible to microbial or fungal attack, they should be refrigerated when not in use and the dry material should be stored in a clean, cool, dry place (Nguyen 2006). In the late 1980s in the former Soviet Union, *ortho*-phenylphenate and pentachlorophenolate were used as fungicides for sturgeon glue, but pigment stability was affected (Rickman and McAusland 1988). Nypagin, an ammonia salt, was used instead with presumably better compatibility with pigments. In the West, fungicides like thymol have been used to inhibit spoilage and fungal growth in stored solutions. However, current practice points to the reduction of fungicide use and instead preparing only enough quantities for short-term use.

FUMIGATION OF ANIMAL GLUES

Fumigation with methyl bromide (MB), methyl iodide, ethylene oxide (EO), MB+EO, propylene oxide or sulfuryl fluoride, and treatment with heat (30°C and 60°C), low oxygen or carbon dioxide did not significantly affect animal glue (Kigawa et al. 2011). This is probably due to the harsh preparation of these glues (using heat, water and chemical reagents), so they are already denatured when used. However, residual bromine and iodine were detected in the samples even after five years.

PAPER

An early thermal aging study by Baer et al. (1976) of various glues that were considered for potential use in paper conservation [hide glue (Ganes Flexible Glue); rubber-based glue (Best Test Paper); dextrin (Yes Stikflat); vegetable glue (849 Sta-flat)] examined fold endurance, reflectance, pH and colour change, and found that none could be recommended. The hide glue showed small changes in reflectance and colour, but large changes in strength and pH.

Various starches, gelatin and cellulose ethers (Klucels) were tested for colour change, surface drying time, ability to form dry films of constant thickness, shrinkage, flexibility, hardness, pH, bond strength and reversibility upon heat and light aging for use in prints and drawings (Shashoua et al. 1990). Gelatin was fairly acidic and showed the same reversibility results as wheat starch; however, its flexibility was better and it was the strongest of all the adhesives tested on paper samples. The cellulose ethers were found to be the most suitable consolidants and wheat starch the most stable adhesive.

Glues have been used as the traditional binding media for pigments and as a sizing for East Asian and Chinese paintings and documents (Winter 1984). Often, alum was added to the size to make the glue harder and less soluble (Winter 1984). Hydrolysis of alum to generate acid would have been partially buffered by the paper's alkaline reserve and the glue, which is amphoteric (Winter 1984).

Cannon (2011) has suggested three reaction pathways that could create insoluble complexes and cause insolubility of natural adhesives (e.g. proteins or starches) on paper. These are the presence of multivalent metal ions, such as copper and iron, added to old adhesives for hardening or preservative purposes; the presence of aldehydes formed during cellulose oxidation upon aging; and condensation reactions of proteins with carbohydrates (Maillard reaction).

BOOKS

Animal glues have been used to bind books for centuries. Bookbinder recipes have, by tradition, been closely guarded secrets and may include additives such as sorbitol, preservatives, defoamers, perfume, plasticizers, thickeners, extenders, ammonium sulfite, alginate, yellow resin, asphaltic emulsion, linseed oil, sugar, methylated spirits, glycerin and ammonia (Gostling 1989).

In an early study by Belaya (1958), several natural glues [wheat starch paste (WSP), dextrin, gelatin, bone glue, sturgeon glue, ethylcellulose] and synthetic adhesives [poly(methyl acrylate), PVAC, PVAL, poly(vinyl butyral), perchlorovinyl glue] were compared (before and after heat and light aging) on leather bindings for layer separation, adhesive power, strengthening and hardness. Sturgeon glue was strong, elastic and unchanged upon aging, but susceptible to bacteria, mould and insect attack; however, it was the most suitable natural glue. The synthetics had varying results, but it was the ethylcellulose with plasticizer that was chosen.

WAX

In an experiment that investigated various adhesives for wax objects using tensile strength and bend testing, isinglass gave acceptable results for

beeswax but not for paraffin or Japan wax (Lang 2011). This was also demonstrated in another study that identified isinglass as a good choice for wax substrates, being of sufficient adhesive strength and quite removable (Fischer and Eska 2011).

Paintings

When a study of the force that can develop in various components of fabric-supported oil paintings as a function of RH was conducted, RSG showed a dramatic increase in stress under desiccation (80→10% RH) when compared to the paint or fabric and is probably the cause of damage in desiccated oil paintings (Mecklenburg 1988). RSG lost 35% of its dry weight when desiccated, contracted by about 3.6%, and developed stress in a restrained sample. In further work, the moisture coefficient of expansion (tendency to shrink or expand) for RSG was lowest at 55% RH, and greatest below 40% RH and above 75% RH. When all easel painting components were taken into account, it was suggested that the best RH for the painting was around 70% RH (Mecklenburg et al. 1998; Ballard 1995; Mecklenburg et al. 1995).

In another study by Karpowicz (1990), the development of cracks in RSG sized and gessoed samples with and without fabric was followed. Different crack patterns were identified during drying and humidification, which depended on the tension of the sample, the humidity range investigated and the length of time in the various humidities.

A study of various RSGs (Holbein, Fredrix, Grumbacher), gelatins (Fezandie, Fisher, Knox) and isinglass (American, Russian, Finnish) in solution and dry films showed that RSG solutions were acidic, had the lowest surface tension of all samples and that some viscosities were increased if the pH was neutralized (Haupt and Dyer 1990; Haupt et al. 1990). The RSG films were more extensible than gelatin films when placed in high RH conditions. The gelatin solutions were acidic, had the highest surface tensions and most viscosities were increased with neutralization. The gelatin films were strong in high RH. The isinglass solutions were near neutral and were the least viscous. Films were brittle under ambient conditions, but were very extensible under high RH and showed the greatest response to changes in RH. In general, neutralizing all the solutions caused a small increase in extensibility of films, while heating the solutions for an extended period caused a larger increase in extensibility. All the glues depressed the surface tension of water.

Glue-paste linings of paintings have been used since the 1600s, but fell out of favour by the end of the 19th century because of the adverse effects of moisture on the canvas, causing shrinkage and loss of paint and ground (Ackroyd 1995). A modern study on the mechanical properties of these linings was conducted using recipes of wheat starch/gelatin, wheat flour/sturgeon glue and wheat flour/bone glue at various ratios (Ackroyd 1995). Generally, peel strength increased with increasing amounts of glue, and the wheat starch/gelatin mixture gave the highest peel strengths. The least amount of gelatin in this mix gave good results (wheat starch/gelatin 8:1) because it afforded better dimensional stability of the lining.

The use of hide glue as a lining adhesive or consolidant is problematic because of its dramatic response to low humidity (Mecklenburg and Fuster Lopez 2008). In experiments on the effect of RH cycling on various paintings, glue-paste lined paintings, either still lined or ones with glue-paste lining removed mechanically, showed very large tensions in dry conditions, likely due to the glue-paste regenerating or reforming during the elevated humidity period of the cycle (Hedley 1988).

Animal glues should not be used in tropical climates to size, mount or line canvases because they can be attacked by micro-organisms and can cause damage from swelling or contracting if the environment is not controlled (Boustead 1968). Mould has been found on easel paintings from Mediterranean countries, which were lined with glue-paste adhesives (Bouillon and Fohrer 2008). The coarse sifted wheat used to make these glue-paste adhesives was more vulnerable, and the use of Venice turpentine in the glue-paste reduced the problem.

Demuth et al. (2011) have examined many different types of adhesives for thread-by-thread tear repair for canvas paintings. They found that a mixture of sturgeon glue with WSP gave relatively high ultimate tensile strength compared to the others tested, higher than mixtures of sturgeon glue with *junfunori*, Tylose MH 1000 or Aquazol 500. The sturgeon glue/WSP mixture also had other redeeming properties such as neutral pH, reversibility in warm water, good workability and good optical appearance (almost invisible). Pre-cooked starches (ready-made) were also found to give good results when mixed with the sturgeon glue, which would afford quicker preparation.

Fluorescent labelling has been used to show that gelatin penetrated painting canvas undesirably when used as a consolidant for flaking paint, while methylcellulose (MC) and a mixture

of MC and gelatin formed the appropriate adhesive layers between paint and canvas (Soppa et al. 2011).

In an experiment examining the consolidation of paintings using porous gypsum/micronized silica blocks and sticks with various dilute adhesives (hide glue, sturgeon glue, Aquazol 200, Plexisol P550, Paraloid F-10, BEVA 371), concentrations of 4–6% were optimum at 40°C for the hide glue and similar results were found for sturgeon glue, but at lower temperatures, which was an advantage (Buzzegoli et al. 2008). The sturgeon glue showed greater penetration than the hide glue at the same concentrations.

When the mechanical behaviour of various adhesives (hide glue, PVAC, epoxy, urea-formaldehyde) with various fillers (rye flour, wood flour, cellulose powder, coconut flour, microballoons) was examined for re-joining panel paintings, hide glue with wood flour and the PVAC with rye flour or with a low proportion of microballoons were found to be weaker than wood and thus may be better suited for use with degraded wood, if the panel is not subject to large RH changes (Young et al. 2002).

DECORATIVE ART

Schellmann and Taylor (2011) found that isinglass and hide glue solutions were the most suitable adhesive for restoring fracture properties of protein-bound foundation layers in gesso-type decorative coatings, as these were the most effective adhesives and penetrants compared to others tested (Paraloid B-72, Paraloid B-48N, Lascaux Medium for Consolidation, Mowiol 3-83, Mowilith 50 and Mowilith DMC2). Adding starch to the isinglass increased the change in fracture energy (toughening).

When gesso (RSG and calcium carbonate), typically used for gilding, was investigated, the ultimate strength of the gesso was found to be lower than the RSG and inversely proportional to the chalk to glue ratio (Mecklenburg 1991). This study also found that stiffness increased with increasing chalk to a ratio of 13.3 and then decreased. RSG can change dimensionally by about 6% in a single RH cycle (5→93%), however, the dimensional change of gesso is considerably less. Repeated cycling of unrestrained samples resulted in permanent shrinkage while permanent change in stress development during RH cycling occurred when samples were restrained. The study also found that a substrate with less dimensional response to RH would provide some partial restraint, and cracking and failure of the gesso could happen under severe RH changes.

WOOD

Three studies on wood (Buck 1990; Hay 1987; Down and Lafontaine 1980) examined certain liquid hide glues and hot-melt hide glues under various RH cycling and agings. Generally, the studies agreed that liquid hide glue was more prone to decrease in strength after RH cycling or aging than a hot-melt hide glue, suggesting that liquid hide glues only be used under normal conditions, while if extreme conditions of heat and high humidity are expected, hot hide glue would be better. Notwithstanding, extreme humidity and heat are not recommended for either glue.

Werner (1968) has said that animal glues and other natural materials such as beeswax, natural resins and shellac may not be satisfactory for use on wood in tropical climates because they are so hygroscopic.

SOLUBILITY AND/OR REMOVABILITY*

Glues swell in cold water and become soluble upon heating. Aqueous glue solutions form a reversible gel upon cooling, which is a useful property for removing glue (Brandis 1990). Most glues and gelatins swell and redissolve even after many centuries (Schellman 2007). The lower the original concentration of glue, the lower the re-solubility of the dry film will be. Bone glues are more re-soluble than hide glues and re-solubility of any dried collagen-based glue can be changed by metal ions, organic pigments, tannins, crosslinking and impurities (Schellman 2007; Paper Conservation Catalog 1994, p. 44).

Glues are insoluble in oils, waxes, organic solvents and absolute alcohol (Brandis 1990). Gelatin is soluble in glycerol and acetic acid, and can be diluted with ethanol and isopropanol (Paper Conservation Catalog 1994, pp. 42–46). Organic solvents will not dissolve dried fish glue, but the liquid fish glue will tolerate some water-miscible solvents such as ethanol and acetone (Norland 1977).

Enzymes have been used to remove insoluble dried glues (Paper Conservation Catalog 1994, p. 44).

* Disclaimer: The solvents listed in this section are ones that might dissolve the polymer or adhesive in question but are not necessarily recommended for conservation treatments. Some may be extremely toxic or may contain elements (e.g. chlorine) that may be harmful to certain materials in your object (artifact). Research the solvent before purchase or use to know its toxicity and chemical nature. It is strongly recommended to test all solvents before use to make sure that they do not adversely affect your object.

New wood glue joins (mortice and tenon, edge-to-edge, veneer) made with different strengths of animal glue were reversed successfully using radio frequency heating, which works on polar water molecules similar to dielectric heating in microwave ovens (Neher 1996). Some moisture at the glue line is needed, but much less than if the object was soaked in water. In all cases, the joins were separated with no damage to the wood. The technique has yet to be evaluated on old animal glue joins.

Current practice to reverse glue-paste linings involves mechanically removing the old lining and glue-paste with or without a small amount of water or aqueous gel. This mechanical removal can pose a risk of damage to the original canvas, so a non-contact laser technique was investigated as an alternative (Spencer et al. 1999). It found that glue-paste lining adhesive was successfully removed from canvas using a UV 248 nm excimer laser. A high fluence regime first removed the bulk of the adhesive followed by a low fluence that was "high enough to ablate the glue slowly but is too low to cause damage to the underlying canvas" (p. 340). This technique showed significant improvement on the traditional method of mechanical removal, as there was less damage to the canvas fibres.

Polysaccharides

Setting mechanism	Category	Water absorption (%)	Refractive index
evaporation			1.53
Glass transition temperature (°C)	**Viscosity (mPa.s)**	**pH**	**Coefficient of linear expansion /°C**
		wheat starch 6.3–6.9 rice starch 7.5 arrowroot starch 7.1	

(Down and Burgess 1990; Roff and Scott 1971, p. 184)

Abbreviation

WSP wheat starch paste

Types, synonyms and/or trade names

agar, Aytex, British gum, dextrin, *funori*, *furunori*, gum arabic, *junfunori, shinnori*, *shofu*, starch (arrowroot, corn, green bean, potato, rice, rye, tapioca, wheat)

Chemistry and/or preparation

Polysaccharides are polymeric carbohydrate structures, formed by repeating units (either mono- or di-saccharides) joined together by glycosidic bonds (Horie 2010, pp. 221–222; Roff and Scott 1971, p. 182). Essentially, they are based on sugar monomers. Starch is an example of a storage polysaccharide, meaning that it is a reserve carbohydrate stored by a plant (Roff and Scott 1971, p. 183). The seeds, roots, bulbs, sap, fruit and leaves of the plant contain the starch that is extracted by grinding, filtering, washing, centrifuging and drying (Masschelein-Kleiner 1995, pp. 51–54; Roff and Scott 1971, p. 183).

To make starch paste, starch is placed in water and heated. It is insoluble in cold water but when heated to its gelatinization temperature (**Table 1**) the starch granules swell (Horie 2010, p. 222). These swollen granules start to erupt, releasing starch molecules, which increases viscosity. Constant stirring and heating at this temperature helps eruption and dispersion of contents, which eventually forms a smooth paste. Upon cooling, "retrogradation" occurs with the helical amylose molecules lining up and attempting to recrystallize due to hydrogen bonding, and the mixture forms a gel (Daniels 1995b).

Starch works as an adhesive because it contains numerous hydroxyl groups that form hydrogen bonds with other functional groups on the substrate (Tímár-Balázsy and Eastop 1998, pp. 316–317). Natural starch consists of two types of polyglucosides: amylose and amylopectin (**Figure 1**). They exist in different proportions depending on the origin of

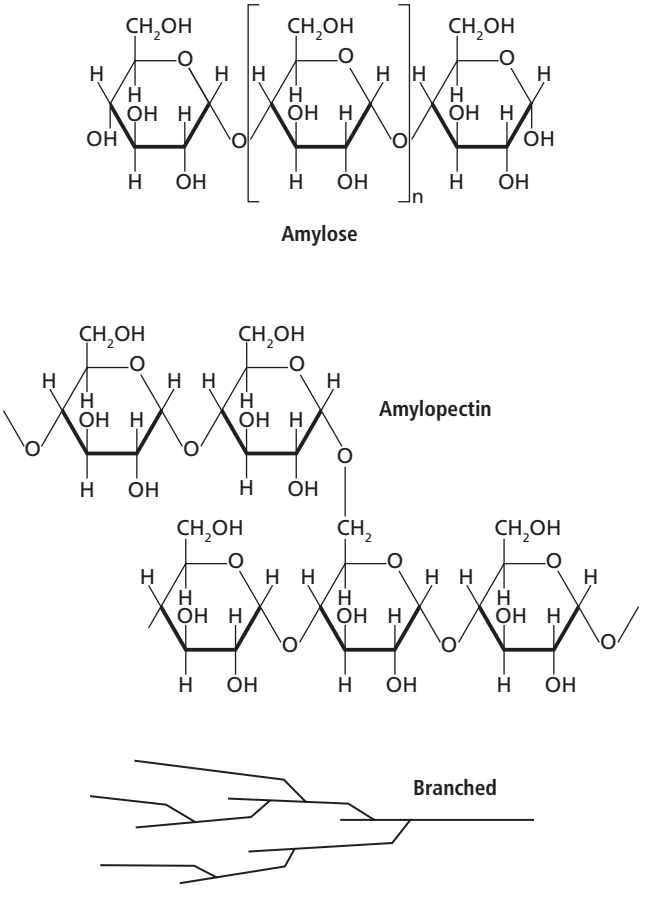


Figure 1. Amylose and amylopectin. (Horie 2010, p. 222; Whistler et al. 1984, pp. 160–168; Davidson 1980, p. 22–7).

Table 1. Properties of various starches
(Horie 2010, pp. 221–228; Tímár-Balázsy and Eastop 1998, pp. 316–317; Masschelein-Kleiner 1995, pp. 51–54; Kruger and Lacourse 1990; Van Steene and Masschelein-Kleiner 1980)

Starch type	Source	Amylose %	Gelatinization temperature (°C)	Gel formation upon cooling	Clarity and colour of cooked starch	Flexibility	Adhesion	Degradation
wheat	seed	20–25	58–64	strong	opaque, white	poor	good	poor
rice	seed	16–19	68–78	little	light beige	poor	poor	good
corn	seed	22–28	62–72	strong	opaque, light green	good	good	poor
rye	seed	~27						
green bean	seed	37–70						
tapioca	root	20	49–70	little	quite clear, white	good	very good	very good
potato	root	25	59–68	little	very clear, white	good	very good	poor
arrowroot	root	23	69–76		light beige	poor	good	good

the starch (e.g. wheat, corn, rice, potato, tapioca, arrowroot; see **Table 1**) (Roff and Scott 1971, p. 182). Amylose is a linear polymer that forms strong water-resistant films (Kruger and Lacourse 1990). High temperatures are needed to dissolve amylose in water and high amylose content causes the solution to gel upon cooling (Horie 2010, pp. 222–223). Amylopectin is a branched polymer, a much larger molecule than amylose. It forms weak water-sensitive films and can be dissolved in cold water (Kruger and Lacourse 1990). Starches from different sources vary in molecular weight and degree of branching of the amylopectin (Daniels 1995b). Also, starches with very high amylopectin content retrograde slowly, while ones with high amylose content retrograde faster (Daniels 1995b).

In Japan, wheat starch is extracted from seeds similar to the Western way as described above, except that the centrifugation and drying steps are eliminated. Instead, the wet starch is poured into vats and allowed to settle naturally into layers with the purest wheat starch (*jinnori*) sinking to the bottom. This is the layer that is withdrawn, stored under water and later cooked to produce a wheat starch paste (WSP) called *shinnori* (Paper Conservation Catalog 1994, pp. 4–12; Wills 1984).

Falling under the polysaccharide umbrella are *funori* and *junfunori* (Michel 2011; Horie 2010, p. 228). *Funori* is made from red algae seaweed *Gloiopeltis*, which is found in the coastal regions of Japan, southern China and the north Pacific coast of the United States. It is prepared from the seaweed in various ways, but in general it is dried, washed in water, bleached (traditionally, bleaching and softening is done through a fermentation process), rinsed, and laid on a screen to dry in the sun. When used, the dried material is cut up, soaked in water overnight, heated (just below boiling or 45–60°C) and filtered through a cloth (Michel et al. 2002; Masuda 1984). It is then diluted with warm water and sometimes isopropanol (5%), and applied warm. *Junfunori* is a more purified version of *funori*.

Modifying starch

Starches can also be modified in the following ways:

Roasting

Starch can be roasted (180–250°C) with or without a basic catalyst, which gives a yellow- or canary-yellow-coloured dextrin of lower molecular weight than natural starch (Roff and Scott 1971, p. 186). Light-coloured dextrins are possible if treated with dilute mineral acid and roasted at 100°C first (Roff and Scott 1971, p. 186). These modifications can lead to complete solubility in cold water. Potato and tapioca starch convert to

dextrins more easily than wheat, corn or sago starch (Masschelein-Kleiner 1995, pp. 51–54; Mantell 1970). British gum is a dextrin and differs from other dextrins in that it gives higher viscosity dispersions, is darker and is usually produced from starch mixtures rather than a single starch (Mantell 1970).

Aging

Japanese WSP, *furunori,* is *shinnori* paste that has been aged under water for 10 years in sealed earthenware or plastic jars (Wills 1984; Masuda 1984). This paste is less viscous, more stable, and displays a weaker more flexible bond than the regular *shinnori* paste (Tímár-Balázsy and Eastop 1998, pp. 316–317). However, it was shown to biodeteriorate and become mildly acidic, depolymerize and retrograde, although the ratio of amylose to amylopectin remained the same (Daniels 1995b). Starch paste of similar quality has been obtained by acid hydrolysis for several hours (see next section) (Van Steene and Masschelein-Kleiner 1980). Preparation of *furunori* in Western studios is difficult for several reasons (Belard et al. 2009). First, Western labs must use *kanosjin* as the starting material rather than *namajin*, which is difficult to import and spoils. Second, higher heat to make the paste is usually necessary. Finally, the storage containers and storage temperatures are not the same as used in Japan. In Western studios, airtight containers are used and these discourage the right bacteria from growing and the paste from fermenting. Also, the storage temperature in Western labs is usually higher than Japanese labs. These issues prevent a good production of the paste in Western labs.

Acid-hydrolyzed starch

When granular starch is heated below its gelatinization temperature in an acidic environment, the resulting product has reduced viscosity and improved flexibility and adhesiveness (Van Steene and Masschelein-Kleiner 1980). These properties can be changed by length of heating and temperature, acid or starch used (Singh and Ali 2000; Van Steene and Masschelein-Kleiner 1980). A suggested recipe for acid-hydrolyzation is to heat (55°C) and stir wheat starch in distilled water and hydrochloric acid (Van Steene and Masschelein-Kleiner 1980). After a period of time, the surface liquid is decanted, the starch is washed a few times, a preservative is sometimes added, the pH is adjusted to neutral and the starch is heated (90°C).

Other

Starches can also be hydrolyzed with enzymes (e.g. amylase), oxidized (e.g. with sodium hypochloride), etherified or esterified, or modified with additives to change various properties (e.g. tack, solubility, viscosity, flexibility, drying time), but these modifications are little used in conservation.

Starch paste recipes

A typical recipe for making WSP is to gradually add distilled water (50 mL) to starch powder (30 g), stirring constantly until smooth (CCI 1993). This is covered and left to stand. Distilled water (250 mL) is poured into a beaker, which is set in a pan of water and allowed to come to a boil. The starch/water mixture is gradually poured into the boiling water in the beaker with constant stirring. Heating is continued for another 5 to 10 minutes. The beaker is then removed and allowed to cool. The paste is often thinned at this point with distilled water, vigorous stirring and sieving. A microwave recipe uses starch powder (100 g) that is soaked for one hour in water (500 mL) and then cooked for 3 minutes in a microwave with vigorous intermittent stirring (IPC 1989). Supposedly, microwaved paste does not need sieving, but many conservators sieve or blend as required.

Rice starch paste can be made by creaming rice starch (30 g) with water (30 mL) to which boiling water (600 mL) is added with constant stirring, followed by simmering in a double-boiler for one hour (Horie 2010, p. 226). Another recipe calls for a slurry of rice starch (50 g) with water (350 mL) in a saucepan. This mixture is heated on a stove for 10 to 15 minutes at the lowest setting until warm, then heated at the high setting with constant stirring for about 2 to 3 minutes, then allowed to cool and then sieved and diluted as necessary (Kohler 1981).

Many other paste recipes are given by Morrison (1980).

Identification of starch

Starch can be identified by light microscopy using polarizing filters. By the amount of birefringence, it is possible to detect uncooked starch granules from partially cooked paste from fully cooked paste (**Figure 2**) (Yahl 1983). An extinction cross image is a typical indicator for uncooked and partially swollen starch. Starch can also be detected using an iodine solution, with amylose giving a blue colour and amylopectin giving a red/violet colour (Daniels 1995b).

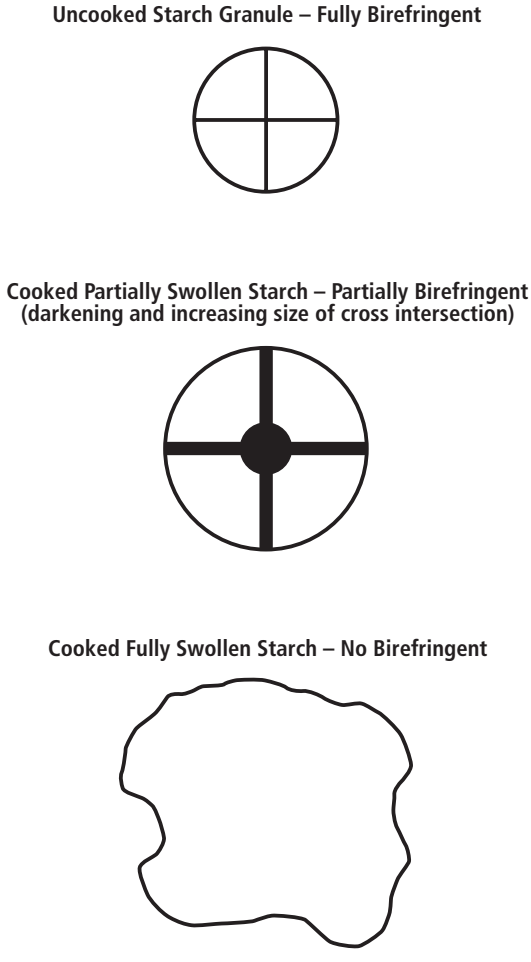


Figure 2. Polarizing characteristics of starch in various stages of cooking—extinction cross is typical for uncooked and partially cooked starch (Yahl 1983).

History and uses

Starch was used in early Egypt (3500 BC) as an adhesive for papyrus and in China (*c.*100 AD) as a sizing for paper (Roff and Scott 1971, p. 185). WSP has been used in Japan since the 15th and 16th centuries and *funori* since 1673 (Liszewska 2005; Wills 1984). Starch was investigated scientifically as early as 1719, but understanding of its composition was not achieved until 1811–14 (Roff and Scott 1971, p. 185). The roasting of starch started in 1821 for British gum and dextrins (Roff and Scott 1971, p. 185). It was not until 1906 that amylose and amylopectin were separated and identified (Roff and Scott 1971, p. 186).

Starch has been used as a size for paper and textiles, filler, printing media, a stiffening agent and a glaze, as well as for labelling, packaging, paper sacks, envelope seaming and sealing, gummed tapes, stamps, cigarette manufacture and wallpaper sizes and pastes (Kruger and Lacourse 1990, pp. 153–166; Lazarus 1983; Roff and Scott 1971, p. 185). In Japan, *shinnori* and *furunori* have been used for the manufacture of hanging scrolls, screens, sliding doors and the mounting of pictorial art (Wills 1984). Traditionally in Japan, *funori* was used for sizing of paper, gilding paper and silk, and pasting paper onto mud walls for tea ceremonies (Masuda 1984). Dextrins have been used for textile finishing and sizing, printing, adhesives for paper, cardboard, stamps, labels and paper tapes (remoistenable) (Masschelein-Kleiner 1995, pp. 51–54; Paper Conservation Catalog 1994, pp. 4–12; Roff and Scott 1971, p. 186). Starch uses are limited to dry interior environments only (Shields 1984, p. 76).

In conservation, starches, primarily wheat starch and sometimes rice starch, have been used as adhesives for paper (e.g. hinging, mending, lining, facing, reinforcement, consolidation and fixing media), bookbinding, textiles and as a relining adhesive for paintings in a glue-paste mixture (Horie 2010, pp. 225–226; Paper Conservation Catalog 1994, pp. 4–12). Starch and maltodextrin have been used to consolidate waterlogged wood (Sanya et al. 2005; Ippoliti 2002). Acid-hydrolyzed WSP has been used as an adhesive for textiles. *Funori* and *junfunori* have been used for the consolidation of matte paint which has little binder in it, and fragile degraded papers (Michel 2011; Michel et al. 2002).

Characteristics, limitations and/or aging studies

General

Pure starch adhesives are stable and do not discolour (Daniels 1995b). Amylose can, however, degrade on exposure to ultraviolet (UV) light, causing chain breaking and production of organic acids (Phillips 1980).

Potato and corn starches can yellow during cooking, and rice starch is weaker and less stable than wheat starch upon natural aging (Horie 2010, p. 224; Paper Conservation Catalog 1994, pp. 4–12).

Pastes made from flour are easier and quicker to make than pure starch pastes because they require less cooking, but they need to be strained to eliminate lumps (Morrison 1980). Due to the gluten content, pastes made from flour discolour and embrittle more than pastes made from pure starch (Wills 1984).

FURUNORI

The properties of *furunori* (WSP aged 10 years) were studied and compared to new WSP (**Table 2**) (Daniels 1988). During the aging process of *furunori*, microbial action reduces the molecular weight, water-soluble sugars (e.g. glucose, maltose, maltotriose, maltotetrose) and organic acids (e.g. glyoxylic, lactic, acetic, malic) form, and the paste becomes more acidic (Kenjo and Arai 1995). The aged adhesive gives weaker bonds on paper than fresh paste, but strong enough (Hayakawa et al. 2007). It has less tack and greater gel stiffness than new paste. Aged paste also prevents fungus growth when used to prepare scrolls (Masuda 1984). This anti-fungal action is attributed to ticks living in the paste. When joins made with *furunori* paste are heated in water, they are more reversible than in cold water. A similar paste to *furunori* was produced using enzymes and aging for only seven days followed by washing with organic acids (Hayakawa et al. 2007). Its properties were similar to 10-year aged *furunori*.

FUNORI AND *JUNFUNORI*

Funori was tested along with cellulose ethers, gelatin and isinglass for tidelines, darkening, gloss, colour on matte pigments and mechanical properties before and after light aging (Michel et al. 2002). The gelatin produced tidelines and became stiff, but had greater adhesion than the *funori*. The *funori* did not darken nor form tidelines. It bleached upon aging and remained relatively flexible. This study suggested that *funori* has the same stability to UV, relative humidity (RH) and temperature as gelatin and sturgeon glue, and better stability than the cellulose ethers. Another study found that *funori* strengthened silk and had good lightfastness (Liszewska 2005).

Further work revealed that *junfunori* remained water soluble after accelerated aging and did not increase susceptibility to fungal attack (Schleuniger and Michel 2005). Also, when *junfunori* was investigated as a consolidant for matte paint, it did not change the appearance, in contrast to Klucel E and Methocel MC, which produced tidelines and darkening (Geiger and Michel 2005). *Junfunori* films showed good stability (tensile strength) to UV and RH changes, whereby the cellulose ethers degraded. These are similar results to the *funori* above.

However, in another study where *funori*, WSP and methylcellulose (MC) were tested for colour changes upon accelerated aging, fold endurance and removability for the purpose of a temporary facing for Eastern paintings conservation, *funori* changed colour upon aging and impacted the paper strength negatively (Fan 2011). It should therefore be removed as much as possible if used as a facing. The WSP had a noticeable colour change. The MC appeared to give the best results for facing these paintings.

MICROBIAL RESISTANCE

Starch and its paste are subject to microbial attack when in damp environments or even when stored for prolonged periods at room temperature (Daniels 1995b; Roff and Scott 1971, p. 185). It is recommended that starch pastes be refrigerated and only stored thus for seven days (CCI 1993).

In a book conservation study that examined rice starch and various other adhesives [cellulose ethers, poly(vinyl alcohol) (PVAL), and poly(vinyl pyrrolidone)] for resistance to microbial growth, rice starch was least resistant and became acidic (Gallo et al. 1989).

Various methods to preserve cooked starch for longer periods without the use of fungicides (e.g. thymol or *o*-phenyl phenol) have been suggested that include covering it with water during refrigeration, sterilizing it in sealed lined aluminum tubes (stable up to two and a half years), using sterile capped syringes for smaller quantities (stable up to six months), and irradiating the surface of the paste with short wave UV light (stable up to eight weeks with re-irradiation after each use) (Miller and Root 1991; IPC 1989; Stone and Morse 1989; Nagin and McCann 1982).

TABLE 2. DIFFERENCE IN PROPERTIES OF NEW AND AGED STARCH PASTE (*FURUNORI*)
(Daniels 1988)

Amylose content	Relative viscosity	Alkali number	Gel firmness	Gel tack	Flexibility	Strength adhesion	Reversibility
new and aged similar	aged – less viscous	aged – more acidic	aged – more stiff	aged – less tacky	aged – more flexible	both stronger than paper	neither soluble in cold water, but aged more reversible

As a possible other means of preserving starch without preservatives, one study examined etherification of starch. Unfortunately, no difference was observed in fungal development of the modified and unmodified maize starches on various papers and glass (Gallo et al. 1996).

PAPER, PHOTOGRAPHS AND BOOKS

Starch films usually have poor flexibility; however, acid-hydrolysis is known to improve starch adhesiveness and flexibility (Van Steene and Masschelein-Kleiner 1980). Nevertheless, acid-modified *shofu* (wheat starch) was found less flexible than expected, and the addition of isinglass and sodium alginate created a more flexible product (Liszewska 2005). Adding MC to wheat starch affords a more flexible and reversible dry film, which has been used as a remoistenable adhesive for paper backings (Brückle 1996).

In a study that examined various modifications to maize starch for paper conservation, etherified corn starch with higher substitution did best after aging in comparison to a lower substitution and a cationic starch (Plossi Zappalà 1996). It displayed fairly good solubility in hot water, retention of pH, and the least change in colour after aging.

Various starches (wheat, rice, Abra), gelatin and cellulose ethers (Cellofas B 3500, Culminal, Klucel G and Klucel L) were tested for colour change, surface drying time, ability to form dry films of constant thickness, shrinkage, flexibility, hardness, pH, bond strength and reversibility upon heat and light aging (Shashoua et al. 1990). The wheat starch gave fairly good results, although it became brittle upon aging and was not very soluble in any of the solvents selected, but could be swollen in water and removed. The rice starch displayed the same flexibility and reversibility as wheat starch, but yellowed upon aging. Gelatin was fairly acidic and showed the same reversibility results as wheat starch; however, its flexibility and strength on paper samples was better. The Klucels were very weak, but gave the best reversibility results, being soluble in all selected solvents even after aging. It was concluded that the Klucels were the most suitable consolidants and wheat starch was the most stable adhesive.

Evaluation of carboxymethylcellulose (CMC), MC, Maida (flour paste) and dextrin with various laminating materials for paper identified that CMC and MC (1%) gave adequate fold endurance to the laminating materials even upon aging and that Maida and dextrin did not (Suryawanshi et al. 1996). Mixing the Maida or dextrin with CMC or MC gave more acceptable results.

WSP was shown to be inadequate for hinging oil-saturated aged newsprint (Baldwin 1998). Various starch pastes (potato, wheat, corn, cationic corn) and MC (with a low degree of polymerization) were tested for leafcasting for their physical, mechanical, surface and optical properties upon heat aging (Vodopivec et al. 2005). The potato starch was found to be too sticky and the corn starch was not stable enough. The short chain MC and wheat starch were recommended for use in leafcasting.

Sodium alginate, which is extracted from brown algae, is used in arrowroot starch pastes (Shashoua 1990; Roff and Scott 1971, pp. 174–181). Alginate-arrowroot starch paste has greater colour stability, retention of flexibility and reversibility than standard WSP when light- and heat-aged (Shashoua 1989). It also gives stronger bonds and greater tack on cellulosic materials. Alginate-arrowroot coatings retain less dust than WSP and Klucel G coatings (Shashoua 1990).

WSP, MC, poly(vinyl acetates) (PVACs), acrylics, and rubber cements were examined in a series of accelerated aging tests using a range of photographic detectors to determine their effect on photographs (Schenck and McCabe 1989). The best adhesives overall were the WSP and the MC.

LEATHER AND SKIN

One study has compiled data on various adhesives that might be suitable for leather (Wouters 1989). Various PVACs, gelatin, methylhydroxyethylcellulose (MHEC) and tapioca starch (10%) were selected for the study because of their peel strength, permeability to water vapour, reversibility in water or acetone, flexibility and least disruption to the surface of the leather. In a continuation of this study, eight adhesives (starch, cellulose ethers, gelatin, parchment glue, PVAC dispersion, acrylics) were examined on parchment and repaired parchment under heat aging (Wouters et al. 1992). Strength, permeability and reversibility were tested. The starch along with the cellulose ethers, parchment glue, gelatin and PVAC passed all criteria. For samples of repaired parchment adhered to goldbeater's skin, only five adhesives were tested (starch, Tylose MH 300, gelatin, parchment glue, Mowilith DM5) and these were aged under cycled RH and light conditions. The most acceptable samples were obtained when the goldbeater's skin was degreased, and starch and the Tylose gave the best results.

When WSP was assessed as an adhesive for gut-skin, it only demonstrated intermittent bonding, as seen from scanning electron microscope images (Horelick et al. 2011). Better contact was achieved when Jade 403 or MC was added to the WSP.

TEXTILES

Examination of various adhesives [hydrolyzed wheat starch, PVAC, PVAL, poly(vinyl butyral), MC, MHEC, CMC, hydroxyethylcellulose] for textile conservation found that hydrolyzed wheat starch was not dangerous to silk even after aging (Masschelein-Kleiner and Bergiers 1984). It remained removable in water and did not discolour, although it did have a tendency to support mould growth in damp environments. Besides the starch, MC was also recommended for textile conservation.

PAINTINGS

A study on the mechanical properties of glue-paste linings was conducted using recipes of wheat starch/gelatin, wheat flour/sturgeon glue and wheat flour/bone glue at various ratios (Ackroyd 1995). According to this study, peel strength increased with increasing amounts of glue, and wheat starch/gelatin (8:1) gave the highest peel strengths. Also, this amount of gelatin in the mix afforded better dimensional stability of the lining.

A further study on glue-paste linings was carried out that investigated the effect of various additives [plasticizers (Venice turpentine, linseed oil and molasses); wetting agents (ox gall and vinegar); a bulking agent (rye flour); and preservatives (phenol, cetyl trimethyl ammonium bromide and hydrogen peroxide)] on the peel strength, load and extension, and preservative performance (Ackroyd 1996). The majority of the additives reduced peel strength and had no effect on flexibility, except for the ox gall, which increased flexibility without reducing peel strength. Hydrogen peroxide suppressed mould growth for two months.

Demuth et al. (2011) have examined many different types of adhesives for thread-by-thread tear repair for canvas paintings. They found that a mixture of WSP with sturgeon glue gave relatively high ultimate tensile strength (UTS) compared to the others tested, and higher UTS than WSP alone. The WSP/sturgeon glue mixture also had other redeeming properties such as neutral pH, reversibility in warm water, good workability and good optical appearance (almost invisible). Pre-cooked starches (ready-made) were also found to give good results when mixed with the sturgeon glue, which would afford quicker preparation.

METALS

In a study that examined the addition of WSP or CMC to PVAC Jade 403 or acrylic Rhoplex N-619, increasing amounts of WSP or CMC decreased shear strength on aluminum substrates, increased equilibrium moisture content and increased swelling under fluctuating humidities (Fulton 1988). The dried samples of Jade 403 with WSP or CMC were soluble in water, while the Rhoplex N-619 samples were not. However, WSP and CMC did increase the swelling of Rhoplex N-619 in water.

SOLUBILITY AND/OR REMOVABILITY*

Dry starch films are not soluble in water, but can be swollen by water mists, steam and hot-water, which afford some removability (Tímár-Balázsy and Eastop 1998, p. 317). This has been shown in a study on various dried starch pastes (wheat, *furunori*, corn, corn amylase, corn amylopectin, rice, potato, wheat flour) that were tested for water solubility (Daniels 1995). None were actually soluble; however, when strips of paper were glued together using the various pastes and then soaked for four hours in water, the wheat starch, rice starch, wheat flour and *furunori* separated easily, while the others did not. This outcome was true even after heat aging.

Starch films will swell and dissolve in aqueous alkali more readily than in water alone (Roff and Scott 1971, p. 184). Starch is soluble in only a few rather toxic solvents [e.g. dilute sodium hydroxide, anhydrous ethylenediamine, dimethyl sulfoxide (DMSO) in water, n-methyl-2-pyrrolidone (NMP)] (Baynes-Cope 1972b; Roff and Scott 1971, p. 183). DMSO and NMP swell starch more than water, with DMSO causing more swelling than NMP. However, the health hazards of DMSO will prevent its use (Daniels 1995; Harding 1977). Also, NMP can cause weakening of paper, and may damage dyes and pigments on Japanese prints that are bound with rice starch, and may move other dyes and pigments as well (Macauseland and Warnes 1978; Baynes-Cope 1972b).

The weakening of paper with NMP was investigated further by Green and Bradley (1988). Soaking paper

* Disclaimer: The solvents listed in this section are ones that might dissolve the polymer or adhesive in question but are not necessarily recommended for conservation treatments. Some may be extremely toxic or may contain elements (e.g. chlorine) that may be harmful to certain materials in your object (artifact). Research the solvent before purchase or use to know its toxicity and chemical nature. It is strongly recommended to test all solvents before use to make sure that they do not adversely affect your object.

for 4 hours in NMP caused a 50–55% decrease in tensile strength, but no further decrease was found up to 48 hours. Acetone, which is used to rinse out the NMP from the paper, only caused a 10–17% decrease in strength. Other 17th century papers showed a smaller decrease in strength (8–22%) due to NMP. Upon aging (light and heat aging), the papers soaked in NMP for 6 hours showed a greater loss in strength than if untreated. These results suggested that NMP, besides disrupting starch bonds, can also affect cellulose bonds in the paper, and its use for removal of starch-adhered backings to water-sensitive papers should be carefully considered.

Sodium CMC increases the solubility of dried WSP when added to the wet paste at levels above 60% (Daniels 1995).

The dextrins are completely soluble in cold water and much more soluble than regular starch (Roff and Scott 1971, p. 186).

ENZYME REMOVAL

Starch can be removed with the aid of enzymes of the diastase type (amylase) (Roff and Scott 1971, p. 184). This process is affected by the purity of the enzyme, pH, length of treatment, temperature, concentration, inhibitors in the solution, application method (immersion, gels, poultices with or without interleaf), additives, thickness of the starch to be removed, and the denaturation and enzyme removal process used when treatment is completed (Shibayama and Eastop 1996; Blüher 1996, 1996b; Erickson 1992). The purity of the enzyme is important (the purer the better) as crude products can cause discolouration of the paper upon aging. The addition of surfactant (e.g. Triton X, gelatin) to an enzyme gel can prevent enzyme absorption by the paper and improve enzyme penetration to the starch.

Some gelatin-based photographic emulsions (even when they test negative for the starch test) are affected by the enzyme α-amylase (Preiss and Cook 1981).

Pre-packaged α-amylase poulticing systems (e.g. Albertina Poultice) are now available (Schwarz 2000; Klug Conservation). These systems are dry paper poultices that have been impregnated with enzymes. They are activated with moisture through blotting paper and can be used for local work. The dried paper poultice can be stored for up to 12 months.

Semi-aqueous amylase poultices have been investigated for the removal of starch for water-sensitive objects such as water-soluble iron gall ink objects (Schönbohm et al. 2004). The addition of solvent to the poultice did protect the ink, but reduced the activity of the enzyme, thus requiring longer treatment times for removal.

Cellulose Ethers

Setting mechanism	Category	Water absorption (%)	Refractive index
evaporation		EC ~1.5 decreases with increasing DS	MC 1.4 or 1.49 EC 1.47 EHEC 1.49 HEC 1.50–1.51 HPC 1.559 CMC 1.515 or 1.59
Glass transition temperature (°C)	**Viscosity (mPa.s)**	**pH**	**Coefficient of linear expansion /°C**
MC 187–197 HPMC 170–198 EC 43 HPC 105 decreases with increasing DS	typically 20–100,000 at 25°C (1 or 2% water-soluble types) but also dependent on type, grade and molecular weight	MC 5.0–5.7 WS-EHEC 5.8–6.0 HEC 6.0–7.0 (2%) or 7.0–8.2 HPC 5.0–8.8 (1%) CMC 7.0 (2% DS 0.7)	EC 90–200 $\times 10^{-6}$

(Sigma-Aldrich b; Gómez-Carracedo et al. 2003; Feller and Wilt 1990, pp. 18, 25; Davidson 1980, pp. 3-12, 4-4, 12-4, 12-6, 13-3, 13-8; Shields 1976, p. 299; BeMiller and Whistler 1973; Roff and Scott 1971, pp. 164–173)

Abbreviations

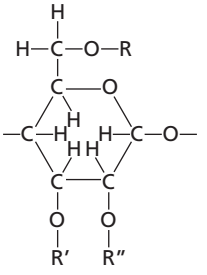
CE	cellulose ether
MC	methylcellulose
HPMC	hydroxypropylmethylcellulose
CMC	sodium carboxymethylcellulose, also referred to as SCMC
EC	ethylcellulose
HEC	hydroxyethylcellulose
EHEC	ethylhydroxyethylcellulose
WS-EHEC	water-soluble EHEC
OS-EHEC	organic solvent-soluble EHEC
HPC	hydroxypropylcellulose
MHPC	methylhydroxypropylcellulose

Types, synonyms and/or trade names

Bermocoll, Blanose, Celacol, Cellofas, Cellosize, Cellulose Gum, Culminal, Ethocel, Ethulose, ethylcellulose, ethylhydroxyethylcellulose, hydroxyethylcellulose, hydroxypropylcellulose, hydroxypropylmethylcellulose, Klucel, Methocel, methylcellulose, methylhydroxypropylcellulose, Modocoll, Natrosol, sodium carboxymethylcellulose, Tylose

Chemistry and/or preparation

Cellulose ethers (CEs) are prepared by treating alkali swollen cellulose with esters of inorganic acids (Roff and Scott 1971, pp. 164–173). The number of anhydroglucose units in the resulting chain is defined as the degree of polymerization (DP) (Bonet et al. 2005). Cellulose is a beta-glucoside, while starch is an alpha-glucoside (Feller and Wilt 1990, p. 10). Alkyl, hydroxyl alkyl and carboxy alkyl groups can replace the hydroxyl sites on each glucose unit. The average number of hydroxyl sites replaced per repeating glucose unit is described as the degree of substitution (DS) (**Figure 1**) (Horie 2010, p. 205; Tímár-Balázsy and Eastop 1998, p. 313). As there are three hydroxyl groups on each glucose unit, the maximum DS is three. When hydroxyl alkyl side-chains form at the hydroxyl sites, these can provide further sites for attachments other than on the base cellulose molecule. In these cases, the term molar substitution (MS) is often used and indicates the number of moles of hydroxyl alkyls per mole of anhydroglucose (Horie 2010, p. 206). This value can exceed three (Davidson 1980, p. 3–2). The properties of the CE depend on its DP, DS and/or MS.



Where R, R′ and R″ can be methyl (-CH_3), carboxymethyl (-CH_2COOH), or ethyl (-CH_2CH_3), etc.

Figure 1. CE fundamental unit (Roff and Scott 1971, p. 164).

History and uses

First investigated in 1905, CEs were further developed after 1912 and well established by the end of the Second World War (Roff and Scott 1971, p. 168).

CEs have such commercial uses as textile finishes, thickening and emulsifying agents, printing inks, adhesives (e.g. wallpaper pastes), sizes, laundry assistant additives [e.g. sodium carboxymethylcellulose (CMC)], binders for pigments, binders for paper and cardboard [e.g. methylcellulose (MC)], and cosmetics, and purified forms are used in pharmaceuticals, dietetic foods and ice cream (Horie 1987, pp. 128–129; Shields 1984, p. 39; Roff and Scott 1971, pp. 167–168, 172).

In conservation treatment applications, CEs have been used since the early 1920s for lacquers; adhesives, consolidants and sizes for paper; consolidants for flaking or friable media; fills; adhesives and consolidants for textiles; reversible facings; pigment fixing; consolidants for waterlogged wood and basketry; adhesives and consolidants for leather and gut-skin; poultice materials; and thickening paste (Horelick et al. 2011; Horie 2010, pp. 211–212). Ethylcellulose (EC) has been used for leather (Horie 2010, p. 211).

Characteristics, limitations and/or aging studies

General

Various commercial uses for CEs along with broad characteristics are summarized in **Table 1**.

CEs form viscous solutions and gels with water or organic solvents. Viscosity depends on type, grade, molecular weight, temperature and concentration of the CE. Some typical viscosities of aqueous solutions are listed in **Table 2**.

Films of CEs have sufficient strength and flexibility that increase with increasing molecular weight (Feller and Wilt 1990, p. 26). Hydroxyethylcellulose (HEC) and hydroxypropylcellulose (HPC) are softer, more flexible and have lower tensile strength than MC and CMC (Feller and Wilt 1990, p. 34).

TABLE 1. SOME PROPERTIES AND COMMERCIAL USES OF CES
(Baumann and Conner 1994; shields 1984, pp. 40–41)

Type	Use	Poor use	Properties
MC	wallpaper leather contact lenses	metals glass ceramics	low DS films clear and odourless resistant to oils and organic solvents less heat-resistant than HEC good wet-tack films strong and resilient no biodeterioration
CMC	wallpaper laundry aid		similar to MC more prone to biodeterioration than MC
EC	paper rubber leather textiles	metals (moderate) wood (only fair)	light-stable resistant to water, alkali and salt fair heat resistance excellent low-temperature durability low flammability no biodeterioration
HEC	paper leather fabric thickener	wood metal glass rubber (fair)	clear resistant to organic solvents resistant to oil and grease hot and cold resistance prone to biodeterioration

TABLE 2. VISCOSITIES OF AQUEOUS SOLUTIONS OF CEs
(Dow.com; KremerPigments.com; Hercules/Aqualon; Hercules/Aqualon b; ShinetSu 2005; Akzo Nobel; Ravines et al. 1989)

Type of CE	Brand name	Viscosity (mPa.s)	
		1%	2%
MC	Methocel A4M	—	4000
MC	Methocel A4C	—	350–550
CMC	Blanose 7HF	1500–2500	—
CMC	Blanose 7LF	—	25–50
HEC	Natrosol 250 GR	—	150–400
HEC	Natrosol 250 HHR	3400–5000	—
MHEC	Tylose MH 2000 YP2	—	2200–3300
EHEC	Bermocoll E 481 FQ	4250–6000	—

Most CEs are less thermally stable than pure cellulose and decrease in thermal stability with increasing DS (Feller and Wilt 1990, p. 22). Manufacturers generally suggest the addition of antioxidants and light stabilizers to products that will be exposed to high temperature and light (Feller and Wilt 1990, p. 40).

For more information on the properties of CEs, see the study by Feller and Wilt (1990). As well as describing the many properties of CEs, this study examined the stability of many CEs by dry-oven aging (90°C, 110°C) and exposure to daylight fluorescent lamps. Changes in viscosity, weight loss and colour change of the powder and films, colour change as a paper size, and peroxide formation upon aging were investigated. Three levels of stability for CEs were established: excellent long-term stability, intermediate stability and unsuitable for long-term applications. A summary of findings for this study is given in **Table 3**. Basically, it was shown that anything with MC in it had excellent stability, while the others showed intermediate or poor stability.

MICROBIAL RESISTANCE

While more resistant to microbial attack than starches, CEs are not completely invulnerable (Roff and Scott 1971, p. 167). MC, EC and ethylhydroxyethylcellulose (EHEC) appear to be unaffected by biodeterioration, while HEC and HPC are prone to it (Shields 1984, pp. 40–41). MC is more resistant to biodeterioration than CMC (Shields 1984, pp. 40–41). Long-term storage of CE solutions is discouraged. In industry, preservatives are added if CE solutions are stored for long periods, but in conservation applications they are not added because they cause discolouration (Paper Conservation Catalog 1994, pp. 21–33). Preparation of solutions should be done under sterile conditions and solutions should be stored in darkness (Horie 2010, p. 210).

PAPER, PHOTOGRAPHS AND BOOKS

Aging studies of various CEs for paper and photo conservation have been conducted (Letnar et al. 2006; Vodopivec et al. 2005; Shashoua and Rugheimer 1998; Strnadovà and Ďurovič 1994; Shashoua et al. 1990; Schenck and McCabe 1989; Ravines et al. 1989; Baker 1984; Hofenk-de Graaf 1981; Zappala-Plossi and Crisostomi 1981; Schaffer 1978; Zappala-Plossi 1977). In these studies, MC performed fairly well in that it remained soluble and showed the least colour change. It increased paper strength somewhat and achieved this at lower concentrations compared to the other CEs. In unaged samples, there was a direct correlation between folding strength, molecular weight and concentration of MC. CMC also remained soluble, but was less effective in strengthening and it yellowed. HPC was generally weak, but had good reversibility and good solubility in various organic solvents.

Instead of investigating the nature of the substituted groups (e.g. methyl, ethyl, hydroxypropyl), one study examined the effect of DP and DS on the properties of the CEs (Bonet et al. 2005). Bending resistance on three types of papers was investigated for variously substituted CEs [MC, hydroxypropylmethylcellulose (HPMC), EC, CMC and HEC]. ECs and CMC gave the highest results and increasing the DS decreased the bending resistance. This decrease is due to a reduction in the number of hydroxyl groups available for hydrogen bonding. Also determined was that the higher the DP, the lower the concentration of paste needed to obtain the same bending resistance. Therefore, DS and DP should be considered when selecting CEs in order to use the absolute least amount to minimize any adverse effects of added material to an object. This research suggested that in selecting a CE from a series of like types (e.g. series of MCs with differing DS and DP), the ones with lower DS and higher DP would be a good choice as less paste would be required for good adhesion.

When HEC was mixed with poly(vinyl alcohol) (PVAL) (10%) and then aged photochemically, it became brown and insoluble (Schaffer 1978).

TEXTILES

In a study of various adhesives for textile conservation that included MC, MHEC, CMC and HEC, it was

Table 3. Summary of Feller/Wilt data for CEs
(Feller and Wilt 1990)

Cellulose ether	Properties of powder/films after thermal and/or light aging	Products studied
Excellent long-term stability		
MC	No discolouration Weight loss minimal No viscosity loss*	Culminal Methocel A4C Methocel A4M
MHPC	No discolouration No weight loss	Methocel E4M Methocel K100LV
CMC	No discolouration but can sometimes occur No weight loss No viscosity loss* Excellent photochemical stability but poor stability has been encountered	Cellulose Gum 9M8 Cellulose Gum 7H4 Cellulose Gum 7L1 Cellofas B 3500
Intermediate stability		
WS-EHEC	Not evaluated thoroughly (with more testing could possess excellent long-term stability)	Ethulose 400 (WS)
HPC lower molecular weight grades	Discoloured but less than higher molecular weight grades Lost weight but less than higher molecular weight grades Lost viscosity* Excellent photochemical stability Low peroxide formation	Klucel G Klucel L
Unsuitable for long-term applications		
HEC	Discoloured Lost viscosity* Lost solubility	Natrosol 250 HR Natrosol 250 LR Natrosol 250 MR
HPC higher molecular weight grades	Discoloured Lost weight Lost viscosity* Excellent photochemical stability Low peroxide formation	Klucel M Klucel H
OS-EHEC	Discoloured Lost weight Lost viscosity* Formed peroxides	OS-EHEC-Low OS-EHEC-High
EC	Initial induction period but then discoloured Lost viscosity* Formed peroxides	Ethocel S-100 Ethylcellulose N-14

* 2% solutions. Loss of viscosity indicates decrease in molecular weight upon aging.

noted that CE solutions increased in viscosity rapidly with concentration, which is a positive result as deep penetration into the textile can be avoided (Masschelein-Kleiner and Bergiers 1984). Also noted was that MC can be soluble in water and also in organic solvents (dependent on DS), which can be used to an advantage for textile applications. In the same study, it was shown that MC, MHEC and CMC did not damage silk before or after light aging (strength of silk with these CEs present was slightly higher than silk alone), but all gave slightly more rigid bonds with silk than hydrolyzed wheat starch did. As HEC caused weakening of the silk after light aging, it was not recommended. The adhesives of choice for textile conservation identified from this study were starch and MC.

CMC (Blanose 7MC), MC (Culminal) and HPC (Klucel G and Klucel L) were examined for textile conservation by investigating various properties (colour change, shrinkage, bonding strength, reversibility) upon heat and light aging

(Shashoua 1995). The results showed that Klucel G yellowed slightly after light exposure, while all others showed no discolouration. Upon drying, MC shrank 5% while the others shrank 2–3%. None shrank upon aging. Both Klucels formed weak paper lap shear bonds and did not age well, while MC formed the strongest bonds. In general, MC and CMC formed weaker bonds than starch and gelatin, but strength was retained better upon aging. Finally, in the same study, the Klucels were soluble in water, acetone and industrial methylated spirits, while all others were only soluble in water. All retained their solubility upon heat and light aging.

LEATHER AND SKIN

One study compiled data on various adhesives that might be suitable for leather (Wouters 1989). MHEC [Tylose MH 300 (2%)], gelatin and tapioca starch were selected in the study because of their peel strength, permeability to water vapour, reversibility in water or acetone, flexibility and least disruption to the surface of the leather. However, the Tylose seemed to be the least reliable of these adhesives. In a continuation of this study, eight adhesives [Tylose MH 300, Klucel G, starch, gelatin, parchment glue, poly(vinyl acetate) (PVAC) dispersion and acrylics] were examined under heat aging for use on parchment (Wouters 1992). Strength, permeability and reversibility were tested. The results showed that Klucel G, Tylose, starch, parchment glue, gelatin and the PVAC dispersion passed all criteria outlined for leather and are ordered by increasing strength. For samples of repaired parchment adhered to goldbeater's skin, only five adhesives were tested (Tylose MH 300, starch, gelatin, parchment glue and PVAC dispersion) and these were aged under cycled relative humidity and light conditions. The most acceptable samples were obtained when the goldbeater's skin was degreased, and Tylose and starch gave the best results.

Klucel G and various other adhesives (PVAC/starch, BEVA 371, wheat starch, corn starch) were examined for their effect on leather bindings (cracking, flexibility and barrier for air pollution) (Lavaquerie and Chahine 2001). New vegetable-tanned calf and sheep leather was covered with two layers of adhesive and aged (under dry heat, humid heat and pollution). Klucel G gave the best results as it protected the leather from air pollutants (including sulfur dioxide), and gave only slight changes in appearance to the leather that were deemed acceptable. Although BEVA 371 remained remarkably flexible, it did darken, was difficult to apply and left brush marks. The remaining three adhesives were not recommended as all three significantly stiffened the leather, with the PVAC/starch adhesive becoming yellow and acidic, and the wheat and corn starches shrinking and cracking after aging.

In a scanning electron microscopy study on adhesives for gut-skin, it was found that MC alone and in combination with Jade 403 showed no intimate contact between the gut-skin and adhesive, but when MC was mixed with wheat starch paste (WSP), the contact was improved (Horelick et al. 2011).

PAINTINGS

Fluorescent labelling was used to visualize the penetration of MC and a mixture with gelatin into painting canvas (Soppa et al. 2011). The gelatin alone impregnated the canvas while the MC alone and in the mixture did not but formed acceptable adhesive layers between the paint and canvas.

METALS

When increasing amounts of CMC or WSP were added to a PVAC (Jade 403) or an acrylic adhesive (Rhoplex N-619), the shear strength on aluminum substrates decreased (Fulton 1988).

SOLUBILITY AND/OR REMOVABILITY*

In general, DS dictates the solubility of CEs (**Table 4**) (Roff and Scott 1971, pp. 165–166). Usually, the higher the DS, the more soluble the CE is in organic solvents. Those CEs with intermediate DS are soluble in water and those with very low DS are alkali-soluble. When DS is higher, there are fewer hydroxyl groups on the glucose unit available to convey water solubility to the CE (Tímár-Balázsy and Eastop 1998, p. 313).

So, generally, except for EC, all CEs are soluble in cold water (Horie 2010, p. 209). CMC and HEC are soluble in hot water, but MC, HPMC, water-soluble EHEC (WS-EHEC) and HPC are not (Horie 2010, pp. 206–211; Feller and Wilt 1990, pp. 13, 20–22; Davidson 1980, p. 3-3; Roff and Scott 1971, pp. 165–166). MC, EHEC and HEC are soluble in dimethyl formamide and dimethyl sulfoxide, while HPC is soluble in polar

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TABLE 4. DS OF CES AND SOLUBILITY
(Roff and Scott 1971, p. 166)

Type of CE	DS of CE and solubility		
	Alkali	Water	Organic solvents
MC	0.3	1.3–2.0	>2.5
CMC	0.1–0.2	0.4–0.9	—
HEC	1	>1.2	>2.1
EC	0.2–0.6	0.8–1.3	1.9–2.6

Light grey highlighting indicates those that are commercially available.

solvents (Horie 2010, pp. 209–210). EC is soluble in organic solvents such as alcohol, acetone, aromatic or chlorinated hydrocarbons, and some esters (Roff and Scott 1971, p. 170).

In general, the organic solvent-soluble EHEC (OS-EHEC) has ethyl and ethylhydroxyethyl groups present, while the WS-EHEC has ethyl and hydroxyethyl groups (Feller and Wilt 1990, p. 21). This difference accounts for the difference in solubility. Also, the OS-EHEC has a much higher substitution of ethyl groups (Davidson 1980, p. 3-2).

CMC can form insoluble complexes in the presence of metal ions such as aluminum and iron (Paper Conservation Catalog1994, pp. 21–33; Baker 1984; Davidson 1980, p. 4-3).

Adding CMC or WSP to Jade 403 produced dried films that were soluble in water, but did not make dried films of Rhoplex N-619 water soluble (Fulton 1988). The addition however did increase the swelling of the dried Rhoplex films.

The reversibility of various MCs (Methocel A4M, Methocel A345, Methocel J5MS, Methocel J75MS, Methocel 311, Methocel F) and ECs (Ethocel Medium 70, Ethocel Standard 10, Ethocel Standard 45, Ethocel Standard 100, Ethocel Standard 200, Ethocel HE10) on paper was investigated using Fourier transform infrared (FTIR) spectroscopy (Bonet et al. 2007). Spectra of paper with no CE were compared with spectra of paper with CE, and with spectra of bonded paper with CE that had been de-bonded by immersion in water for the MC products or ethanol for the EC products. Even though the bonded paper de-adhered easily, considerable CE remained in the paper even after 30 minutes immersion—so even when there is good solubility, it does not mean that all traces of adhesive are removed.

Cellulose Esters - Cellulose Acetate

Setting mechanism	Category	Water absorption (%)	Refractive index
evaporation	thermoplastic	1.9–7 (24 hrs)	1.48 (pure) 1.48–1.51 (plasticized)
Glass transition temperature (°C)	**Viscosity (mPa.s)**	**pH**	**Coefficient of linear expansion /°C**
			$70–180 \times 10^{-6}$

(Omnexus; CAMEO 2010; Shields 1976, p. 299)

Abbreviation

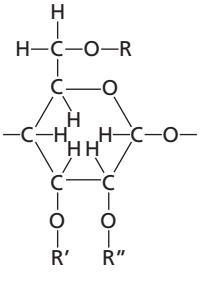
CA cellulose acetate

Types, synonyms and/or trade names

acetylcellulose, acetylated cellulose, Bexoid, Celastoid, Cellastine, Cellomold (cellophane), Clarifoil, Gelastoid, Kodapak (films), Lansil, LePage Cement, Rhodoid, Tenite, Trolitul

Chemistry and/or preparation

Cellulose acetate (CA) is made from cotton linters or wood pulp, acetic acid, acetic anhydride and a sulfuric acid catalyst (Roff and Scott 1971, pp. 150–151). The extent to which the CA is acetylated is termed degree of substitution (DS). The acetyl groups can be added at three places on the fundamental unit (**Figure 1**). The resulting CA can therefore be a secondary acetate if partially acetylated (DS about 2.4) or a triacetate if fully acetylated (DS 3) (Horie 2010, p. 213).



Where R, R′ and R″ are acetyl (CH_3CO-) groups (partial or complete replacement).

Figure 1. CA fundamental unit (Roff and Scott 1971, p. 149).

History and uses

CA was first prepared in 1865, further investigated in 1894 and commercially introduced in 1903 (Roff and Scott 1971, p. 156). It was used to coat, or dope, airplane fabric in the First World War, replacing cellulose nitrate, which is very flammable (Horie 2010, p. 213; Roff and Scott 1971, pp. 155–156).

CA has been used to make films, fibres, fabrics and adhesives. It has been used commercially as an adhesive to glue model aircraft (balsa wood), CA plastics, photographic film, leather, paper, wood, glass and fabrics (Hurd 1959, p. 21). It has fair adhesion to ceramics, but poor adhesion to rubber, metals and glass (Shields 1984, p. 39).

Very early uses of CA in conservation were for fixing pigments on paper and stone (1923), laminating paper (1934), and consolidating cloth (1934), but usage declined by the 1960s (Horie 2010, pp. 213–214).

Characteristics, limitations and/or aging studies

CA is a water-clear adhesive. It is less flammable, more colour stable, more heat-resistant, but less water-resistant than cellulose nitrate (Hurd 1959, p. 21). It is subject to cold-flow, so it is not a structural adhesive (Hurd 1959, p. 21). It is supposedly resistant to mould growth (Hurd 1959, p. 22).

CA can oxidize at room temperature and become weaker and more brittle (Horie 2010, p. 213). If there is residual acid left from the manufacturing process, which was often the case in the older

processes, this can speed degradation, instability and insolubility (Horie 2010, p. 213; Roff and Scott 1971, p. 155). The deterioration can be reduced by incorporating buffers.

Solubility and/or removability*

Secondary acetates are soluble in ketones (e.g. acetone if <57% acetylated; cyclohexanone), 80/20 methylene chloride/methanol, methyl acetate, ethyl lactate, ethylene glycol derivatives, pyridine, dimethyl formamide, phenols and glacial acetic acid (Roff and Scott 1971, p. 151).

Triacetates are soluble in less polar solvents and have more restricted solubility (Roff and Scott 1971, p. 151). They are soluble in chlorinated hydrocarbons with a little alcohol, nitromethane, pyridine, dimethyl formamide, formic acid and glacial acetic acid.

CA is relatively unaffected by hydrocarbons, oil, grease, carbon tetrachloride, perchloroethylene, alcohol, ether, weak acids and weak alkalis (Roff and Scott 1971, p. 151).

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Cellulose Esters - Cellulose Nitrate

Setting mechanism	Category	Water absorption (%)	Refractive index
evaporation	thermoplastic	1–3	~1.50
Glass transition temperature (°C)	**Viscosity (mPa.s)**	**pH**	**Coefficient of linear expansion/°C**
53† 100††			$70–160 \times 10^{-6}$

(†Hamrang 1994, p. 37; ††Paper Conservation Catalog 1994, pp. 38–41; Shields 1976, p. 299; Roff and Scott 1971, p. 161)

Abbreviation

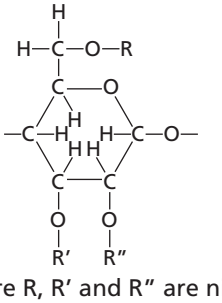
CN cellulose nitrate

Types, synonyms and/or trade names

Agateen, Ambroid, Celluloid, collodion, Duco Cement, Durofix, HMG, nitrocellulose, pyroxylin, Randolph's, UHU Hart, Xylonite

Chemistry and/or preparation

Cellulose nitrate (CN) is prepared by soaking cellulose linters or paper pulp in a mixture of concentrated nitric and sulfuric acids (Horie 2010, p. 214; Roff and Scott 1971, pp. 159–160). The concentration of the acids dictates the number of nitrate groups that can be substituted onto each cellulose molecule (**Figure 1**), to a theoretical maximum of three. This is known as the degree of substitution (DS) (Roff and Scott 1971, p. 160). Following acid soaking, the product is washed to remove the acid and then boiled in alcohol to remove sulfate groups (Roff and Scott 1971, p. 160). If the acid is not removed properly, it can catalyze the decomposition of the CN later (Stewart et al. 1995, 1996b). The DS dictates CN usage with adhesives having a DS of 2.0–2.2 (**Table 1**).



Where R, R' and R" are nitro ($-NO_2$) groups (partial or complete replacement).

Figure 1. CN fundamental unit (Roff and Scott 1971, p. 159).

To convey some flexibility to CN and reduce its propensity to be explosive, it is compounded with plasticizers (e.g. camphor, triphenyl phosphate, tricresyl phosphate, dibutyl phthalate) (Horie 2010, p. 214; Shashoua et al. 1992). A typical CN adhesive is composed of CN (15% by weight, molecular weight about 25,000), plasticizer (5–6%), acetone (11%), ethanol (44%) and amyl acetate (24%) (Koob 1982).

History and uses

CN was first investigated in the 1830s (Roff and Scott 1971, p. 163). Later, in the 1840s, it was developed commercially for "gun cotton", an explosive, and then collodion, which was used in the wet-plate process for photography (Roff and Scott 1971, p. 163). It was

Table 1. Relationship between DS, % nitrogen and uses of CN
(Horie 2010, p. 214; Roff and Scott 1971, p. 160)

Use	% Nitrogen	Approx. DS
plastics, lacquers	10.5–11.5	1
lacquers, films	11.5–12	2
adhesives, lacquers	11.2–12.3	2.0–2.2
explosives (high molecular weight)	12–13.5	2.9

plasticized with camphor in 1864–69 and called Celluloid (one of the early plastics) (Roff and Scott 1971, p. 163). CN was used to coat, or dope, fabric on early aircraft and then was used as a lacquer in the auto industry (Roff and Scott 1971, pp. 162–163).

Its commercial uses were for military explosives; moulded articles; clear and pigmented sheets; photographic film; surface coatings for leather and bookbinding cloth; water-proof finishes; lacquers; general-purpose-household cements for glass, paper, leather, textiles, paper, metals, wood and cellulose acetate (but not rubber); and cardboard manufacturing adhesives (Shields 1984, p. 40; Roff and Scott 1971, pp. 162–163; Hurd 1959, p. 22). Because of its flammable nature, it has been suspended from many uses.

Since the late 19th century, CN has been used in conservation applications as an adhesive, consolidant and coating, and its use continues but with certain reservations (Horie 2010, pp. 214–216). It has been used as a consolidant for stone, earth, soil, wall paintings, pigments, plants, wood and other organic materials; an adhesive for pottery, earthenware, tile, ceramics, glass, enamel, ivory and stone; and a coating for glass, silver, copper, bronze, brass and iron (Horie 2010, pp. 214–216; Selwitz 1988, pp. 1–7).

Characteristics, limitations and/or aging studies

General

CN was favoured in conservation because it is easy to use, dries rapidly and, generally, is completely soluble even after prolonged periods of time (Horie 2010, pp. 214–216). However, in most cases it is no longer favoured because it shrinks, evolves nitric oxides and acids, can be explosive, is very flammable and is unstable when heated, discolours and becomes brittle in sunlight or after exposure to high temperatures (Horie 2010, pp. 214–216; Koob 1982; Roff and Scott 1971, p. 162).

Thermal decomposition of CN occurs at temperatures <135°C, and it will spontaneously degrade at room temperature to one-third of its original molecular weight in about 160 years (Selwitz 1988, pp. 17–34). It is less stable than cellulose acetate (Horie 2010, p. 214). Photochemical degradation of CN occurs in the region of 280–300 nm with yellowing, denitration and carboxyl formation as the effects (Chang 1995). The degradation of CN is autocatalytic, meaning that the degradation products catalyze further degradation unless they are removed. Much of the older CN is more unstable than modern grades that are manufactured to a higher standard (Green and Bradley 1988b; Green 1987). The addition of soluble bases to CN to neutralize the slow release of acids is not a good idea as they can further degrade the film (Selwitz 1988, pp. 31–32). In well-made and processed CN films, this slow release of acids may not be such an issue as illustrated by CN animation cells made without soluble bases for Disney in the 1930s that were still flexible, clear, odourless, of good strength and only slightly discoloured in 1988 (Selwitz 1988, pp. 30–32).

Generally, the more nitrate groups within the CN molecule, the less stable it is (Selwitz 1988, p. 35). Studies show that the addition of zinc oxide considerably lowers the degradation rate and that high humidity increases it (Stewart et al. 1996). CN is compatible with most synthetic and natural resins, but causes epoxy resins to yellow quickly, so care should be taken when previously glued CN bonds are re-adhered using an epoxy (Shashoua 1995b).

Even with all of this degradation evidence, there has been considerable disagreement over whether CN should be used in conservation (Watkinson and O'Connor 1982). While some conservation specialists do not recommend it (Koob 1982), others have found that it has performed fairly well over many years (Shashoua et al. 1992). These latter researchers found that plasticized CN adhesive (e.g. HMG Heat and Waterproof Adhesive) retained its flexibility and discoloured only slightly under unfavourable aging conditions when compared to an unplasticized CN that lost its flexibility and discoloured. Also, they showed that documented naturally aged CN repairs in a museum were stable for 30 years and "may be extrapolated to suggest a lifetime of 50 to 100 years under museum conditions in a temperate climate" (p. 113).

Recently, it has been shown that a chromatographic test, which involves swabbing an artifact made of CN, can be done to identify those with high sulfate content, loss of plasticizer and presence of oxalate, which indicates those most susceptible to degradation (Quye et al. 2011). This chromatographic test could possibly be used for CN adhesives and coatings, too.

CN has fairly high shear strength at room temperature, but because it is thermoplastic it is unsuitable for structural applications, especially at higher temperatures (Hurd 1959, p. 22). It is water-resistant and resistant to bacteria, fungi and insects (Koob 1982; Roff and Scott 1971, pp. 160–162).

Basketry

When CNs (Duco Cement, Scotch Super Strength), poly(vinyl acetate) (PVAC) resins and dispersions,

and a wheat starch paste mixed with MC were tested for their strength and reversibility for use in basketry conservation, the PVAC dispersions had the best characteristics, while the CNs had little tack, were difficult to use and needed excess glue to achieve a good bond (Hartley and Grosso 1981).

CERAMICS AND METALS

The main uses for CN in conservation are as an adhesive for ceramics and as a coating for metal (Selwitz 1988, pp. 1–7). In a review of CNs for conservation by Selwitz (1988), it was suggested that CN is liked as an *adhesive* (especially for ceramics) because it is reversible, quick drying, easy to use (usually supplied in convenient squeeze tubes), has good working consistency, has desirable tackiness, does not penetrate and gives strong bonds. It is liked as a *coating* (especially for metals) because of its ease and speed of application and drying, adhesion, good levelling qualities, dust-free dry finish and optical properties. In this review, it was suggested that adhesives should have a lifetime of stability of 200 to 400 years so that delicate, time-consuming and costly re-repair of objects is minimized. On the other hand, since coatings are more easily removed and replaced, they need only 15 to 25 years of life before replacement is necessary. As it was estimated that properly stabilized CN had a lifetime before degradation of only 25 to 200 years, it makes a better coating than adhesive. For instance, CN would be suitable as a silver coating since silver tarnishes faster than the coating degrades. In other words, the coating would need to be removed for silver re-polishing far sooner than it would need to be removed due to degradation. In this review, it was suggested therefore that other more stable adhesives such as acrylic polymers be considered for adhesive applications.

Ceramics

A study by Castro and Doménech Carbó (1999) reinforced some of the reservations regarding the use of CN in ceramics conservation. They appraised nine different adhesives (PVAC, acrylics, cyanoacrylates, an epoxy resin and a CN, Imedio Banda Azul) for tensile strength, yellowness index (before and after cyclic heat aging), thickness of bond, working time and overall health hazard for Spanish medieval ceramics, and found that Paraloid B-72 gave the best results, while the CN yellowed and had greater toxicity.

Principal component analysis of infrared spectra was used to identify two groups of CN adhesives used to conserve a Cypriot pottery collection in 1972 and in 1987 (Nel et al. 2011). The plasticizer content in the CN used in 1972 was about 15%, while that used in 1987 was about 2%. Since plasticized CN has been shown to be more stable than pure CN (Shashoua et al. 1992), it was decided to give higher priority to re-conserve the pieces previously conserved in 1987 with lower plasticizer content than the pieces conserved with CN in 1972 with higher content.

Metals

In a study that examined CN, acrylic and PVAC products for their suitability as a silver coating (permeability to water and hydrogen sulfide, hardness, elasticity, distensibility, abrasion resistance, adhesion, reversibility and colour upon aging), the CN had poorer resistance to abrasion than the other films tested; was comparable in hydrogen sulfide permeability, distensibility and hardness; had much better adhesion and elasticity; had poorer reversibility upon aging; and was more yellow and damaged than the rest (De Witte 1973). In another study, CN was found not to corrode or tarnish copper, lead and silver (Oddy 1975).

As protective coatings for silver exposed to a hydrogen sulfide environment, one CN, Agateen Air Dry Lacquer #27, performed well (low tarnishing) while another CN, HMG Heat and Waterproof Adhesive, was less effective (Grabow et al. 2007). The Agateen-protected silver had a lower tarnish rate than silver coated with Paraloid B-72 or B-48N (Reedy et al. 2000).

SOLUBILITY AND/OR REMOVABILITY*

CN plastics, fibres and lacquers are soluble in alcohol, alcohol/ether, ketones, esters (e.g. amyl acetate), mixtures of ketones and esters, ethylene glycol monoethyl ether, monoalkyl ether acetates, diethylene glycol, and glacial acetic acid (Roff and Scott 1971, p. 160). Those CN products with 12% nitrogen or more are soluble in ketones, esters and alcohol/ether mixtures (Roff and Scott 1971, p. 160).

CNs are relatively unaffected by cold dilute acids and mild alkalis, aliphatic hydrocarbons and chloroform, and are resistant to water (Shashoua et al. 1992; Roff and Scott 1971, p. 160).

CN will react with metal oxides (e.g. lead, calcium, silver, tin, iron, copper, zinc) to produce an insoluble gel (Horie 2010, pp. 214–215).

* Disclaimer: The solvents listed in this section are ones that might dissolve the polymer or adhesive in question but are not necessarily recommended for conservation treatments. Some may be extremely toxic or may contain elements (e.g. chlorine) that may be harmful to certain materials in your object (artifact). Research the solvent before purchase or use to know its toxicity and chemical nature. It is strongly recommended to test all solvents before use to make sure that they do not adversely affect your object.

Shellac

Setting mechanism	Category	Water absorption (%)	Refractive index
evaporation and/or liquid solidification	thermoplastic and thermosetting when heat cured		1.5210–1.5272
Glass transition temperature (°C)	**Viscosity (mPa.s)**	**pH**	**Coefficient of linear expansion/°C**
melting point 77–90 softens 40–50			91×10^{-6}

(ShellacEPC; Baynes-Cope 1972)

Types, synonyms and/or trade names

black buttonlac, bleached shellac, buttonlac (seedlac, melted and filtered), Diatite, Garnet lac, gum lac, Katki, Kusmi, lemon flakes, orange flakes, seedlac (sticklac, washed and sorted), shellack, shell lacca, sticklac (crude material), Smith's Patent American Composition

Chemistry and/or preparation

Shellac is a resin obtained from trees in southern Asia. It is secreted by parasitic insects (*Tacchardia lacca, Coccus lacca*) as they make cocoons (sticklac). The resin is extracted by melting and filtering, and is then cooled into drops or sheets and dissolved in alcohol (Roff and Scott 1971, p. 237). The tree on which the insect feeds and the time of harvest determine the colour of the resulting shellac resin (Buch et al. 2009). Shellac is a complicated mixture of compounds containing aleuritic acid and sesquiterpene acids that can be plasticized with phthalates or phosphate plasticizers and castor oil (Horie 2010, pp. 258–260; Roff and Scott 1971, pp. 237–238).

History and uses

The use of shellac goes back at least 3000 years (Hoyas 2003). In the 10th century, it was introduced into Europe. By 1220, it was cited as an artist's medium in Spain and, by the mid-16th century, it was in widespread use (Hoyas 2003; Roff and Scott 1971, pp. 237–238).

Shellac has been used to varnish furniture, floors and wood panelling; to mould goods (e.g. gramophone records, boxes, buttons, frames for ambrotypes and daguerreotypes); to coat tires, handlebar tape, Braille text and leather; to glaze pills, candy and apples; to laminate mica and paper; and to stiffen felt hats. It has also been used as an electrical insulator; a stain blocker in paint primers; an additive for sealing wax, printing inks and fireworks; and an adhesive for porous materials, metals, ceramics, cork and mica (Columbini and Modugno 2009, p. 17; Ebnesajjad 2008, p. 100; Mills and White 2003, p. 115; Roff and Scott 1971, p. 238).

In conservation applications, shellac has been used as an adhesive, consolidant and surface coating (Hanna and Lee 1988). It has been used as an adhesive for stone, ceramics and dry-mounting photographs; as a varnish for decorative paintings and woodwork; and as a French polish (Horie 2010, pp. 258–260; Hanna and Lee 1988; Koob 1979; Baynes-Cope 1972). It was once used as a geological-specimen coating and consolidant in the 1860s (Elder et al. 1997).

Characteristics, limitations and/or aging studies

General

As an adhesive, shellac was generally used as a hot-melt (heat-set) adhesive or in alcoholic solutions (Hurd 1959, p. 36). In the past, it was favoured in conservation as a heat-set adhesive because it melted below 100°C, was non-tacky when set, did not creep but was flexible, and was impermeable to water (Baynes-Cope 1972). It was also easy to use and

readily available (Koob 1984b). Its disadvantages are that it becomes brittle, loses strength, discolours, requires strong solvents to remove it and leaves permanent stains (Horie 2010, pp. 258–260; Hanna and Lee 1988). It can crosslink upon heating and prolonged storage (Horie 2010, pp. 258–260). It is no longer recommended for conservation use.

STONE

Shellac shows only medium resistance to microbial growth on stone (Koestler 2000).

SOLUBILITY AND/OR REMOVABILITY*

Shellac is soluble in alcohol, alkalis (e.g. aqueous borax, sodium carbonate or ammonia), pyridine and formic, acetic and lactic acids (Roff and Scott 1971, pp. 237–238). Resistance to water is poor (Roff and Scott 1971, pp. 237–238). On prolonged storage, shellac becomes insoluble in cold alcohol, but dissolves on heating (Roff and Scott 1971, pp. 237–238).

Freshly set shellac can be removed with 2-methoxy ethanol or ethanol/acetone (50:50) (Koob 1979). Aged crosslinked shellac is very hard to remove mechanically. It has been removed successfully from photographic prints with methyl cellosolve followed by methanol (Baynes-Cope 1972). On the other hand, soaking in pyridine (1–4 hours) was needed to remove aged shellac and stains from ceramics (Koob 1979). Other solvents such as dichloromethane and ammonia/industrial methylated spirits (50:50) were less effective or not recommended for glazed ceramics (Koob 1979).

* Disclaimer: The solvents listed in this section are ones that might dissolve the polymer or adhesive in question but are not necessarily recommended for conservation treatments. Some may be extremely toxic or may contain elements (e.g. chlorine) that may be harmful to certain materials in your object (artifact). Research the solvent before purchase or use to know its toxicity and chemical nature. It is strongly recommended to test all solvents before use to make sure that they do not adversely affect your object.

Poly(vinyl acetates) and Copolymers

Setting mechanism	Category	Water absorption (%)	Refractive index
evaporation (dispersions and resins in solution; dispersions coalesce) liquid solidification (hot-melts)	thermoplastic	1–8	1.46–1.47
Glass transition temperature (°C)	**Viscosity (mPa.s)**	**pH**	**Coefficient of linear expansion /°C**
28–40	~2000 (typical dispersion)	4–5 liquid dispersions 4–7 water-extracted dry films (unaged)	PVAC 85×10^{-6} † PVAC $120–130 \times 10^{-6}$ VAE $160–200 \times 10^{-6}$

(Alderson et al. 2012; Down et al. 1996; Shields 1976, p. 299; †Roff and Scott 1971, pp. 68–69)

Abbreviations

PVAC is the preferred abbreviation for poly(vinyl acetate) (ASTM D1600-08 2008), but PVAc and PVA have also been used. Since PVA has been used to represent both poly(vinyl alcohol) and poly(vinyl acetate), it is no longer used as an abbreviation for either.

VAC vinyl acetate monomer

EVA ethylene/vinyl acetate copolymer (more ethylene than VAC in copolymer)

VAE vinyl acetate/ethylene copolymer (more VAC than ethylene in copolymer)

VADBM vinyl acetate/di-butyl maleate copolymer

Types, synonyms and/or trade names

PVAC Aquadhere, AY series* (e.g. AYAC), Bondfast, CM Bond, Elmer's, Gelva, Planatol, Ponal, Titebond, Vinac, Weldbond

EVA BEVA, Elvax

VAE Dur-O-Set, Elvace, Evacon, Jade, Vinamul, Vinnapas

Other Adhesin, Appretan*, Clariant*, Duvilax, Everflex, Mowilith*

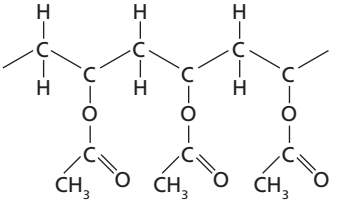
Chemistry and/or preparation

Vinyl acetate (VAC) monomer is prepared in various ways, for example, with acetylene, acetic acid and mercuric compounds (Roff and Scott 1971, pp. 66–67). Polymerization of VAC to poly(vinyl acetate) (PVAC) is effected in solution or aqueous suspension by ultraviolet (UV) or free-radical initiators (Roff and Scott 1971, pp. 66–67). PVACs can be bought as solid resins or dispersions, which can be homopolymers or copolymers (**Figure 1**) with varying molecular weights. Many copolymers exist (e.g. with ethylene, alkylmaleates or alkylacrylates). Plasticizers are often added to homopolymer dispersions to increase flexibility. Typical protective colloids for PVAC dispersions are partially hydrolyzed poly(vinyl alcohol) (PVAL), surfactants or cellulose ethers.

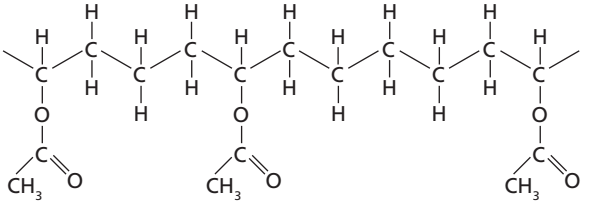
Besides plasticizers and protective colloids, dispersion formulations may also contain extenders, fillers, thickeners, solvents, wetting agents, tackifiers, defoaming agents, biocides, humectants, freeze-thaw stabilizers, pigments or dyes.

The ethylene copolymers, vinyl acetate/ethylene copolymer (VAE) and ethylene/vinyl acetate copolymer (EVA), are prepared by the

*At the time of publication, the Dow Chemical Company had acquired the Union Carbide Corporation, who made the AY series of resins (Watson 2009). Over the years, there have been many such mergers, or takeovers, of adhesives companies, as well as product name changes. It is sometimes difficult to trace the products that are available today (even if they are the same product) because the names have changed or production has stopped. For example, Mowilith DMC2 was renamed Appretan MB Extra and eventually renamed Clariant T1601 because of company takeovers, while the AY series of resins have disappeared altogether.



PVAC Homopolymer



VAE Copolymer

Figure 1. Examples of a PVAC homopolymer and VAE copolymer

copolymerization of ethylene with VAC (Roff and Scott 1971, p. 17). VAE copolymers are usually water-based dispersions with 60–95% VAC content, while EVA copolymers are usually solid materials used for hot-melt applications with 10–40% VAC content (Chemquest).

History and uses

VAC was first prepared in 1912 in Germany and later, in 1917, was produced in large-scale in Germany and Canada (Roff and Scott 1971, p. 70). EVA was first developed by Dupont in 1956 and introduced commercially as Elvax in 1960 (Plastics Historical Society 2011).

PVACs and VAEs have many commercial uses, such as for paints, coatings, adhesives, fabric finishes, packaging, laminating and shrink-wrap. As an adhesive, PVACs adhere to more surfaces than any other adhesive (Jaffe et al. 1990). Thus, they are among the most popular adhesives and are certainly the most common wood glue. EVAs are used extensively in commercial bookbinding, packaging, nonwovens (e.g. disposable diapers), furniture parts and heat-seal labels (Eastman and Fullhart 1990).

In the 1930s, PVAC *resins* started to be used in conservation. They have been employed for facings, varnishes, linings [paintings and drawings (Jirat-Wasiutynski 1980)], pottery, bone, fossils and geological specimens (Horie 2010, pp. 137–142). *Dispersions* became widely used in conservation after 1940 for textiles, paper, waterlogged materials, wet and dry bone, fossils, inpainting, lining paintings and wall paintings (Horie 2010, pp. 137–142; Marxen-Wolska 1991). EVA resins form the basis of BEVA 371, which was developed in the 1960s and has been used for lining paintings, and backing textiles and leather.

Characteristics, limitations and/or aging studies

General

PVAC resins are clear and colourless. They may creep under sustained load, which might exclude them from certain structural applications (Shields 1984, p. 71). Some PVACs have a glass transition temperature (T_g) around room temperature and are thus subject to cold flow and retention of dust (Tímár-Balázsy and Eastop 1998, pp. 304–331). PVACs have low resistance to weather and moisture, and are for interior use only (Shields 1984, p. 71).

PVAC resins can be dissolved in non-aqueous solvents, which make them useful when water must be avoided. They are quick-drying (depending on solvent), adhere to slick surfaces [e.g. poly(ethylene glycol)-treated surfaces] and are glossy when set. Generally, the resins have fewer additives to interfere with stability than dispersions do.

Advantages of aqueous dispersions over comparable resins in solution are that higher molecular weight resins (if higher molecular weight is desired, which is not always the case in conservation, especially where reversibility is concerned) and higher solids content with relatively low viscosity are possible (Jaffe et al. 1990). PVAC dispersions are generally viscous, non-penetrating, strong, flexible and quick-drying with good tack. Sometimes, cellulose ethers and starch are added to improve reversibility, increase viscosity or reduce penetration. PVAC dispersions are frequently acidic because acetic acid is often added by manufacturers to stabilize the dispersion or dissolve additives such as thickening agents (Tímár-Balázsy and Eastop 1998, pp. 304–331; Tétreault and Stamatopoulou 1997).

PVAC copolymers with alkylmaleates and alkylacrylates are able to adhere to a vast number of difficult-to-bond surfaces, but have poor heat resistance, lower strength and are slower setting than the homopolymers. Typical alkylmaleate copolymers that have been used in conservation (e.g. Mowilith DMC2) have low T_g (10°C) and are flexible yet strong. Ethylene copolymers have good strength (comparable to homopolymers), good heat resistance,

TABLE 1. VAE DISPERSIONS – EFFECT OF ETHYLENE CONTENT
(Jaffe et al. 1990)

Property	Ethylene content (%)		
	17	25	31
T_g (°C)	0	-20	-30
water resistance	good	fair	poor
paper adhesion	excellent	good	fair
setting speed	excellent	good	fair

and better adhesion and creep resistance than the ester copolymers (Jaffe et al. 1990). Also, ethylene copolymers retain their peel strength upon aging better (Jaffe et al. 1990). They may be glossy when dry and can stain when applied wet. The amount of ethylene in a VAE copolymer affects the polymer's T_g, water resistance, adhesion and setting speed (**Table 1**).

Copolymers have lower T_g and higher flexibility than homopolymers because the pendant acetate groups have greater intermolecular movement and better separation in copolymers than homopolymers (Jaffe et al. 1990). In homopolymers, the closeness of the acetate groups causes more intermolecular rigidity or stiffness. Therefore, little or no plasticizer is needed with these copolymers. Adding plasticizer to homopolymer dispersions lowers T_g and increases flexibility, but plasticizer can be lost in a few decades (Barrow 1965), migrate into the object with time, cause bond failure (Jaffe et al. 1990), cause loss of flexibility and stain the substrate (Krutzel 1989).

PVAC branching during polymerization will increase its tensile strength, toughness, heat and creep resistance, and reduce the tackiness of the dried film (Jaffe et al. 1990). Addition of functional groups to the PVAC structure can increase the adhesion to difficult-to-bond substrates and the degree of crosslinking (Jaffe et al. 1990).

Dispersions protected with partially hydrolyzed PVAL have larger particle size, better wet tack and flow, and are more rapid setting than surfactant-protected dispersions (Jaffe et al. 1990). Dry films made from PVAL-protected PVAC dispersions are hazy, flat (matte) and water-sensitive as the particles cannot form a tightly coalesced film, while films from surfactant-protected dispersions are clear, glossy and water-resistant as particles are coalesced more tightly (Jaffe et al. 1990). The water-sensitivity of the PVAL-protected film is attributed to the PVAL (Jaffe et al. 1990).

PVAC is not subject to degradation by oxygen, ozone or UV radiation; however, it does degrade above 130°C (Jaffe et al. 1990; Roff and Scott 1971, p. 69). PVAC dispersions can hydrolyze to acetic acid and PVAL either in an acid or alkaline medium, but *films* are very resistant to hydrolysis (Ďurovič et al. 1991). Some PVACs release esters that in moist acidic conditions hydrolyze to acids, which could be damaging to acid-sensitive objects (Tímár-Balázsy and Eastop 1998, p. 321). PVAC film yellowing is likely caused from the formation of double bonds when acetate groups are eliminated, which appears to occur at a slow rate under "appropriate museum conditions" (Tímár-Balázsy and Eastop 1998, p. 321). This causes the remaining polymer chains to "fit together more closely", which increases crystallinity, rigidity and brittleness, and decreases solubility due to a decrease in the number of polar side-groups (Tímár-Balázsy and Eastop 1998, pp. 320–321). Yellowing initiated by sunlight may be less intense due to the competing reaction of bleaching (Ďurovič et al. 1991).

For EVAs, clarity, adhesion, flexibility, toughness and solvent solubility increase with increasing VAC content (Eastman and Fullhart 1990). Copolymers with >25% VAC content exhibit negligible tendency to crosslink under UV radiation (Feller and Curran 1970).

Some general properties of a few typical PVAC resins and dispersions are given in **Table 2**.

MICROBIAL RESISTANCE

Dried PVAC films are somewhat susceptible to microbial attack even though handbooks (Jaffe et al. 1990; Roff and Scott 1971, p. 70) state that they are unaffected. The conservation literature cites several examples. For instance, PVAC copolymers (Mowilith DM55, a dispersion, and Mowilith 35/73, a solid grade) used to consolidate crypt mural paintings were attacked by micro-organisms (Heyn et al. 1996). In another study, PVAC (Mowilith 60, a solid grade) supported microbial activity after parchment, treated with this adhesive and microbes, was aged for several weeks (Altibrandi and Sclocchi 1996). Diluting PVAC dispersions may have a negative impact on mould growth as *undiluted* PVAC dispersion (Aquadhere) used on Aboriginal bark paintings was more resistant to mould than if *diluted* (50:50) (Edmonds 1990). When bacteria and fungi were cultured with PVAC (latex, water-based) and seven other canvas lining adhesives, the PVAC supported microbial growth (Seves 1993). Finally, when 16 adhesives used with stone were tested for microbial attack in a five-week assessment, AYAA, a PVAC, did fairly well, but AYAF and AYAT showed medium resistance, while AYAC was the worst (Koestler 2000). These studies suggest that

TABLE 2. PROPERTIES OF SOME TYPICAL PVAC RESINS AND DISPERSIONS

(Manufacturer data sheets, available from mfrs; Horie 2010, pp. 137–142; Down 2015, 2009; Kronthal et al. 2003; Down et al. 1996; Horton-James et al. 1991; Edmonds 1990; Raw Materials Index 1981)

Adhesive	Composition	Particle size (μm)	Viscosity (mPa.s)	pH a) mfr b) unaged c) dark aged d) light aged	a) Colour b) Discol-ouration upon aging	Mechanical properties: a) Cohesive strength b) Flexibility	T_g or T_s (°C)	Soluble in
PVAC HOMOPOLYMER RESINS								
AYAA	PVAC MW = 83,000			a) — b) 5.71 c) 6 to 7 d) 4.5 to 5.3	a) water-white b) fair to poor when aged	a) strong b) brittle	T_s = 150	most solvents except aliphatic hydrocarbons
AYAC	PVAC MW = 12,800			a) — b) 4.22 c) 6 to 7.4 d) 4.4 to 6.5	a) water-white b) fair to poor when aged	a) medium b) brittle	T_s = 90	most solvents except aliphatic hydrocarbons
AYAF	PVAC MW = 113,000			a) — b) 5.06 c) 6 to 7.3 d) 4.6 to 6.6	a) water-white b) fair to poor when aged	a) strong b) brittle	T_s = 171	most solvents except aliphatic hydrocarbons
AYAT	PVAC MW = 167,000			a) — b) 5.48 c) 6 to 7 d) 4.6 to 5.7	a) water-white b) fair when aged	a) strong b) brittle	T_s = 187	most solvents except aliphatic hydrocarbons
Vinac B-15 (McGean B-15)	PVAC MW = 90,000		100 (20% in toluene)	a) — b) 4.05 c) 4.3 to 7 d) 4 to 6.5	a) clear, colourless b) fair to poor when aged	a) strong b) brittle	T_s = 130	wide range of solvents. lower alcohols require 5–10% water
PVAC HOMOPOLYMER DISPERSIONS								
Resin W	PVAC + soap + UN				a) translucent	a) stronger than wood		
Aquadhere 501	PVAC + soap + DBP + UN (emulsifier/ protective colloid = PVAL)	1	2500–4000	a) 3 to 4 b) 7.0 c) — d) 4.4	a) clear and translucent b) some colour change on aging	a) stronger than wood b) flexible	T_g = 40	
VAE COPOLYMER DISPERSIONS								
Vinamul 3252	VAE + CMC (emulsifier/ protective colloid = PVAL)	1–2	1200–2400	a) 4.5 to 5.5			T_g = 3	
Jade 403	VAE + soap + PAM	6–7	4000–5000	a) 7.1 to 7.3 b) 7.04 c) 7 to 7.5 d) 6 to 7.3	a) clear b) good to fair when aged	a) medium b) flexible		
EVA COPOLYMER								
BEVA 371 Original Formula (solution)	EVA + cyclohexanone + phthalate ester of hydroabietyl alcohol + paraffin + toluene + naphtha			a) — b) 7.57 c) 6.7 to 7.6 d) 5.8 to 6.4	a) white opaque b) fair to poor when aged, especially poor when light aged	a) medium b) flexible	T_s = 60–65	naphtha benzine acetone
VADBM COPOLYMER DISPERSION								
Mowilith DMC2 (Appretan MB Extra, Clariant T1601)	VADBM + MC (emulsifier/ protective colloid = HEC)	0.3–2	5000–12,000	a) 4–5 b) 6.62 c) 6 to 6.7 d) 4.6 to 6.5	a) clear b) good to fair when aged	a) medium b) flexible	T_g = 10	

resistance testing is advisable prior to adhesive treatment, especially if the substrate is heavily contaminated with micro-organisms.

HEALTH ISSUES

PVACs have low toxicity (Jaffe et al. 1990). However, some PVAC dispersions contain VAC *monomer*, which is a suspected carcinogen (Letts 1990). These products should be used in well-ventilated areas or fume hoods. Consult material safety data sheets to determine if VAC monomer is present. There is no evidence of toxicity associated with the PVAC *polymer*.

SHELF LIFE

The shelf life of PVAC *resins* stored at 20°C is indefinite (Shields 1984, p. 71), while that for *dispersions* kept in cool conditions is 6–12 months after production (Horie 2010, p. 141; Down et al. 1996).

PVAC CONSERVATION STUDIES

General

Dispersions of PVAC homopolymers (Resin W, Vinamul 9910), copolymers (Vinamul 6815, Vinamul 3252, Vinamul 3254, Mowilith DM5, Mowilith DMC2) and various acrylics were tested for weight, colour, solubility, tensile strength and pH changes after exposure to natural conditions (both dark and light through lab window), direct sunlight, fluorescent lighting and thermal dark aging (at 59°C and at 83°C) (Howells 1985). Vinamul 3252 showed little change after testing. The most common change was yellowing, which was related to removability. Resin W appeared to crosslink, while Vinamul 6815 and Vinamul 9910 showed evidence of chain-breaking.

PVACs (Mowilith DM5, Mowilith DMC2, Mowilith DM155, Mowilith DM22, Vinnapas), PVAL, acrylics and hydroxymethylcellulose resins were tested by thermal aging and after exposure to light for durability and long-term reversibility (Wächter 1995). The PVACs showed a tendency for degradation, browning and insolubility/irreversibility, while the acrylic (Paraloid F-10) gave the best results. The PVAC samples were compared to PVAC that had been naturally aged for 30–40 years. The artificially aged samples showed degradation, while the naturally aged samples did not. The conclusion was that artificial aging is not necessarily representative of the normal aging process.

A Canadian Conservation Institute study (Down 2015, 2009; Down et al. 1996) evaluating pH, emission of harmful volatiles, strength, flexibility and yellowing of 27 PVAC adhesives under dark [22°C, ~45% (RH)] and fluorescent light aging (190 µW/lm, 700–800 lux) found the following:

- The extracted-dry-film *pH* of PVAC homopolymers tended to be more acidic than copolymers, and light aging caused the films to become more acidic than dark aging. Dispersion pHs (the liquid right out of the container) were acidic and became more acidic upon storage of these dispersions for three years, resulting in the recommendation not to store dispersions longer than one year. Of all tested, Jade 403 and BEVA 371 were the most acceptable as far as pH was concerned after 15 years of light aging and 27 years of dark aging.
- As far as *emission of harmful volatiles* is concerned, dried films of PVACs released the most volatile acetic acid in the first three months of aging and then the emission decreased to almost zero after two years. Generally, light exposure caused a greater increase in acetic acid emission than dark aging. AYAA, AYAC, AYAF, AYAT, Rabin's mixture, Vinac B-15, BEVA 371, UHU All-Purpose Clear Adhesive and Jade 403 released the least amount of acetic acid of all PVACs tested. Recommendations from this study were to avoid adhesives with high acetic acid emission, and it was strongly recommended to air dry the adhesive for at least a month (longer is better) prior to sealing in an enclosed space. Interestingly, in a related study by another researcher, it was shown that areas on artwork (paper and wood) treated with PVAC adhesives in 1980 were less acidic and discoloured years later than untreated areas when examined, which bodes well for PVAC use (Campo Francés 2009).
- Aging (14 years of light aging and 25 years of dark aging) tended to decrease the *flexibility* of PVAC films, especially those composed of homopolymers; however, most of the copolymer films actually increased in flexibility. The flexibility of the copolymers and the fact that they retained medium cohesive strength upon aging are both desirable adhesive properties in most conservation applications.
- In general, light exposure increased *yellowing* of PVAC films compared to dark aging; however, some products actually bleached. UHU All-Purpose Clear Adhesive, Weldbond, Elvace No.1874 and Rabin's mixture yellowed the least of all products tested after 15 years of light aging and 26 years of dark aging.
- The above *overall results* assessed together showed that Jade 403, BEVA 371, Rabin's mixture and R-2258 ranked the best overall, but even these had their drawbacks. BEVA and Jade yellowed somewhat (BEVA much more than Jade); Rabin's mixture became acidic upon light aging (dark aging was fine);

and R-2258 became acidic and yellowed upon light aging. Also, it must be remembered that removability was not investigated. The VAE copolymers did well as a group and were examined in a separate study that evaluated a VAE dispersion (Dur-O-Set E-150) with no additives except for the PVAL protective colloid (Down 2000). Adding known quantities of various modifiers (plasticizers, solvents, wetting agents, fillers, thickeners, freeze-thaw additive and humectant) to the Dur-O-Set E-150 proved that no modifier improved its stability to pH, yellowing, flexibility/strength, gloss and removability changes with dark and light aging. However, most added solvents, wetting agents, and thickeners only altered the properties slightly, while plasticizers and the humectant modified the properties more negatively (lower pH, poorer colour, lower swelling or cohesively weaker) (Down 2014).

Paper, photographs and books

In 1965, a landmark study was published on PVACs for bookbinding (Barrow 1965). Many homopolymers and copolymers were examined and it was concluded that the copolymers were more stable than homopolymers (with or without plasticizer). It was estimated that these copolymers would keep their flexibility for about 450 years.

Ten years later, two studies on PVAC dispersions applied to paper reported the aging properties of homopolymers and copolymers (Baer et al. 1975, 1975b). Homopolymers reduced the fold strength of coated paper, while copolymers increased it. Jade 403 alone and mixed with methylcellulose (MC) showed the least discolouration, while homopolymer Elmer's Glue-All and copolymers Everflex A and Everflex G severely discoloured. Generally, however, homopolymers discoloured more than the copolymers. Complete reversibility with water or toluene was not achieved, but swelling in toluene occurred. From these studies, PVAC dispersions were not recommended for paper conservation, but Jade 403 and the mix with MC looked promising.

In another study, various internally plasticized PVAC dispersions (copolymers) were examined. The one containing starch (10.5%) (Adhesin A22) was the best overall because it did not penetrate the paper, was non-tacky, had fair removability, and exhibited only slight discolouration and pH changes after heat aging (Banza 1977).

A PVAC homopolymer dispersion (Duvilax BD-20 with DBP plasticizer), a VAC/butyl acrylate copolymer dispersion (Duvilax KA-1), and various other acrylic copolymers were tested for double-fold resistance, tensile strength, whiteness, pH, resistance to hydrolysis, adhesive power of the film, removability and microbiological resistance upon thermal, cyclical thermal and RH aging, and artificial sunlight exposure (Ďurovič et al. 1991). Interestingly, the PVAC homopolymer with plasticizer and the VAC/butyl acrylate copolymer were found to be the most suitable, and not the acrylic copolymers.

The mechanical properties of deteriorated paper and its aging stability were improved when treated by different methods with VAC/vinyl versatate copolymer (different concentrations, plasticizers, heated and pressed, pre-washed) (Nada et al. 1999).

PVACs (AYAA/AYAC in toluene, Elvace No.1874 dispersion), wheat starch paste (WSP), MC, acrylics and rubber cements were examined to determine their aging effects on photographs (Schenck and McCabe 1989). The PVACs showed some mottling, fading and orange spots on the photographic material, but, in general, they did fairly well in comparison to the acrylic and rubber adhesives. The best overall (least effect on photographs after aging) were WSP and MC.

When dry-mounting adhesives were investigated for adhesive bond failure under various aging conditions (in the dark and in the light of a window) and also with and without use of solvents (ethanol, acetone and toluene) or heat (100°C, 2 minutes; 150°C, 3 minutes), BEVA 371 bonds released with toluene and with heat (100°C, 2 minutes) after dark and light aging, but acetone and ethanol had no effect (Fairbrass 1992). The most removable dry-mounting adhesives were Archival Mount, Twin Release and Texicryl.

BEVA 371 was used successfully for lining old leather bookbindings that had suffered red rot, powdering, embrittlement and/or fragmentation. It penetrated into the leather and enhanced flexibility without significantly altering the appearance, although a slight colour change resulted (Thompson 1978).

Evacon-R has been used for the conservation of books, sometimes in combination with MC or WSP, but not to conserve actual paper (Scheper 2005). According to its company literature, it is water soluble, non-plasticized and supposedly neutral. When it was tested, the solubility and tensile strength were negatively affected by artificial aging, but not enough to be reconsidered for its use to conserve books (Scheper 2005).

Leather and skin

BEVA 371 solution has been used successfully (cold, full strength) for repair of native-tanned skin, and oiled and waxed skins (Fenn 1984). Its advantages when heat-set are that it has a lower heat-seal temperature than other thermoplastic adhesives, is not tacky on cooling, and produces strong flexible bonds that remain so upon aging (Calnan et al. 1991b).

At the American Museum of Natural History, older skin and leather adhesive treatments were re-examined to assess their repair stability (Kronthal et al. 2003). With the BEVA-treated objects, only a few repairs showed signs of failure and these were not due to adhesive deterioration, but to excessive handling, poor choice of backing material, insufficient preparation of the skin, not enough adhesive and insufficient heat or dwell-time in the heat-sealing process. The other PVACs (AYAA and AYAC) and acrylic-treated objects showed more failures for similar reasons (poor treatment decisions rather than adhesive deterioration).

A preliminary study showed that the shrinkage temperature of leather treated with BEVA 371 and aged naturally for eight years significantly decreased in comparison to untreated leather (Young 2007). The leather treated and aged for only six months showed no decrease in shrinkage temperature. Since any decrease in shrinkage temperature indicates leather deterioration, use should be considered carefully; further research in this area is needed.

BEVA 371, Elascol MIC 55 (a PVAC/starch mixture), and other adhesives (Klucel G, rice and corn starch) were tested on new vegetable-tanned calf and sheep leather upon aging (heat, humidity, pollution) (Lavaquerie and Chahine 2001). Klucel and BEVA performed well, while the others did not and should be avoided.

In a scanning electron microscopy study on adhesives applied to gut-skin, Jade 403 dispersion and AYAA resin (15% in acetone) showed no intimate contact (bond) at the interface between the adhesive and the gut-skin when set; however, when WSP was added to the Jade, the contact was improved (Horelick 2011). BEVA 371 applied in a solution of xylene had intermittent contact, but contact was better when BEVA heat-set film was used. Elvace 45675 CX showed intermittent contact. In general, the acrylics in this study appeared to give better contact (bonds) than the PVACs.

A PVAC copolymer (Vinamul 3254) was found to give strong bonds to leather when applied to a backing fabric and bonded with wet adhesion, but it was not as strong as BEVA (Calnan et al 1991b). Although a dot matrix nap bond reduced bond strength, it was more flexible than a continuous film.

Textiles

In the 1950s, Vinamul 6515 and Vinamul 6525 were introduced into textile conservation in England, and their use continued until the 1960s (Blum 1982). These were applied onto nylon net and heat-sealed as a backing onto the object. Unfortunately, this treatment stiffened objects, and these tacky adhesives retained dust. The backing technique using Mowilith DMV1 (later DMC2) mixed with Mowilith DM5, applied and dried on nylon tulle, and then heat-set, was first introduced in Amsterdam and later perfected at the Victoria and Albert Museum (V&A), where it was used extensively (Landi 1966). This combination also caused inflexibility and affected the colour and gloss of the object (Lodewijks 1965). Meanwhile in Sweden, a similar technique was developed using Vinnapas Dispersion EP1 and silk crepeline (Bøgh et al. 1975). At the same time in North America, BEVA 371 was being investigated for lining paintings (Berger 1972), and the flocking technique was eventually transferred to backing textiles (Muir and Yates 1987). Also in North America, the mixture of AYAA and AYAC was successfully used to heat-seal deteriorated textiles onto silk crepeline. This AY combination had the advantages of a wide range of possible softening points, flexible overall support, no visual disturbance, and removability with solvents, heat or easy peeling (attachment to fibres only) (Himmelstein and Appelbaum 1976).

In the 1980s and 1990s, an ethical controversy ensued on adhesive use for textiles. Some conservators believed that stitching was the only ethical form of treatment, while others thought stitching had problems with breaking fragile threads and that adhesives, if used properly, had a place in conserving hopelessly friable and brittle objects, especially deteriorated silk flags and banners. Besides PVACs, animal glues, starches, cellulose ethers, PVAL, poly(vinyl butyral) (PVB) and acrylics were tried, each with their own unique problems and advantages. In 1984, PVACs were not recommended for textile conservation because of their low T_g (dust retention, creep), dissimilar reactions to RH variations than silk (could cause tears at glue line), and difficult removal (Masschelein-Kleiner and Bergiers 1984). Instead, starch and MCs were favoured. PVACs were further discouraged in a heat and light aging study comparing yellowing, solubility and mechanical resistance with various other adhesives (acrylics, polypropylene glycol, hydroxymethylcellulose and starch) (Verdu et al. 1984). The PVACs that were

tested tended to yellow and become insoluble, while the acrylic (Paraloid F-10) did best overall.

By 1992, the V&A was hunting for an alternative to the difficult-to-obtain Mowilith DMC2/DM5 mixture, and settled on Vinamul 3252 (Tinker 1992). This was the prelude to their adhesive testing program (Pretzel 1997). In this program, PVACs (Mowilith DMC2 alone and with Mowilith DM5, Vinamul 3252, Vinamul 3254, Vinnapas EP1 and BEVA 371) and various acrylics were tested for bond strength (peel), removability (hot peel), reversibility (cold versus hot peel), acidity, corrosiveness (staining), ease of application and ease of handling, and information was gathered on toxicity, heat-sealing temperature and T_g. In general, the dispersion PVACs (VAE, vinyl acetate/di-butyl maleate copolymer) performed best overall (if all properties were considered equal) because most were strong, removable, reversible, not acidic, non-staining (most), did not require fume extraction, and were easy to apply and handle. Their disadvantages were their high heat-seal temperatures and low T_gs (dust retention). An interesting matrix with weighting factors for adhesive selection was developed that allowed a conservator to weigh the different properties according to their needs for a specific application.

A study of BEVA 371, Clariant T1601 (a.k.a. Mowilith DMC2), Dur-O-Set E-150, Vinamul 3252 and two other acrylic adhesives on silk and nylon fabric laminates with various support fabrics found that BEVA gave the strongest bonds with most adhesive transfer after peel tests compared to the Clariant T1601, Dur-O-Set E-150 and Vinamul 3252, which had intermediate bonds, with Clariant T1601 being weaker than Dur-O-Set E-150 and Vinamul 3252 (Karsten and Kerr 2011). Doubling the concentration of the adhesives doubled the peel strength of the laminate.

Wax

Wax objects are difficult to bond and present a host of treatment issues and problems. In a study on the mechanical properties (peel and shear strength) of BEVA 371, various acrylics and a PVB for use on a wax object, BEVA displayed fairly low strengths in comparison to the others tested (Hincelin and Roche 2011). Three acrylics were found to have sufficient strength and reliability for this wax object, which was a work on waxed paper.

In another study that examined various adhesives (PVACs, isinglass and acrylics) for several types of waxes, Mowilith DMC2, a PVAC dispersion, was eliminated from use because of its difficult availability and tendency to yellow, even though the strength seemed adequate (Lang 2011). In the same study, Mowilith 30, a PVAC resin (applied 40% in ethanol/water 1:19), was favoured for some types of waxes because of its rapid drying, which meant that it needed little support while setting. However, solvents that could harm wax might be needed for its reversal, which was not good. Mowilith 50, also a PVAC resin (applied 40% in ethanol/water 1:19), was not favoured as it had lower strength than the Mowilith 30, was slower drying, needing support while setting, and also might need harmful solvents for reversal. Isinglass and the Lascaux 498 HV were found somewhat acceptable, depending on the type of wax and situation.

Paintings

In the late 1960s, the adhesive formulation BEVA 371, which is based on EVA, was developed by Berger for consolidating and lining paintings (Berger 1970). According to Berger (1972b), this adhesive suited the requirements by being:

- soluble in petroleum benzine or toluene (same as varnishes);
- non-tacky at low temperatures, but very tacky as temperature approached 70°C (heat-sealable after drying);
- easily applied with minimum heat and pressure with or without impregnation;
- strong;
- transparent;
- flexible;
- non-shrinking;
- non-crosslinking;
- compatible with waxes and tackifiers;
- able to give a wide range of viscosities;
- versatile (adheres to most surfaces);
- non-staining to textiles or paper; and
- non-yellowing and reversible after heat and light aging.

BEVA 371 Original Formula is composed of several commercial products and two solvents: Elvax 150 (EVA), Laropal K80 (earlier versions contained Ketone Resin N), Cellolyn 21 (phthalate ester of hydroabietyl alcohol), A-C Copolymer 400 (EVA), paraffin (highly refined, melting point 65°C), toluene and naphtha (Talas; Berger 1975). The high molecular weight A-C Copolymer and the Elvax provide strength and toughness, but have high softening temperatures (83°C, 116°C). The lower molecular weight Laropal and Cellolyn resins have lower softening temperatures (75–85°C, 65°C), which reduce the softening point and improve the wetting of BEVA 371. The wax protects against crosslinking (Berger 1976). More recently (in 2010), there has been a change in the formulation of BEVA 371 and it is now sold as BEVA 371b. This reformulation was necessary when

Laropal K80 was discontinued (in 2008). According to the formulator, Conservator's Products Company, BEVA 371b contains an aldehyde ketone resin instead of the Laropal K80 (Chludzinski 2010).

The peel strength of BEVA 371 hot-table linings increases with increasing temperature of application (50°C to 70°C), the number of coats (three to six), the lining time or thickness, and decreases if the lining fabric is impregnated with BEVA beforehand (Pullen 1991; Hardy 1992; Gayer 1992; Forest 1997). Operator variation, equipment eccentricities and canvas condition (weave, nap, presence or absence of size, or other contaminants) also affect peel strength. Strict temperature control is necessary as BEVA becomes molten at temperatures above 66°C and can stain (Berger 1989). Indeed, when an impregnated lining is desired, temperatures above this are used.

As a lining adhesive, BEVA 371 has exceedingly high strength in comparison to wax-resin (Daly-Hartin et al. 1993; Berger 1972b) or Plextol D360 (Hawker 1987); perhaps 2–15 times stronger than the 100 N/m peel strength that is considered adequate (Daly-Hartin et al. 1993; Katz 1985; Phenix and Hedley 1984). When BEVA was compared to Lascaux 360 HV, solvent-activated linings produced stronger bonds on canvas than heat-activated linings for both adhesives. Sizing with Paraloid B-72 tended to increase peel strength; however, bonds of BEVA 371 were weaker than Lascaux 360 HV bonds (Katz 1985).

In a lining study, BEVA 371 *linen* linings were very moisture sensitive and only moderately protected the lined painting from RH change, while BEVA 371 *polyester sailcloth* linings offered slightly better protection, but still less than the linen wax-resin lining, which afforded the best protection (Young and Ackroyd 2001). The study poses a conundrum: should lining be conducted with reversible BEVA 371/sailcloth, which yields only slight protection from moisture effects, or with wax-resin to obtain better protection against moisture, but with less reversibility?

BEVA 371 is also available as a film [25.4, 63.5 µm (1.0, 2.5 mil)], which makes for easy preparation for heat-sealing. There are also two other BEVA products available, BEVA Gel and BEVA D8. BEVA Gel is an aqueous dispersion of a VAE copolymer and a dextrin (Williams 2013b). It adheres well to canvas and polyester and reverses in water, toluene, xylene, isopropanol or ethanol (Talas c). BEVA D8 is a VAE dispersion made for strip-lining, which dries clear and colourless (Williams 2011b). Initially BEVA D8 needs aromatic mixtures for removal, but on prolonged aging it becomes soluble in alcohols (Talas b).

The peel strength of BEVA D8 (for adhering a painting to a rigid support) is weak (<300 N/m) and becomes weaker with thermal aging (Loudermilk 2005).

At the same time as BEVA 371 was being developed, another PVAC heat-seal process for lining paintings was being investigated by Rabin (1972). The technique was suggested for use when the paint film or fabric support could be affected by moisture or wax penetration. This Rabin's mixture employed AYAA, AYAC and Multiwax #445 all combined in toluene. It was supposedly non-staining and left little residue behind after lining removal. According to one study, BEVA 371 is however easier to reverse without impregnation, stronger both in strength and peel modes, and more creep-resistant than Rabin's mixture (Bantock 1980).

Numerous adhesives and mixtures (acrylics, PVACs, EVAs, polyamides, sturgeon glue mixtures and starch mixtures) have been tested for their suitability for thread-by-thread tear repair for canvases (Demuth et al. 2011). The PVACs (Lineco, Evacon-R and Mowilith D50) demonstrated adequate strength for the task. The Lineco and Evacon-R had high ultimate tensile strength, good handling and working properties, and were reversible in cold water but needed more testing before they could be recommended. The recommended adhesives were mixtures of sturgeon glue and WSP.

Panel paintings

When the mechanical behaviour of various adhesives and fillers was examined for re-joining panel paintings, the use of Evostick Resin W (PVAC) with coconut flour was found to be stronger than wood (Young et al. 2002). With the addition of rye flour or microballoons instead of coconut flour, it became weaker than wood and more suitable for degraded wood that was not to be subject to large RH changes. For slightly stronger and stiffer joints, Evostick Resin W with wood flour was suggested, but better consistency, handling and carving properties were found with the addition of microballoons or coconut flour. The presence of microballoons ensured failure within the joint.

Decorative art

In a study by Schellman and Taylor (2011) who examined consolidants for fragile gesso-type foundations in multi-layered decorative coatings, Mowilith 50 (20% in toluene) was found to be an effective penetrant but not an effective adhesive or toughener, while Mowilith DMC2 (10% in water) was a good adhesive and toughener but did not penetrate well. The isinglass and hide glue gave the most promising results.

Wood
When two PVAC dispersion wood adhesives (Bondfast and Mastercraft Carpenter's Glue) were compared to traditional hot hide glue, the results of strength loss on humidity cycling were similar (Hay 1987).

A PVAC copolymer (Vinamul 3252) showed good dimensional and chemical stability after light aging, although the pH increased (Shashoua 1989b). It retained flexibility, shrank little and was most soluble in acetone (less so in industrial methylated spirits or water). As it remained tacky upon aging, it was not recommended for surface consolidation, but was recommended for attaching paint layers to wood.

When various resins [AYAT (PVAC), Paraloid B-72 (acrylic), and Butvar B-98 (PVB)] made up in solvents were compared to a PVAC dispersion (Elmer's) for consolidating deteriorated wood, the dispersion gave the strongest bonds, and, for the most part, resins applied in polar solvents gave stronger bonds than those applied in non-polar solvents (Sakino and Schniewind 1990). In another study, PVAC resins (AYAA, AYAC, AYAF and AYAT) improved strength of deteriorated wood by 3–13%, but were not as effective as Butvar B-98 (20%) or Paraloid B-72 (16%) (Schniewind and Kronkright 1984).

Ceramics
When Rayt (likely a dispersion) and UHU (a solution), both PVACs, were assessed for bonding ceramics, they were found to be only moderately strong, had average resistance to yellowing and formed very thick bonds (Castro and Doménech Carbó 1999). UHU had a good working time, while the Rayt did not.

Friable ceramics consolidated with AYAC showed this resin to be very resistant to abrasion, but it darkened the ceramic and yielded a shiny surface (Fox 1979).

Stone
In a study on stone, PVAC resin Mowilith 50 (in acetone) was not recommended as a filler for fractures due to bubbling from acetone evaporation, but it worked well as a sealing mortar in a mixture with silicone and marble aggregate (Santamaría and Calatayud 1997).

Metals
AYAT protected silver exposed to hydrogen sulfide (low tarnishing) (Grabow et al. 2007).

In a study by Keene (1984), PVAC was shown to be ineffective at stabilizing archaeological iron. PVAC-treated samples showed signs of splitting, flaking, 'weeping' or rusting.

Although shear strength of Jade 403 on aluminum substrates decreases with increasing amounts of added WSP or carboxymethylcellulose, the mixtures are more responsive to RH change and more soluble in water, which is helpful for removal (Fulton 1988).

Other
Bainbridge (2011) has devised a method to create BEVA 371 hot-melt glue sticks for use in a glue gun. The advantages of this application method are that no solvent is used, it provides excellent gap-filling properties with no shrinkage, it provides good adhesion to many surfaces, is versatile, it has fast tack—so no clamping is required—it is a known tested material with good aging properties, and bonds are reversible with heat or aromatic solvent.

SOLUBILITY AND/OR REMOVABILITY*
PVAC resins are soluble in aromatic and chlorinated hydrocarbons (e.g. toluene, chloroform), ketones, lower alcohols (with a little water), esters (e.g. ethyl acetate), and ethylene dichloride/alcohol (20:80) (Roff and Scott 1971, p. 67). Solubility in many solvents is improved with water added in small amounts (Norris and Dragetti 1962, p. 352).

PVAC resin films swell slightly and become white and opaque on immersion in water, but regain clarity upon drying, while other PVACs with acrylic co-monomers and some surfactant-protected dispersions are more resistant to water (Jaffe et al. 1990, p. 388; Roff and Scott 1971, pp. 66–71). Often dry films cast from PVAC dispersions are not soluble in water or solvents, but will swell, aiding removability (Down 1999).

In a test on deteriorated wood, PVAC resin AYAT was the most reversible consolidant compared to Paraloid B-72 or Butvar B-98, but even with drastic removal techniques (agitated soaking or Soxhlet extraction) residue still remained in the wood, emphasizing the difficulty of removing such consolidants (Schneiwind 1988). However, in another consolidant removal experiment, PVACs (Bondfast, AYAF) were acceptably removed by Soxhlet extraction so that future bone morphological study and chemical analysis (e.g. diet isotope analysis) was possible (Unruh 1994).

* Disclaimer: The solvents listed in this section are ones that might dissolve the polymer or adhesive in question but are not necessarily recommended for conservation treatments. Some may be extremely toxic or may contain elements (e.g. chlorine) that may be harmful to certain materials in your object (artifact). Research the solvent before purchase or use to know its toxicity and chemical nature. It is strongly recommended to test all solvents before use to make sure that they do not adversely affect your object.

Poly(vinyl alcohols)

Setting mechanism	Category	Water absorption (%)	Refractive index
evaporation	thermoplastic	6–9 when conditioned to 65% relative humidity	1.50–1.53
Glass transition temperature (°C)	**Viscosity mPa.s (20°C)**	**pH**	**Coefficient of linear expansion /°C**
70–85	5–40 (4%) 1500–100,000 (20%) depends on grade	4.5–7	$70–100 \times 10^{-6}$

(Horie 2010, p. 143; Sekisui; Jaffe and Rosenblum 1990; Finch 1973, p. 168; Roff and Scott 1971, pp. 72–78)

Abbreviation

PVAL is the preferred abbreviation for poly(vinyl alcohol) according to the International Union of Pure and Applied Chemistry (IUPAC), which is the world authority on chemical nomenclature and terminology (IUPAC 1987; ISO 1043-1 2011). It is the abbreviation used for poly(vinyl alcohol) throughout this book. Other abbreviations are PVOH (ASTM D1600-08 2008) and PVA. Since PVA has been used to represent both poly(vinyl alcohol) and poly(vinyl acetate), it is no longer used as an abbreviation for either.

Types, synonyms and/or trade names

Celvol, Elvanol, Fluka, Gelvatol, Mowiol, Polyviol, Rhodoviol, Selvol, Vinarol, Vinol

Chemistry and/or preparation

Poly(vinyl alcohol) (PVAL) (**Figure 1**) is made by the alcoholic hydrolysis of poly(vinyl acetate) (PVAC) in which acetate groups are replaced by hydroxyls. PVAL is graded according to the degree of hydrolysis and the molecular weight (Roff and Scott 1971, p. 73). Most products have un-hydrolyzed acetate groups present because fully hydrolyzed PVAL is difficult to make (Jaffe and Rosenblum 1990). Thus, PVAL is really a copolymer of vinyl alcohol and vinyl acetate (Horie 2010, p. 142).

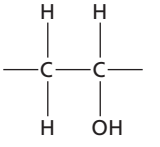


Figure 1. Simplest fundamental unit of PVAL (Roff and Scott 1971, p. 72).

History and uses

PVAL was first prepared in Germany in 1924 and further developed in Germany, Japan and the United States from 1930 to 1950 (Roff and Scott 1971, p. 78).

Commercially, PVAL has been used for flexible sheeting, tubing, protective aprons and gloves, polarizing screens, gas impermeable fabric coatings, adhesives for porous materials (e.g. leather, cork, cloth, paper), pigment binders, textile and paper sizes, thickening and emulsion stabilizing agents, surgical sutures, cosmetics, binder for ceramics, fibres (e.g. for fabrics, carpets, cord, bristles), adhesive for cellulosics (e.g. wood, paper), and remoistenable adhesives (e.g. stamps, envelopes) (Ebnesajjad 2008, p. 115; Jaffe and Rosenblum 1990; Shields 1984, p. 72; Roff and Scott 1971, pp. 78–79).

PVAL has been used in conservation applications since the 1950s as a textile size and consolidant, heat-seal adhesive for paper, adhesive for parchment, strengthener for paper, consolidant for paintings and stone, pigment fixative, inpainting medium, release agent for moulding and fill for lacquered objects (Horie 2010, pp. 144–145; Webb 1994; De Witte 1976/77). It has been used in palaeontology as a labelling separator adhesive and for surface

consolidation (Elder et al. 1997). More recently, PVAL fibre has been used to reinforce cement-based composites for the restoration of buildings and monuments (Knapen and Van Gemert 2007; Atsuhisa et al. 2006).

CHARACTERISTICS, LIMITATIONS AND/OR AGING STUDIES

GENERAL

PVAL is a white- or cream-coloured powder that dissolves in water and produces tough, transparent films upon drying (Roff and Scott 1971, p. 72). The films are highly impermeable to most organic vapours, but are affected by moisture and high relative humidity (Roff and Scott 1971, p. 72). As PVAL is very hygroscopic, it should not be stored or used in humid places (De Witte 1976/77). It is the easy solubility in water that made PVAL so appealing for conservation applications (De Witte 1976/77). Few synthetic adhesives are completely water soluble. However, by the 1960s, it fell out of favour because it crosslinked and became insoluble under certain circumstances (see *Solubility and/or Removability* section below) (Horie 2010, p. 145).

Physical properties such as flexibility, water sensitivity and strength of film can be affected when molecular weight and percent hydrolysis of PVAL are altered (**Table 1**). Viscosity of PVAL solutions is affected by molecular weight, concentration and temperature, but not degree of hydrolysis (Jaffe and Rosenblum 1990). Using low molecular weight grades decreases viscosity and insolubility (De Witte 1976/77).

Adding PVAL to dispersion adhesives reduces their surface tension and thus improves their wetting (Jaffe and Rosenblum 1990). Upon exposure to heat, PVAL yellows (De Witte 1976/77); however, it is reported to be light stable (Horie 2010, p. 144; De Witte 1976/77; Roff and Scott 1971, p. 77).

MICROBIAL RESISTANCE

PVAL (Fluka 72000) did not increase the biodegradability of paper, parchment or leather when tested for book restoration (Gallo et al. 1989). Roff and Scott (1971, p. 77) suggest that PVAL is generally unaffected by bacteria, fungi or insects, but solutions may need a preservative to prevent mould from growing.

PAPER AND TEXTILES

PVAL can react with the hydroxyls in paper and textiles (accelerated by heat) and bond irreversibly to these substrates (De Witte 1976/77). Nevertheless, in the last 20 years, PVAL was investigated for use in paper conservation. One series of studies examined low molecular weight PVAL before and after thermal aging and found that it was suitable for reinforcing and lining paper (6% solution), mending tears (8%) and fixing pigments (5%) (Bicchieri and Mucci 1996; Bicchieri et al. 1993). Another study found that upon thermal aging PVAL (Vinarol DV) improved mechanical properties of bleached pulp sheets but not brightness levels while, if mixed with poly(ethylene glycol), both mechanical and brightness factors were improved (Nada et al. 2000). Yet another study showed that resizing using a PVAL-borax system

TABLE 1. PROPERTIES OF PVAL
(Jaffe and Rosenblum 1990)

If % *hydrolysis* held constant	
Decreasing molecular weight increases: flexibility water sensitivity ease of solvation	**Increasing molecular weight increases:** viscosity tensile strength water resistance adhesive strength solvent resistance
If *molecular weight* held constant	
Decreasing % hydrolysis increases: flexibility water sensitivity adhesion to hydrophobic surfaces	**Increasing % hydrolysis increases:** water resistance tensile strength solvent resistance adhesion to hydrophilic surfaces

(Mowiol grade 28-99) helped to improve the strength and aging resistance of alum-rosin sized paper in comparison to PVAL alone (Basta 2004). Meanwhile in textile conservation, PVAL was rejected because of its insolubility upon aging in the presence of salts, tannins and certain colourants (Masschelein-Kleiner and Bergiers 1984).

GILDING

Various PVALs were tested as a substitute for animal glue in gilded wood conservation and it was found that they performed satisfactorily and that the low molecular weight, low hydrolysis type (Rhodoviol 4/125) was most suitable for this purpose (Hebrard and Small 1991).

SOLUBILITY AND/OR REMOVABILITY*

Fully hydrolyzed PVAL (98–100%) must be boiled to dissolve in water, while at lower hydrolyzed levels (87–89%) PVAL dissolves in both hot and cold water (Jaffe and Rosenblum 1990; Roff and Scott 1971, p. 71). At even lower levels (75–80%), PVAL is soluble in cold water, but precipitates upon heating (Jaffe and Rosenblum 1990). Decreasing molecular weight and particle size increases the solubility of the PVAL (Jaffe and Rosenblum 1990).

PVAL is unaffected by many organic solvents (e.g. xylene, methanol, acetone), oil and grease (Jaffe and Rosenblum 1990; Roff and Scott 1971, p. 74). Lower alcohols can be added (up to 50%) to PVAL water solutions without precipitation (Jaffe and Rosenblum 1990).

Although PVAL has been estimated to remain soluble in a good museum environment for 300 years, this is questionable as it easily crosslinks due to the hydroxyl groups that are very reactive (Horie 2010, p. 144; De Witte 1976/77; Roff and Scott 1971, pp. 73–74). PVAL has been shown to crosslink to an insoluble material under mildly acidic or alkaline conditions, and with certain polyformaldehydes (e.g. urea-formaldehyde) and acid catalysts, metal chelating salts, pigments (e.g. congo red), dyes and heat (>100°C) (Horie 2010, p. 144; Jaffe and Rosenblum 1990). Crosslinking probably accounts for the insolubility of some PVAL-stabilized PVAC dispersions (Horie 2010, p. 144).

* Disclaimer: The solvents listed in this section are ones that might dissolve the polymer or adhesive in question but are not necessarily recommended for conservation treatments. Some may be extremely toxic or may contain elements (e.g. chlorine) that may be harmful to certain materials in your object (artifact). Research the solvent before purchase or use to know its toxicity and chemical nature. It is strongly recommended to test all solvents before use to make sure that they do not adversely affect your object.

Poly(vinyl butyrals)

Setting mechanism	Category	Water absorption (%)	Refractive index
evaporation	thermoplastic	3–5	~1.49
Glass transition temperature (°C)	**Viscosity (mPa.s)**	**pH**	**Coefficient of linear expansion /°C**
48–68 depending on hydroxyl content			$80–220 \times 10^{-6}$

(Tímár-Balázsy and Eastop 1998, pp. 304–331; Shields 1976, p. 299; Roff and Scott 1971, p. 84)

Abbreviation

PVB poly(vinyl butyral)

Types, synonyms and/or trade names

Butacite, Butvar, Mowital, Pioloform, Rhovinal

Chemistry and/or preparation

Poly(vinyl butyral) (PVB) (**Figure 1**) belongs to a family of resins called poly(vinyl acetals). These acetals are made by hydrolyzing poly(vinyl acetate) (PVAC) and then treating the resulting poly(vinyl alcohol) (PVAL) with an aldehyde under acidic conditions to produce poly(vinyl formal), if formaldehyde is used, poly(vinyl acetal), if acetaldehyde is used, or PVB, if butyraldehyde is used (Roff and Scott 1971, pp. 82–83). This is somewhat confusing because poly(vinyl acetal) can refer to the family of resins or to the specific resin made with acetaldehyde. The resulting polymer is a terpolymer—for PVB, it is a terpolymer of vinyl butyral, vinyl alcohol and vinyl acetate.

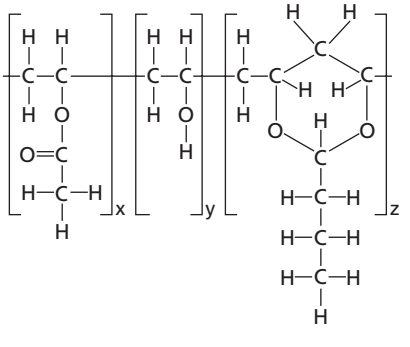


Figure 1. Terpolymer of PVB (Horie 2010, p. 146; Roff and Scott 1971, p. 82).

History and uses

PVB was first developed in Canada in the late 1920s (Roff and Scott 1971, p. 86).

PVB has been used commercially as a primer for metal to improve paint adhesion, as a coating for fabrics, and as an adhesive for car windshield laminates (Farmer and Jemmott 1990; Roff and Scott 1971, p. 86). PVB has excellent adhesion to a variety of substrates, especially glass, wood and textiles (Farmer and Jemmott 1990).

In conservation, PVBs have been used as consolidants for textiles, fossils, wood and paper; and as adhesives for wood, fossils, glass and paint films (Horie 2010, pp. 146–147; Howie 1984). PVB replaced poly(vinyl acetal) in AJK dough, which was a modelling material for conservation (Horie 2010, pp. 146–147).

Characteristics, limitations and/or aging studies

General

The properties of PVB depend on molecular weight and proportions of the three monomer units in the copolymer (**Table 1**).

PVB is a white powder that deposits clear transparent films from solution. Films of PVB are only slightly affected by sunlight and exterior weathering (Roff and Scott 1971, p. 85). To confirm the stability of PVB, one study aged films (Butvar B-98) thermally and under ultraviolet (UV) radiation and indeed little change in chemical (infrared spectra) and physical (colour, hardness, solubility) properties was noted (Toutloff 1999).

TABLE 1. PROPERTIES OF SOME PVBS
(Butvar 2006)

Property	Butvar B-76	Butvar B-98
molecular weight	90,000–120,000	40,000–70,000
acetate (%)	0–2.5	0–2.5
hydroxyl (%)	11.5–13.5	18.0–20.0
butyral (%)	88	80
viscosity (mPa.s) (10% ethanol)	200–450	75–200
refractive index	1.485	1.490
water absorption (% 24 h)	0.3	0.5

PVB with a high number of hydroxyl groups (17–21%) (e.g. Butvar B-98) usually has a high glass transition temperature (T_g) (62–68°C), while PVB with a low number of hydroxyl groups (0–13%) (e.g. Butvar B-76) has a lower T_g (48–55°C) (Tímár-Balázsy and Eastop 1998).

When mechanical properties of a film of PVB (Butvar B-98 in ethanol) were followed over a two-month period, PVB was initially fairly extensible, but once the ethanol had disappeared, it became relatively inextensible (Wolbers 2008). Also, it initially gained strength, which was lost after the two months.

MICROBIAL RESISTANCE

Roff and Scott (1971, p. 85) state that PVB polymers are not attacked by bacteria, fungi or insects; however, Mowital B 20 H exhibited only medium resistance to microbial growth when tested on stone in comparison to 15 other acrylic, PVAC, silicone and Dammar polymers (Koestler 2000).

PAPER

PVB (3%) was found to strengthen paper (increased strength by a factor of four times that of untreated paper), which did not alter upon thermal aging or upon exposure to light (Eliazyan et al. 2001).

TEXTILES

PVBs were rejected in one study for textile conservation because they contain PVOH, which could crosslink either to itself or to the textile and become insoluble upon aging; they have an affinity for metals and could react with mordants and dyes; and they were shown to degrade silk (Tímár-Balázsy and Eastop 1998; Masschelein-Kleiner and Bergiers 1984). Another study investigating textile consolidants found that one PVB (Mowital B 30 H) gave good colour stability, was fairly reversible and retained flexibility upon heat aging, while another PVB (Rhovinal B10-20) became slightly yellow and lost solubility and flexibility upon aging (Blackshaw and Ward 1983).

WAX

As an adhesive for a waxed-paper object, PVB (Mowital B 60 HH) was found to give moderately thick, slightly stiff and brittle films that were fairly strong in shear strength compared to the others tested (various acrylic adhesives and BEVA 371), but had a wide scatter of results indicating unreliability of the strength (Hincelin and Roche 2011). Its peel strength on wax was the lowest of all adhesives tested and thus the Mowital was not selected to conserve the waxed-paper object.

WOOD

PVB (Butvar B-98) has been tested for the consolidation of degraded wood and furniture (Spirydowicz et al. 2001; Grattan 1980). It was shown to coat the cell walls efficiently, to increase the mechanical strength and hardness of the wood, to be stable under various aging conditions, and to be soluble in alcohols. The disadvantages were high viscosity in solutions (methanol better than ethanol) and staining, but it was noted that most consolidants stain wood (Grattan 1980). In other studies, PVB (Butvar B-98) has been shown to improve the strength of deteriorated wood by 20% (Schniewind and Kronkright 1984; Wang and Schniewind 1985).

FOSSILS

PVBs have been used for the consolidation and repair of fossils, but there have been adhesion difficulties with the higher hydroxyl types (e.g. Butvar B-98) (Howie 1984).

STONE

In a study of protectants for stone (limestone, sandstone), PVB (Butvar B-73) was imperceptible as a coating, was reversible, gave no colour change upon exposure to light (UV radiation) or upon thermal aging, withstood freeze-thaw cycling, and did not absorb water (Whiteford 1982). However, it did not do well in an acidic environment. Except for this difficulty, it faired relatively well when compared to the others tested in this study [Paraloid B-72, Ethocel, Lexan 121 (a polycarbonate resin), Saran F310].

METALS

In a study of protective coatings for silver exposed to hydrogen sulfide, PVB (Butvar B-98) was found to only moderately prevent tarnish compared to PVAC, acrylic and cellulose nitrate coatings, which were better (Grabow et al. 2007).

SOLUBILITY AND/OR REMOVABILITY*

Generally, PVBs are soluble in alcohols, glycol ethers, acetone, aromatic hydrocarbons, esters, ethylene dichloride and tetrahydrofuran (Farmer and Jemmott 1990; Roff and Scott 1971, p. 83). However, solubility is affected by the number of hydroxyls in the PVB. For instance, PVB with a high number of hydroxyl groups (17–21%) (e.g. Butvar B-98) is soluble generally in very polar solvents [e.g. *n*-butanol, diacetone alcohol, dimethylformamide (DMF), 95% ethanol, 95% isopropanol, methanol—but not acetone, methylethyl ketone (MEK) and toluene], while PVB with a low number of hydroxyl groups (0–13%) (e.g. Butvar B-76) is soluble in a wider range of solvents (e.g. acetone, *n*-butanol, diacetone alcohol, DMF, 95% ethanol, 95% isopropanol, MEK, toluene—but not methanol) (Tímár-Balázsy and Eastop 1998).

PVBs are not affected by aliphatic hydrocarbons, turpentine, vegetable oils, water, cold alkalis and dilute acids (Roff and Scott 1971, p. 83).

Heat aging of PVB (Mowital B 30 H, Rhovinal B10-20) causes loss in solubility (Blackshaw and Ward 1983).

In a study on the reversibility of wood consolidation treatments using extreme methods of extraction (Soxhlet extraction and solvent soaking with agitation), PVB (Butvar B-98) was found to be the least removable compared to AYAT and Paraloid B-72 (Schniewind 1988).

* Disclaimer: The solvents listed in this section are ones that might dissolve the polymer or adhesive in question but are not necessarily recommended for conservation treatments. Some may be extremely toxic or may contain elements (e.g. chlorine) that may be harmful to certain materials in your object (artifact). Research the solvent before purchase or use to know its toxicity and chemical nature. It is strongly recommended to test all solvents before use to make sure that they do not adversely affect your object.

Acrylics

Setting mechanism	Category	Water absorption (%)	Refractive index
evaporation (dispersions or resins dissolved in solvent)	thermoplastic	0.3–0.4 (immersed 24 h) ~2 (prolonged immersion)	PMA 1.47 PMMA ~1.49 PBMA or PEMA 1.48 PIBMA 1.475
Glass transition temperature (°C)	**Viscosity (mPa.s)**	**pH**	**Coefficient of linear expansion/°C**
PBA -54 PBMA 20 PEA -22 PEMA 65 PMA 9 PMMA 105	typical dispersion about 50–300	7–9 liquid dispersions 6–7 water-extracted dry films (unaged)	PMMA 3–8 × 10^{-5}

(Down et al. 1996; Gehman 1990; Shields 1976, p. 299; Roff and Scott 1971, p. 91)

Abbreviations

BA, PBA	butyl acrylate or *n*-butyl acrylate, poly(butyl acrylate) or poly(*n*-butyl acrylate)
EA, PEA	ethyl acrylate, poly(ethyl acrylate)
MA, PMA	methyl acrylate, poly(methyl acrylate)
BMA, PBMA	butyl methacrylate or *n*-butyl methacrylate, poly(butyl methacrylate) or poly(*n*-butyl methacrylate)
IBMA, PIBMA	isobutyl methacrylate, poly(isobutyl methacrylate)
EMA, PEMA	ethyl methacrylate, poly(ethyl methacrylate)
MMA, PMMA	methyl methacrylate, poly(methyl methacrylate)

Types, synonyms and/or trade names

Acrifix, Acrilem, Acronal, acrylate, Acryloid*, Acrysol, Elvacite, Lascaux, Loctite, Lucite, methacrylate, Neocryl, Norland, Paraloid*, Perspex, Plexiglas, Plexisol, Plextol, Primal (name used for Rhoplex in Europe), Rhoplex (name used for Primal in North America), Sokrat, Solucryl, Tensol, Texicryl, Vinacryl

Chemistry and/or preparation

Acrylic adhesives are based on acrylates derived from acrylic acid, and methacrylates derived from methacrylic acid (**Figure 1**) (Horie 2010, p. 153).

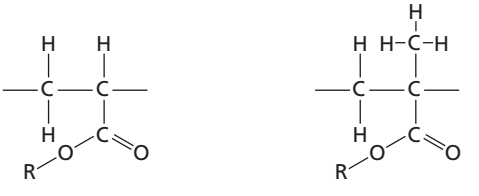


Figure 1. Acrylic monomer units.

* Prior to 1997, "Paraloid" was the trade name given to a series of acrylic resins made by the Rohm and Haas Company that were available in Europe while the same products available in North America were called "Acryloid". In 1997, Rohm and Haas adopted the name "Paraloid" as the sole name for all international markets (CAMEO 2010b; Gottsegen 2006, p. 81). For this book, the name "Paraloid" has been used throughout, even if the original study cited used an "Acryloid" product. At the time of publication, the Dow Chemical Company and Rohm and Haas had merged, and Dow had also acquired the Union Carbide Corporation (Watson 2009). Over the years, there have been many such mergers, or takeovers, of adhesives companies, as well as product name changes. It is sometimes difficult to trace the products that are available today (even if they are the same product) because the names have changed or the products have disappeared altogether.

HISTORY AND USES

Work on acrylates and methacrylates began in about 1843, but Röhm began the main study on these polymers in 1901 (Roff and Scott 1971, p. 94). Acrylate commercial production commenced in about 1927 in Germany, while methacrylate development began in the 1930s in Canada (Roff and Scott 1971, p. 94).

Acrylics are used as resins in sheet form for windows and light fittings; as solvent solutions for varnishes and printing inks for rubber, leather and paper; and as dispersions for paints, adhesives and textile finishes (Roff and Scott 1971, p. 94).

In conservation applications, Lucite was first used as a picture varnish in the 1930s (Horie 2010, p. 159). Later, acrylics were used as consolidants for pigments, paper, wood, leather, plaster, stone, earth, bone, wall paintings and fossils; as adhesives (wet, heat-set, pressure-sensitive, contact) for paper, textiles, wood and relining canvases; and as lacquers for silver, mosaics and iron (Horie 2010, pp. 159–167).

CHARACTERISTICS, LIMITATIONS AND/OR AGING STUDIES

GENERAL

Acrylics are clear, colourless, lightfast, resistant to weathering and aging, easily formulated, water-resistant, non-toxic, and have low temperature performance and a broad adhesion spectrum (Gehman 1990; Roff and Scott, pp. 87–94). Acrylic adhesive products come in different forms, for example, solids (pellets), organic solutions, aqueous dispersions, pressure-sensitive tapes or carrier-free films. The polymers range from hard and glassy to soft and tacky. The methacrylates have higher glass transition temperatures (T_gs) than the acrylates (**Table 1**), which tend to be tackier and can collect dust.

The T_g for these polymers is influenced by (Gehman 1990):

- how far apart side chains are separated from one another (the farther apart, the more mobility);
- the methyl group in methacrylates that causes more steric hindrance compared to the acrylates;
- branching;
- copolymerization; and
- crosslinking.

The methacrylates are cohesively stronger and less flexible than the acrylates. This strength decreases with increasing length of the alkyl chain. The higher alkyl methacrylates and all the acrylates tend to crosslink under ultraviolet (UV) radiation, but the methyl and ethyl groups are not as involved in crosslinking as the amyl, butyl and propyl groups (Feller et al. 1981; Feller 1957). This is one reason why Paraloid B-72 has performed well in crosslinking tests compared to the higher alkyl esters; it is an *ethyl* methacrylate/*methyl* acrylate copolymer (Feller et al. 1981). Poly(methyl methacrylate) (PMMA) is more resistant to the effects of aging because of the methyl group, which prevents reaction with oxygen and crosslink formation (Ďurovič et al. 1991).

TABLE 1. PROPERTIES OF SOME ACRYLIC POLYMERS
(Gehman 1990)

Acrylic	Tensile strength (MPa)	Elongation (%)	T_g (°C)
Methacrylates			
methyl	62	4	105
ethyl	34	7	65
butyl	7	230	20
Acrylates			
methyl	7	750	9
ethyl	0.2	1800	-22
butyl	0.02	2000	-54

ULTRAVIOLET-CURING ACRYLICS

UV-curable adhesives may be epoxy-based or acrylic-based (Lumen Dynamics Group, Inc.). The epoxy-based adhesives cure by a cationic process, while the acrylic-based adhesives cure by a free radical mechanism (Lumen Dynamics Group, Inc.). These adhesives are one-part products that may contain monomers that control viscosity, crosslinking and cure speed; prepolymers that mainly determine mechanical and adhesive properties; photoinitiators that absorb the UV photons and transfer energy to the cure process; fillers that are added for reinforcement or to reduce costs; adhesion promoters; and stabilizers (Lumen Dynamics Group, Inc.; Haddon and Smith 1991). They cure quickly when exposed to UV radiation without emitting solvent vapours, as is the case with adhesives that set by solvent evaporation. For these adhesives to work properly, the UV radiation must reach the adhesive through the substrate, so at least one transparent substrate is required of limited depth. If this is not the case, then the adhesive could under-cure and remain tacky (Haddon and Smith 1991).

UV-curable acrylics (Norland NOA 61, Norland NOA 63) have been tested and recommended for glass conservation where colour change was undesirable (Robson 1987). In this study, they retained colour and clarity and could be injected by syringe into cracks easily. Another study found that Loctite 350 had superior bond strength and good handling properties, but yellowed slightly upon light exposure and thermal aging, and became insoluble (Bradley 1990b). It was, however, removable with solvents and mechanical action.

Two photosensitive adhesives (Loctite 3492, Holdtite ST3222) were compared to Hxtal epoxy for tensile strength, stability and reversibility (Swierenga 2000). The photosensitive adhesives were stronger than the Hxtal, but discoloured after exposure to UV. Reversibility of all three was about equal.

ANAEROBIC ACRYLICS

Anaerobic adhesives contain acrylic monomers and resins and are blends of dimethacrylates or diacrylates with hydroperoxide initiators and other ingredients present (Rooney and Malofsky 1990). They remain fluid in air, but polymerize when pressed between close-fitting surfaces (e.g. metal, glass or ceramics) where oxygen is excluded (Roff and Scott 1971, p. 98). The cured adhesive is highly crosslinked. They contain no solvents and clean-up of excess adhesive outside the bond area is easy as it does not set (Rooney and Malofsky 1990). Frequently, artifact surfaces are not smooth and close-fitting, so anaerobic adhesives would not work well on them.

PRESSURE-SENSITIVE ADHESIVES

Pressure-sensitive adhesives (PSAs) can be composed of many different polymers, including natural rubbers, polyisobutylene, poly(vinyl ether), synthetic rubbers, polyurethane and acrylics (Gehman 1990). They form a permanently tacky film after the solvent has evaporated or when they cool, if they are hot-melt. Once dry, they form a bond with the substrate when gentle pressure is applied; no solvents or heat are required (Rotunda 1977). Many PSA tapes are coated on one side or both sides of a carrier that could be paper, cloth (linen), polyester, polypropylene, polyethylene, poly(vinyl chloride) (PVC), cellulose acetate, foil or cellophane (Down et al. 2006). Tapes with cellophane carriers can be brittle when dry, be soft when wet, shrink with time and give off harmful volatiles (O'Loughlin and Stiber 1992). Those with cellulose acetate carriers can discolour, lose strength and also give off harmful volatiles (O'Loughlin and Stiber 1992). PVC carriers usually contain plasticizers that can migrate into the substrate.

Acrylic PSAs are pure polymeric systems with few additives, while rubber PSAs can have added tackifiers and antioxidants (Fries 1982). In comparison to rubber PSAs, acrylic PSAs are more inert to oxidation, clearer, more resistant to darkening and staining, better at low temperatures, and adhere better to polar surfaces than non-polar surfaces.

"Archival" PSA tapes began appearing in the 1970s, but they were not manufactured with acrylic adhesives until much later. Acrylic PSAs can be highly crosslinked, which makes them stronger, stiffer and less tacky (e.g. reversible tape such as Post-It Notes, 3M Removable Magic Tape) (O'Loughlin and Stiber 1992). Certain crosslinking additives cause adhesive strengthening and difficult removal with time (O'Loughlin and Stiber 1992). Acrylic PSAs are subject to creep, can cause paper to become transparent, and may cause inks to bleed. Vinyl acetate/acrylic ester copolymer PSAs display high tack, but have poorer aging properties than acrylic PSAs and the adhesive bond may strengthen with time (O'Loughlin and Stiber 1992).

PSAs can be permanent or removable (Bentley 1997). Permanent PSAs are impossible to remove without damage to the substrate, while removable PSAs are removed without damage or residue, although some do leave residues. Permanent PSAs adhere to more materials than removable PSAs. Removable PSAs adhere better to smooth surfaces than rough or nap surfaces, increase in strength with time, decrease in strength at low temperatures, and increase in strength at higher temperature. Some may contain plasticizers or tackifiers that can bleed and stain after application (Norman 2001).

Of 80 acrylic PSA products tested in a screening program for a Canadian Conservation Institute tapes and heat-set tissues project, about 55% had a neutral composite pH (pH 6–8, adhesive and carrier together), about 40% were acidic (pH<6), with 11% being very acidic (pH<4), and about 5% were alkaline (pH>8) (Down et al. 2011, 2013b). So, being an acrylic PSA does not necessarily ensure neutrality. In a continuation of this study that selected 19 of these 80 acrylic PSA products for further testing (the majority selected being neutral), most failed the photographic activity test (PAT) and many of the carriers discoloured upon oven aging [28 days, 80°C, 65% relative humidity (RH)] (Down et al. 2011). None of the colour from the carrier or adhesive sank through to the other side of a paper substrate, except for two products that showed only very slight colour change. As well, it was found that most tapes were only removable

from paper (1870s periodical paper and resin-coated photographic paper) in more toxic solvents such as toluene, ethyl acetate and acetone, as opposed to water, ethanol or mechanical removal.

Some PSA tapes have been developed for industrial structural applications that are as strong as a flexible epoxy resin and maintain this strength after exposure to high humidity or water immersion (Bennett et al. 1996). For example, structural tape 3M VHB was used to secure metal plates and display cases to marble flooring in a gallery when regular screws would have damaged the floor (Anonymous 1999). The tape did not stain the floor, and the tape and metal plates were easily popped-off afterwards by twisting nylon screws in pre-drilled threaded holes.

PSA tapes have a recommended shelf life of 6 to 12 months in temperate climates and should not be exposed to sunlight nor corrosive or solvent fumes (Rotunda 1977).

MICROBIAL RESISTANCE

Although handbooks (Gehman 1990; Roff and Scott 1971, p. 93) have suggested that acrylics are not subject to microbiological attack, some are. In a microbial study of various stone adhesives, Rhoplex AC-234, an acrylic dispersion, had the highest resistance to supporting microbial growth of all tested, while Paraloid B-72 showed only medium resistance, and Paraloid F-10 was one of the least resistant (Koestler 2000). Pre-testing should be conducted prior to use, especially if the substrate is heavily contaminated with micro-organisms.

ACRYLIC CONSERVATION STUDIES

General

Dispersions of acrylics [Texicryl 13-002; Plextol B500; Primal CA-12, Primal AC-33, Primal AC-634 and Primal AC-634 plus thickeners, i.e. toluene, ASE60 or Natrosol (hydroxyethylcellulose)], and poly(vinyl acetate) (PVAC) homopolymers and copolymers were tested under natural dark aging (10–25°C), natural light aging through window glass, sunlight aging in a box with various filters, fluorescent light aging and thermal dark aging (59°C and 83°C) for weight, colour, solubility, tensile strength and pH changes (Howells and Burnstock 1985). All of the acrylics had good flexibility. Primal AC-33, Primal AC-634 with Natrosol, Texicryl 13-002 and Plextol B500 yellowed or were unstable to heat. Primal AC-634 and Primal CA-12 had restricted solubility, while the other acrylics had wider solubility.

Six weeks after casting films of Paraloid B-72 from trichloroethane, 7% solvent remained in the film, which had the effect of plasticizing it. Films cast from acetone or toluene only retained 0.7% solvent (Hansen 1995).

Acrylic (Paraloid F-10, Primal N-580, Plexisol), PVAC and hydroxymethylcellulose resins were tested by thermal aging and exposure to light for durability and long-term reversibility (Wächter 1995). Paraloid F-10 proved to be the most durable in these tests.

Canadian Conservation Institute study

In a study (Down 2015, 2009; Down et al. 1996) evaluating pH, emission of harmful volatiles, strength, flexibility and yellowing of 25 acrylic adhesives under natural dark aging (22°C, ~45% RH) and exposure to fluorescent light (190 µW/lm, 700–800 lux), it was found that:

- As far as *pH* was concerned, the acrylics were generally fairly neutral as a group, however, light exposure tended to make the adhesives more acidic than dark aging, and the pH of the dispersions were in the alkaline range (pH>8), which was not surprising as manufacturers typically add ammonia to their products for stabilization purposes. The Rhoplex AC dispersions had an alkaline pH to start (pH about 9), but after one month drying, the dry-film-extracted pH fell into the neutral range. The dry films then became acidic after aging (pH as low as 3.5). This is a drastic change from wet dispersion to aged dry film and therefore these adhesives would not be suitable for pH-sensitive objects. On the other hand, the Rhoplex N pressure-sensitive dispersions displayed somewhat better pH properties in that most of the dispersion and dry-film-extracted pH measurements were in the acceptable neutral range (5.5–8), only falling to 5 or 4.5 after 15 years of light aging or 27 years of dark aging. It must be remembered, however, that these Rhoplex N products tend to crosslink with time, as stated in the manufacturer's data sheet (Rohm and Haas 1980). The acrylic products that were neutral and showed the least change in pH after 15 years of light aging and 27 years of dark aging were Paraloid B-44S, Paraloid F-10, Paraloid B-82, Elvacite 2013, Elvacite 2028, Paraloid B-99 and Paraloid B-67.
- Concerning the *emission of harmful volatiles*, the acrylic adhesives did not release any harmful volatiles except for Lascaux 360 HV, which released a very low amount of acetic acid.
- For the acrylic adhesives, aging increased the *flexibility* of about half of the products tested and decreased the flexibility of the other half.

The most flexible products were Rhoplex N-580, Lascaux 360 HV, Rhoplex N-560, Rhoplex N-1031, Rhoplex N-619, Rhoplex LC-40, Rhoplex AC-234 (and the mixture with AC-73), Unsupported Texicryl, Paraloid C-10-LV, Rhoplex AC-235, Rhoplex AC-33 and Pliantex. Many of the pressure-sensitive dispersion adhesive dry films (e.g. Rhoplex N-580, Lascaux 360 HV) were very weak cohesively and should not be used for structural applications as they might creep under load.

- Exposure to light caused the acrylic adhesives to *yellow* more quickly than dark aging, but, as a group, the acrylics tended not to yellow excessively, with Paraloid B-48S and Paraloid B-67 being the best of all tested after 15 years of light aging and 27 years of dark aging.
- The *overall results* assessed together suggested that Paraloid F-10, Rhoplex AC-33, Rhoplex AC-235, Rhoplex AC-234 (and the mixture with AC-73) and Paraloid B-82 ranked the best overall according to the criteria specified, with Paraloid B-72, Lascaux 360 HV, Paraloid B-67, Pliantex, Paraloid B-44S, Rhoplex N-560 and Paraloid NAD-10 following somewhat behind. Removability was not tested, however, and even the best of these had some drawbacks. For instance, Paraloid F-10 yellowed somewhat and became brittle upon light aging. Rhoplex AC-33, Rhoplex AC-235 and Rhoplex AC-234 (and the mixture with AC-73) became acidic upon dark and light aging and also had some yellowing issues. Paraloid B-82, Paraloid B-67 and Paraloid B-72 were either too brittle to measure or became brittle upon aging, while Lascaux 360 HV was weak cohesively and became slightly acidic upon light aging.

Paraloid B-72

Paraloid B-72 is a well-studied resin for conservation applications and is used in almost every subdiscipline. It is a copolymer resin with 70% ethyl methacrylate and 30% methyl acrylate (CAMEO 2013; Down et al. 1996). As can be seen, it is included in many of the studies mentioned in this chapter and generally does quite well in most of the evaluations. It is reputed to be a stable material, and remains soluble in non-polar solvents after aging (Phenix 1993, 1992, 1992b). Its drawbacks can be low refractive index and high gloss (but gloss can be modified). It is a good binding medium for pigments, but can be viscous in aromatic solvents and becomes sticky and stringy quickly. It has limited wet tack and flexibility, although cellulose ethers have been added to improve flexibility, and increase viscosity and tack.

Lascaux 360 HV, Lascaux 498 HV and Lascaux 303 HV

Acrylic dispersions Lascaux 360 HV and Lascaux 498 HV are also used frequently in conservation applications and are included in many research studies mentioned in this chapter and book. They are composed of copolymers of butyl acrylate and methyl methacrylate, with Lascaux 360 HV containing more butyl acrylate than the Lascaux 498 HV (Down et al. 1996). According to the Lascaux company, they remain soluble in acetone and toluene and are mechanically reversible (Lascaux 1999). They are viscous, but still can impart moisture to soften a substrate if needed. Lascaux 360 HV (T_g = -8°C) produces a soft flexible tacky film at room temperature that can be used like a PSA or can be heat-reactivated at low temperatures (50°C). Lascaux 498 HV (T_g = 13°C) produces flexible stiffer films with only slight tackiness (Lascaux 1999). The two can be mixed to produce the tack and flexibility desired.

In 2012, Lascaux 360 HV was discontinued by the Lascaux company because the binder was discontinued by the manufacturer. A new product, Lascaux 303 HV, was substituted in its place. Lascaux 303 HV contains 2-ethylhexyl acrylate/ethyl acrylate copolymer with greater than 50% 2-ethylhexyl acrylate (Williams 2013). The dry-film-extracted pH of Lascaux 303 HV is 7.77 ± 0.06 (Down 2013). According to the data sheet, Lascaux 303 HV (T_g = -31°C) can be heat-sealed at 50°C, remains permanently tacky when dried and is soluble in acetone, alcohol, toluene and xylene (Lascaux 2012).

Paper and photographs

In the late 1960s, a study of heat-set tissues was conducted for mending paper tears (Hey and Waters 1977). Before this study, many resins used to coat tissues caused blocking (tissues sticking together), and various anti-blocking techniques (e.g. coating with microcrystalline wax or rice starch) were unsatisfactory. In this study, 5 acrylics and 16 PVAC products were examined for anti-blocking behaviour, discolouration, easy reversibility without prolonged use of solvents, hardness, formation of a continuous film at room temperature and plasticizer content. The study found that a mixture of Plextol B500, Rhoplex AC-73 and distilled water (1:1.5:1) on tissue was best and became known as the Library of Congress Heat-set Tissue.

When used to strengthen paper, three styrene/acrylate copolymers (Sokrat 2802, Sokrat 2804, Sokrat 2403), a methyl methacrylate/butyl acrylate copolymer (Sokrat 6492), a vinyl acetate/butyl acrylate copolymer (Duvilax KA-1), and

a PVAC homopolymer with plasticizer were tested for double-folding resistance, tensile strength, whiteness, pH, resistance to hydrolysis, adhesive power of the film, removability and microbiological resistance upon thermal, cyclical/thermal and artificial sunshine aging (Ďurovič et al. 1991). It was concluded that the acrylic copolymer Sokrat 6492 and the PVAC dispersion were suitable for conservation practice.

Paraloid B-72 (in toluene), acrylic dispersions (Rhoplex AC-73, Plextol B500, Rhoplex AC-234, Liquitex Acrylic Gloss Medium), wheat starch paste (WSP), methylcellulose (MC), various PVACs and a rubber cement were aged and tested using various photographic detectors (different types of albumen or silver gelatin step tablets, films or photographic papers) to determine their effect on photographs (effect on silver, common binder layers and paper supports) (Schenck and McCabe 1989). In most of the tests, the acrylics caused deterioration of the photographic detectors and thus gave unfavourable results, while WSP and MC did not damage the photographic substrates and performed best overall. This agrees with PATs done on various adhesive tapes and heat-set tissues studied by Down et al. (2011). In this study, many of the products containing an acrylic adhesive did not pass the PAT, while the product made with wheat starch did.

Leather and skin

Elvacite 2044 and Paraloid F-10, both butyl methacrylate polymers, were examined (Shashoua and Kawanobe 1993). They were found to have similar chemical and mechanical properties, but the Elvacite was slightly less stable to light. It was concluded that the Elvacite 2044 was a good substitute for Paraloid F-10, which had been used for consolidating vegetable-tanned leather and for facing papyrus.

The bonds of various adhesives for gut-skin, including four acrylics (Lascaux 498 HV, Lascaux 360 HV, Paraloid F-10, Paraloid B-72), were examined by scanning electron microscopy (Horelick et al. 2011). The mixture of Lascaux 498 HV and Lascaux 360 HV (3:1) was found to give better contact to gut-skin when the gut was dampened first, while Paraloid F-10 gave better contact when applied from a solution (40% in mineral spirits) than when heat-set. In the same study, Paraloid B-72 did not display intimate contact with the gut-skin when applied from a solution (20%) in acetone.

Textiles

An early study of Paraloid B-72 for textiles showed that it had good colour stability, reversibility and retained flexibility upon aging (Blackshaw and Ward 1983). Another acrylic, Elvacite 2013, although remaining reversible after aging, discoloured and became very brittle.

Films of Primal N-580 formed strong bonds on textiles and leather; were resistant to the effects of light exposure; discoloured only slightly upon thermal aging; were reversible in acetone and partially reversible in industrial methylated spirits (IMS); had suitable pH (8.3) for organic material (between 5.5 and 8.5); did not corrode silver, copper and lead; remained tacky when dried and thus unfortunately gathered dust; and had shear and peel strengths that increased with thermal aging (Shashoua 1992). It was concluded that Primal N-580 was a suitable stable material for conservation, but removal of it could damage weak or friable objects.

In an adhesive testing program for textile conservation, four acrylics (Lascaux 360 HV with Lascaux 498 HV; Texicryl 13-002; Lascaux P 550-40 TB; Paraloid F-10) and six PVACs were evaluated for bond strength (peel), ease of removability (hot peel), reversibility (cold versus hot peel), acidity, corrosiveness and staining, toxicity (fume extraction required), heat-sealing temperature, T_g, ease of application, and ease of handling (Pretzel 1997). While a PVAC dispersion performed best overall, the acrylic solvent adhesives behaved poorly because they were weak, had difficulties with reversal, needed fume extraction, had high heat-seal temperatures, and could be difficult to apply and handle. The acrylic dispersion adhesives were slightly better as they gave strong bonds, did not need fume extraction, and had lower heat-seal temperatures; however, they could be somewhat corrosive, acidic and difficult to apply and handle.

Diluting the dispersion mixture Lascaux 360 HV/Lascaux 498 HV (1:1) with water and an organic solvent (water/acetone 50:50 or 60:40) makes the adhesive spread more evenly as the solvent reduces the high surface tension of water (Rogerson 2002). The resulting film on silk crepeline or Stablitex gives stronger bonds when reactivated with acetone or IMS than if only water is used. Also, less adhesive is needed.

The peel strength of silk fabric specimens adhered with the mixture Lascaux 360 HV/Lascaux 498 HV (1:2) by using solvent reactivation (acetone or IMS) or heat reactivation (temperatures from 75 to 155°C) was investigated for various adhesive concentrations, pressures and reactivation times (Karsten and Down 2005). Acetone reactivation gave

stronger bonds than IMS or heat reactivation for the same time and pressure. A range of peel strengths versus adhesive concentration, time, pressure and temperature is given, which could be useful for the textile conservator.

Acrylic dispersions—Acrilem RP6005, which is a poly(ethyl acrylate), Acrilem 674, which is an ethyl acrylate/methyl methacrylate, and Acrilem ST 1997, which is a styrene/acrylonitrile/butyl acrylate—were investigated (Cocca et al. 2006). The Acrilem RP6005 had a T_g close to room temperature, a stress-strain curve typical of ductile uncrosslinked elastomeric materials, and acceptable yellowing after aging, which indicated possible suitability for textile conservation.

In another study on supports for silk artifacts, A-45K, a copolymer synthesized from vinyl acetate, butyl acrylate and acrylic acid (50:45:5), was investigated after natural aging (20 years) and accelerated light aging (Ragauskienė et al. 2006). After 20 years of natural aging, the adhesive film had lost its integrity, and dust and residues were present; however, it was still flexible. The changes in the adhesive chemical structure indicated that the acetate group in the copolymer aged faster than the acrylate group.

In another recent study, a new technique to consolidate naturally oxidized cellulose-based textiles was investigated that grafted ethyl acrylate/methyl methacrylate (EA/MMA) (75:25) and EA/MMA/2,2,2-trifluorethyl methacrylate (EA/MMA/TFEMA)(73:24.5:2.5) onto oxidized and untreated cotton and linen (Princi et al. 2007). This improved mechanical resistance and flexibility, thus consolidating the textiles. Water repellence, especially with the fluorinated terpolymer (EA/MMA/TFEMA), was also achieved. These polymer combinations were chosen because they resisted biological attack and had the appropriate T_g (10°C) (Princi et al. 2005).

Wax

In a study that examined four acrylic adhesives (Paraloid B-72, Plexisol P550, Solucryl 356 and Lascaux 360 HV), BEVA 371 and a poly(vinyl butyral) (Mowital B 60 HH) to adhere waxed paper, Plexisol P550 was found to be the adhesive of choice because it displayed relatively high shear and peel strength and the standard deviation range was small, indicating good reliability of the results (Hincelin and Roche 2011).

In another wax study when several adhesives were tested for adhering different types of waxes, Lascaux 498 HV was found to give varying results, depending on the type of wax, and it dried relatively slowly, which necessitated support during set, which could be problematic for soft waxes (Lang 2011). Another problem was that the Lascaux is not reversible in water but is reversible in solvents that might damage waxes. In this same study, Paraloid B-72 (in acetone) did not exhibit good application and drying behaviour, caused fractures on the wax surface and gave deviating strength values, so it was not recommended. The Lascaux was recommended if greater strength than the suggested isinglass was needed for certain waxes (but not soft wax; in this instance, Mowilith 30 was suggested if greater strength was needed).

Paintings

In an experiment examining sizing (Paraloid B-72 in xylene) on original canvas and/or polyester lining, and drying time (1 h or 24 h) of the lining adhesive (Lascaux 360 HV), it was found that sizing affects bond strength more than drying time (Allard and Katz 1987). The strongest bonds were obtained when both original canvas and lining were sized, while the weakest bonds occurred when the canvas was unsized.

When various acrylic adhesives (Plextol B500, Rhoplex AC-33, Rhoplex AC-234, Lascaux 360 HV and Lascaux 498 HV) were studied for peel strength, colour change and solubility after exposure to light, Plextol B500 and Lascaux 498 HV were found to be the most suitable for lining paintings where high strength is required (Duffy 1989). The Rhoplex products were suggested for lighter bond applications (paper, fine fabrics), while Lascaux 360 HV gave very high (2–3 times higher) peel strengths, which could limit its application. Toluene was found to be the best solvent to swell the films. The adhesives discoloured and increased in peel strength upon aging.

When various adhesives were investigated for strength for thread-by-thread tear repair for canvas, the acrylic adhesives did not display adequate strength for this application (Demuth et al. 2011). The PVAC and sturgeon glue/WSP mixtures proved to give the higher, more acceptable strengths in this study for this application.

Decorative art

The mechanical fracture behaviour of gesso-type foundations in multi-layered decorative coatings and their consolidation was investigated (Schellmann and Taylor 2011). The acrylic Lascaux Medium for Consolidation gave high increases in fracture energy, appeared to strengthen the specimens and was a

good gap-filler. However, being so strong, it might encourage fracture in new areas—as opposed to only in areas of old fractures, which is more desirable. In the same study, Paraloid B-72 samples did not strengthen the specimens but tended to form starved joins.

Objects – flaking paint

Nineteen acrylic resins along with PVAC, cellulose ether, starch and protein adhesives were evaluated for adhering flaking paint to ethnographic objects (Horton-James et al. 1991). Three acrylic resins (Plextol B500, Rhoplex AC-33, Neocryl A1044) gave good results under the testing regime employed, although the pH was quite low in two instances. Plextol B500 was selected for the application.

Wood

In a study on strengthening deteriorated wood with various resins, it was shown that the strength of wood treated with Paraloid B-72 can be increased by 16% (Schniewind and Kronkright 1984).

Plaster

Several acrylics (Acrysol WS-24, Experimental Emulsion E-2014, Neocryl BT-520, Neocryl BT-175) that can be re-solubilized in water by the addition of ammonia (in other words alkali-soluble) were tested as consolidants for plaster (Phillips 1987). These can afford a non-flammable possibility with low toxicity for strengthening plaster with a water-based product that, when dried, is not as affected by RH changes as water-soluble resins might be. In architectural situations, plaster surfaces are typically very large, so solvent-based resins are undesirable due to high fire and toxicity hazards. In the testing program, Neocryl BT-520 and Acrysol WS-24 were found to be more flexible than the others, while Acrysol WS-24 had the best water resistance. They all consolidated plaster to some extent, with Neocryl BT-520 being the most effective. Yellow stains did appear on the plaster; however, Neocryl BT-520 had much better resistance to yellowing and loss of solubility than Acrysol WS-24.

In a continuation of this study, aqueous acrylic/epoxy resin consolidants were proposed for the consolidation of plaster (Phillips 1995). These consolidants are even more moisture-resistant when set than the alkali-soluble acrylics mentioned above. The acrylic/epoxy resin consolidants also employ alkali-soluble acrylics to help solubilize the epoxy in a water system. Once applied to plaster, the consolidant crosslinks with time. The treated plaster was assessed by air abrasion and field tests and found to be considerably strengthened and durable, but this technique was not recommended for use because definitive testing had not been completed and has still not been completed to date, as far as can be determined.

In another study, two acrylic dispersions (Primal AC-33, Vinacryl 40224) were evaluated for securing loose paint flakes to plaster and consolidating friable surfaces (Shashoua 1993). Both showed good colour stability, fairly low shrinkage upon aging (~4%) and passed the Oddy test. As Primal had a higher pH and greater solubility in acetone than the Vinacryl, it was suggested as the more suitable product for the intended use.

Poly(methyl methacrylate)

Several acrylics and epoxy resins were tested for repairing PMMA objects (Sale 1993). In this evaluation, most of the acrylics caused crazing of the PMMA under load except for the UV-cure types (Norland OA65, Norland OA68). The solvent types [Paraloid B-67 with Paraloid F-10 (1:1); Paraloid B-72] gave weak bonds, while the UV-cure ones gave bonds about half the strength of epoxy resins. The two-part acrylics (Tensol 70, Acrifix 90) gave strong results. Hxtal NYL-1, Norland OA65 and the Paraloid B-67/Paraloid F-10 mixture gave the best results. These adhesives did not craze the stressed PMMA. They formed a range of strengths, had refractive indices closer to PMMA and had lower T_g than PMMA. After 20 years of natural aging, the test samples were re-evaluated for yellowing and the Paraloid B-67/Paraloid F-10 mixture and Hxtal NYL-1 experienced minimal yellowing of samples open to the air, while the Norland samples appeared to have yellowed more than these (Sale 2011).

Ceramics

Paraloid B-72, Paraloid B-67 and Elvacite 2044 were examined for consolidating friable ceramics (Fox 1979). Paraloid B-67 had good resistance to abrasion, while Paraloid B-72 had the least, but was good visually.

Nine different adhesives (Paraloid B-72, Scotch acrylic resin, PVACs, cellulose nitrates, cyanoacrylates, epoxy resins) were appraised for tensile strength, yellowness index (before and after cyclic heat aging), thickness of bond, working time and overall hazard for Spanish medieval ceramics (Castro and Doménech Carbó 1999). Of all, Paraloid B-72 (50% in acetone) was found to give the best results to these tests.

Glass

Paraloid B-72 has been recommended as an adhesive for ancient glass, which is more fragile than modern glass (Koob 2000). Paraloid B-72 was considered

more appropriate than epoxy resin adhesives because it has moderate strength, sufficient flexibility to tolerate stress, stability, reversibility, adjustability by low heat or solvents, and has a better matching refractive index to ancient glass than epoxy resins adhesives. The slight visibility of bubbles within the dried Paraloid B-72 was considered of little consequence for ancient glass as it is not perfectly clear and bubble-free itself. Paraloid B-72 has also been suggested for making fills for glass, which were very successful (Koob et al. 2011).

Stone

Tabasso (1995) has published a good review of acrylic polymers for stone consolidation (techniques and products). In this review, she notes that PMMA and poly(butyl methacrylate) (PBMA) were used in the 1960s as a consolidant and as a protective coating. Later, Paraloid B-72 came into use for the same applications. Paraloid B-72, however, had poor penetration and was less water repellent than the silicone polymers. Therefore, Paraloid B-72 was incorporated into methyltrimethoxysilane, which showed better water repellency, but "retained much more dust than other protective products not containing acrylics" (p. 19). Following this development, a PMMA consolidation technique was found to yield better mechanical resistance and hydrophobicity than untreated stone, but the absorption of small amounts of water caused stresses in the stone by expansion and swelling. Tabasso also mentioned that acrylic *monomers*, which penetrate more easily than *polymers* into the stone and are polymerized in-situ or are used in precipitation polymerization, have been tried, but these techniques can cause the stone to fracture due to stresses developing during polymerization. Nevertheless, some MMA monomer techniques have been tested and were shown to give satisfactory results.

In an earlier study, 25 consolidants, including acrylics, silicones, silanes, epoxy resins, fluorides and a polyurethane, were tested for their consolidating abilities on sandstone and limestone (tensile and compressive strength) (Clifton 1984). Only some of the consolidants passed this test and were further tested for depth of penetration, appearance, depth of erosion and colour change after acid drip, freeze-thaw stability, and thermal expansion tests. Some of the acrylics (PMMA, acrylic ester) consolidated the stone, while others [poly(butyl acrylate), poly(methyl acrylate)] did not. Most of the acrylics gave a good appearance and had a coefficient of thermal expansion close to stone, which was also considered a good property. An epoxy resin, however, gave the best results.

Evaluation (depth of penetration, curing in high RH conditions, microabrasion resistance, water abrasion resistance and change in porosity) of various consolidants for stabilizing weak Maya limestone under various types of in-situ aging and simulated laboratory aging for hot humid tropical climates showed that an acrylic-epoxy mixture improved the water and particle erosion resistance, while acrylic dispersions and solutions did not protect from erosion (Kumar and Ginell 1995).

The behaviour of several consolidants and water repellents on fractured stone specimens filled with resins was examined (Santamaría and Calatayud 1997). The treatments improved the resistance to aging, with Sinocril, an acrylic resin, combined with a water repellent [a modified siloxane (Tegosivin HL100)] giving the best results.

In a study examining Paraloid B-72, Paraloid B-67 and silicone-based Dri-Film 104 for their performance upon exposure to light, Dri-Film crosslinked and the acrylic resins showed chain scission and/or reticulated structures, which gave a reduction of solubility and removability and colour changes (Favaro et al. 2006). When a mixture of Paraloid B-72 and Dri-Film 104 was applied in a thin film, the photodegradation was enhanced, but was limited in a thicker film by poor oxygen penetration. A deterioration process was proposed that was also identified on outdoor stone surfaces that had been treated with this mixture 20–30 years previous, so the state of deterioration could be monitored for future maintenance (Favaro et al. 2007).

A photodegradation study of various acrylic resins for stone conservation showed that chain scission was the principal degradation mechanism and continued to the point where all coatings were eliminated (Melo et al. 1999). The copolymers [Paraloid B-72 and poly(ethyl methacrylate/methyl acrylate) (PEMA/MA)] showed higher photostability than PEMA alone due to the acrylic co-monomer MA. Exposure to Xenon-arc irradiation and to sunlight outdoors resulted in similar chemical processes, but physical damage suffered by outdoor samples was different (Bracci and Melo 2003).

An acrylic barrier coating (Paraloid B-72) increased the reversibility of an epoxy resin and a polyester resin used for adhering stone (Podany et al. 2001). To afford reversibility, the barrier coating must be thin, conformal, continuous and fully dried before the epoxy or polyester is introduced. Use of the barrier coating in this way gave a bond that was sufficiently strong for structural joints,

but long-term creep was not examined and could possibly affect the bond.

The surface of stone monuments is sometimes protected with polymers and pigments and hence the photodegradation of Paraloid B-72 films was studied with the addition of titanium dioxide pigments (anatase, anatase/rutile mixture) at different concentrations (Spathis et al. 2003). The anatase (30%) alone afforded better protection than the mixture.

New techniques in nano-technology fluorescing quantum dots are being used successfully with a solution of Paraloid B-72 to show the depth of penetration and concentration effects in the consolidation of a weak lime/sand matrix (Zucker et al. 2011).

Researchers tested the tensile strength of Paraloid B-72 on limestone samples in order to see if it would be suitable as a structural adhesive for fossil bone in palaeontological collections (Russell and Strilisky 2011). Other adhesives (Paleobond PB100, a cyanoacrylate, and Devcon-2-ton, an epoxy) were stronger; although, the epoxy was stronger than the limestone, which was deemed undesirable. However, it was suggested that the Paraloid B-72 would be suitable if the tensile force exerted was <2.215 MPa. The Paraloid B-72 was more reversible, stable and gave the lowest surface damage compared to the other two.

Metals

In a study examining protective coatings for silver exposed to hydrogen sulfide, acrylic Acrysol WS-24 performed very well (low tarnishing), while Paraloid B-72 and Paraloid B-48N were less effective (Grabow et al. 2007).

Modified Oddy testing of Acronal 300D and Acronal 500D (*n*-butyl acrylate/vinyl acetate/vinyl chloride copolymer) coatings showed reactions on lead and copper with silver being unchanged (Farnsworth 1996). With these results and the pH being very low, these products were ruled as unsuitable for storage and display of artifacts.

The addition of increasing amounts of WSP or carboxymethylcellulose (CMC) to Rhoplex N-619 decreased the shear strength on aluminum substrates, and increased the equilibrium moisture content with an increased tendency to swell under higher humidity (Fulton 1988). The dried samples of Rhoplex N-619 with added WSP or CMC were not soluble in water; however, the additions did increase swelling.

SOLUBILITY AND/OR REMOVABILITY*

Acrylic resins are soluble in aromatic and most chlorinated hydrocarbons (e.g. toluene, chloroform), esters (e.g. ethyl acetate), ketones and tetrahydrofuran (Roff and Scott 1971, p. 90). Butyl polymers, as well as higher esters, are soluble in aliphatic hydrocarbons (e.g. white spirits) (Roff and Scott 1971, p. 90). Crosslinked polymers are insoluble but swell in chlorinated hydrocarbons (Roff and Scott 1971, p. 90). Some acrylics are swollen by alcohols (Roff and Scott 1971, p. 90).

Photochemically (daylight fluorescent lamp) deteriorated films of Paraloid B-72, Paraloid B-67, Elvacite 2045 and Elvacite 2046 were easily removed with a mixture of toluene and cyclohexane (40:60), which is considered a weak solvent (Feller 1976). When exposed to more intense light (Xenon arc lamp), the films became somewhat less soluble and needed 100% toluene for removal. According to Feller et al. (1981), Paraloid B-72 "should remain colourless and soluble in the solvents in which they were originally soluble, for more than 200 years" if exposed to daylight fluorescent lights and natural illumination on a gallery wall (p. 196).

Paraloid B-72 is a good binding medium for pigments, but can be viscous in aromatic solvents and becomes sticky and stringy quickly. It is not soluble in white spirit, aliphatic hydrocarbons, water or methanol (Phenix 1992b). Solvents for Paraloid B-72 should not evaporate quickly (BP 110–170°C) and should have similar solubility parameters to the cohesion parameter of Paraloid B-72 (Phenix 1992b). Alternative solvents, such as Methyl PROXITOL, Methyl PROXITOL Acetate, ethoxy propanol and ethoxy propanol acetate, were suggested because they met these requirements and fell in the same occupational exposure limit range as toluene (Phenix 1993). The acetates have a stronger smell than corresponding ether alcohols, so the latter may be the more pleasant choice. These alternative solvents could replace the aromatic solvent completely or be blended with it to reduce the aromatic content. They were suggested for retouching media, but were not recommended for varnishes or paint consolidation.

* Disclaimer: The solvents listed in this section are ones that might dissolve the polymer or adhesive in question but are not necessarily recommended for conservation treatments. Some may be extremely toxic or may contain elements (e.g. chlorine) that may be harmful to certain materials in your object (artifact). Research the solvent before purchase or use to know its toxicity and chemical nature. It is strongly recommended to test all solvents before use to make sure that they do not adversely affect your object.

Consolidants are difficult to remove. Even with extreme extraction techniques (Soxhlet extraction or agitated soaking), residues of Paraloid B-72 were left in wood (Schniewind 1988). In another Soxhlet bone consolidation removal experiment, Acrysol WS-24 was acceptably removed and should not interfere with further bone analyses; however, Paraloid B-72 was retained after extraction, and could alter bone analysis (Unruh 1994).

Poly(*n*-butyl methacrylate) varnishes were still removable after 10–49 years after exposure to UV-free light, even though higher polarity solvents were required to remove them (Quillen et al. 1990).

Cyanoacrylates

Setting mechanism	Category	Water absorption (%)	Refractive index
chemical reaction	thermoplastic		methyl cyanoacrylate 1.4406 ethyl cyanoacrylate 1.4349 isopropyl cyanoacrylate 1.4291 butyl cyanoacrylate 1.4330 isobutyl cyanoacrylate 1.4352
Glass transition temperature (°C) (anionic polymerization; molecular weight varies)	**Viscosity (mPa.s)**	**pH**	**Coefficient of linear expansion /°C**
methyl-cyanoacrylate 100 ethyl cyanoacrylate 115 isopropyl cyanoacrylate 66 *n*-butyl cyanoacrylate 56 isobutyl cyanoacrylate 51 *n*-hexyl cyanoacrylate 86 *n*-hept cyanoacrylate 88	2–5 depends on type of cyanoacrylate and additives		~80–100 × 10^{-6}

(Chemtools; Permabond Engineering Adhesives 2011; Coover et al. 1990; Kulkarni et al. 1973)

Abbreviation

Sometimes CA is used but this can be confused with cellulose acetate. For this book, no abbreviation for cyanoacrylate has been adopted.

Types, synonyms and/or trade names

alkyl-2-cyanoacrylate, Eastman, Glustitch, Hot Stuff, Krazy Glue, Loctite, PaleoBond, Permabond, Super Glue, Zap

Chemistry and/or preparation

Cyanoacrylates polymerize (cure) by an anionic and/or free-radical mechanism, although anionic polymerization (**Figure 1**) is predominant as it can be initiated more easily by weak bases (Coover et al. 1990). Even a thin film of water or alcohol is sufficiently basic to initiate the cure process. On the other hand, free-radical polymerization for this monomer requires prolonged periods of high temperatures, ultraviolet light or heat with peroxides for initiation (Coover et al. 1990). The nitrile (-CN) and alkoxycarbonyl (-COOR) groups are electrophilic (electron withdrawing), which makes the double bond in the monomer very reactive. Most materials have a thin film of moisture on the surface and it is the hydroxyl groups in this water that initiates rapid polymerization. This polymerization is initiated at the adhesive-substrate interface and proceeds from there through the adhesive. If the adhesive is thick, the chance of polymerization termination increases, thus, thin bonds are preferable and gap-filling is unlikely if the gap is large.

Cyanoacrylate formulations may contain stabilizers (e.g. acids, hydroquinone) to improve shelf life. Acids are added to neutralize weak bases that could attack the highly susceptible double bond and cause anionic polymerization (Coover et al. 1990; Kotzev et al. 1987; Millet and Withee 1982; Millet 1981). Hydroquinone is added to inhibit free-radical polymerization (Coover et al. 1990). For a fast cure, the use of fresh adhesive is best because added acid stabilizers can gradually hydrolyze the monomer and make it ineffective.

Proper surface preparation of substrates is recommended for good cyanoacrylate bonding. Clean surfaces are essential. Degrease surfaces before application, if possible. Surface roughening, particularly for metals, improves bond strength, but is not always possible or ethical for artifacts. Surface activators are sometimes needed to improve bonding, especially on acidic or porous substrates

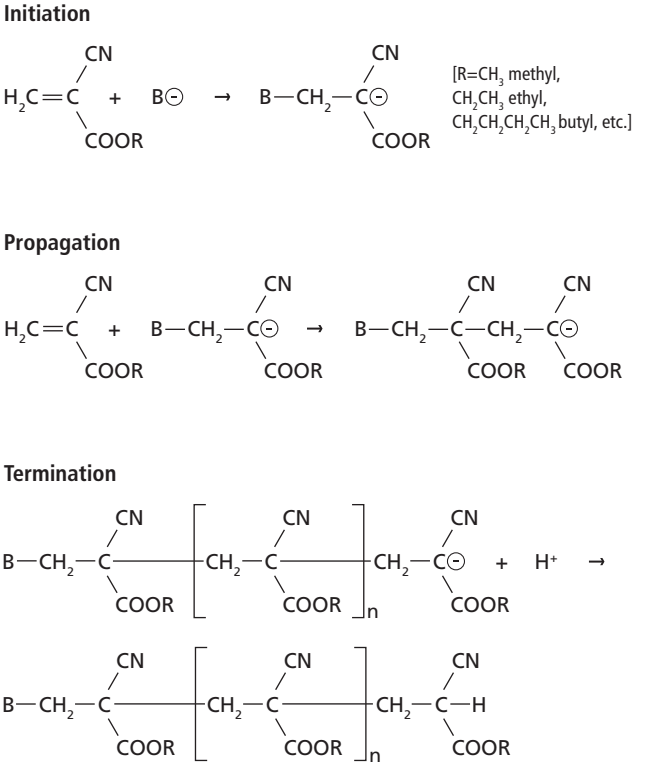


Figure 1. Anionic polymerization mechanism (Coover et al. 1990).

(e.g. paper, wood), or on polyethylene, polypropylene or plasticized poly(vinyl chloride) (Millet 1981). For acidic surfaces, the surface activator often used is a mild alkaline wash (Coover et al. 1990). Other activators are amines dissolved in solvents (Millet 1981) or crown ethers, silacrowns, calixarenes or polyalkylene ethers (Coover et al. 1990).

Cyanoacrylates can be esters of methyl, ethyl, butyl, octyl, etc. The majority of commercially available cyanoacrylates in North America are ethyl cyanoacrylates. Butyl and higher ester-substituted cyanoacrylates are more difficult to obtain, but are available from medical suppliers.

History and uses

The earliest patents for cyanoacrylates were issued in 1949 to the B.F. Goodrich Company (Ardis 1949). The adhesive properties of cyanoacrylates were first discovered inadvertently at the Kodak laboratories during the Second World War (Coover 1983). Ten years later, a cyanoacrylate adhesive became commercially available as Eastman 910 (Schoenberg 1990). Cyanoacrylates were suggested as a wound closure adhesive and were used in the Vietnam War to stop bleeding (Coover 1983).

Cyanoacrylates bond well to non-porous materials that contain traces of surface water. They have been used as structural industrial adhesives for metals, alloys, plastics, rubbers, electronics and model aircraft. They are also used to develop latent fingerprints on surfaces for forensic science and have been used since the early 1960s as surgical adhesives for tissue closure and bone fractures.

In conservation, cyanoacrylates have found limited use (Fiorentino and Borrelli 1975) for stone (Jahont 1979), metal (Herczeg 1992), glass (Robson 1993; Davison 1984) and fossils (Elder et al. 1998; Elder et al. 1997; Vestergaard and Horie 1996; Howie 1984). Conservators and fossil preparators have used them as early as the 1970s (Howie 1984). Their use for geological materials began in the 1980s.

Characteristics, limitations and/or aging studies

General

An excellent overview of cyanoacrylate polymers is found in Coover et al. (1990). The advantages of cyanoacrylates are that they are easy to use (no mixing of two parts), rapidly cured, strong, solvent-resistant (but not to acetone, nitromethane, dimethyl sulfoxide or dimethyl formamide) and versatile (bond well to a variety of substrates) (Coover et al. 1990; Millet 1981).

Their limitations have also been well-documented (Coover et al. 1990; Drain et al. 1984; Al-Khawam et al. 1983; Millet 1981). They have a strong odour, although alkoxy-alkyl esters have been added to produce odourless products (Millet 1981). They tend to be brittle, especially the lower alkyl esters (methyl, ethyl), while the higher esters (butyl) are less brittle (Coover et al. 1990). They exhibit poor peel strength; poor impact resistance; poor moisture resistance (especially on metal surfaces; better on plastics and rubbers) (Drain et al. 1984); poor heat resistance, although allyl esters and acrylates have been added to improve this (Samatha et al. 2000; Millet 1981); and poor gap-filling properties, although thickeners such as polymers, plasticizers and hydrophobic silicas have been added to improve gap-filling properties (Millet 1981). They (Loctite 406 and Loctite 460) have been shown to craze Perspex or Plexiglas when stressed under load (Sale 2011, 1993).

Successful cyanoacrylate bonds depend on substrate composition, the alkalinity of the surface of the substrate (acidic surfaces do not work as well),

surface porosity (best if not wicked away by a porous substrate), surface preparation (clean surfaces are best), stress applied, humidity and temperature (bonds are susceptible to high humidity and temperature), adhesive formulation, and age of the cyanoacrylate (Coover et al. 1990). Compared to acrylics, epoxies and urethanes, cyanoacrylate bonds exhibit low durability and low resistance to high and low temperature (Bishari et al. 2003; Brewis 1985). Cyanoacrylate bonds deteriorate with time when joining alkaline surfaces such as glass (Schoenberg 1990). On the other hand, cyanoacrylate-glued pieces of old marble and alabaster sculptures held together without visible changes ten years after repair (Jahont 1979). Hydrolysis of cured cyanoacrylate leads to the formation of formaldehyde and to gradual strength loss (Schoenberg 1990).

MEDICAL RESEARCH AND THE DEGRADATION OF CYANOACRYLATES

The medical profession is interested in cyanoacrylates primarily for tissue closure and repairing bone fractures. The bulk of scientific cyanoacrylate research has been conducted by the medical profession and some of it may be relevant for conservation.

Results from medical research for tissue closure suggest that cyanoacrylates degrade by hydrolytic scission, which produces formaldehyde and alkyl cyanoacetate as by-products, both toxic compounds (**Figure 2**) (Leonard et al. 1966). Alkaline conditions increase the degradation rate and the lower esters (e.g. methyl, ethyl) degrade faster than the higher esters (e.g. butyl, octyl) (Leonard et al. 1966).

Medical researchers concerned with tissue closure (Toriumi et al. 1990; Al-Khawam et al. 1983) are concentrating on isobutyl and *n*-butyl cyanoacrylates, which produce the toxic by-products sufficiently slowly so that they are easily absorbed and tolerated by the body. By contrast, methyl or ethyl cyanoacrylates produce the toxic by-products faster than can be absorbed by the body, leading to dermatitis, carcinomas and other problems.

In regard to the use of cyanoacrylates for repairing bone fractures, isobutyl esters have been shown to have the greatest tensile strength in the cyanoacrylate/bone bond, and the presence of hydroxyapatite (10%) improves the bond strength (Papatheofanis 1989). In another study, isobutyl and isoamyl cyanoacrylates were found to be stronger than ethyl cyanoacrylate after temperature cycling (5–55°C) in a water bath for 30 days (Bauer et al. 1979). Investigation of space-filling properties of cyanoacrylates for bone showed that water (immersion) reduced bond strength (15–30%) after storage (3–26 weeks); strength of thin bonds was 6–11 MPa, while gap-filling bonds were much weaker (0.3–0.4 MPa); pre-treatments with acids to increase bone porosity increased bond strength; and isobutyl esters were more effective for space-filling (Bhat et al. 1992). This medical research was conducted in saline solutions to replicate wound conditions so results may not translate well to conservation. Perhaps degradation rates at 50% relative humidity (RH) (or higher RH levels) would be less than degradation in saline solutions, making these cyanoacrylates more acceptable.

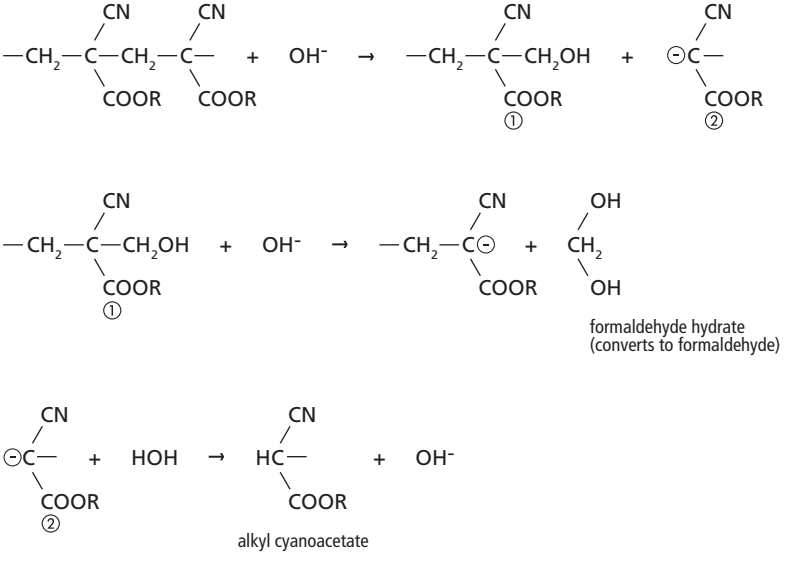


Figure 2. Cyanoacrylate degradation by hydrolytic scission (Toriumi et al. 1990).

CYANOACRYLATES AND FOSSIL PREPARATION

Cyanoacrylates are favoured for fossil preparation because they are easy to use, strong, set rapidly and remain clear, while their disadvantages are that they are difficult to remove, can stain iron-rich matrices, and have uncertain aging properties (Howie 1984). One study on adhesives for fossils, which included an ethyl cyanoacrylate, used scanning electron microscopy, infrared spectroscopy and tensile strength testing to evaluate five different adhesives for repairing a mastodon tooth (Vestergaard and Horie 1996). Cyanoacrylates filled the gap between the adherends but suffered internal shrinkage cracks and some loss of adhesion, causing spalling of the tooth. This study revealed that, while cyanoacrylates produced strong joins, epoxy resins were the strongest, and a poly(vinyl acetate) (PVAC) was the most suitable overall adhesive for the tooth.

Reasons why cyanoacrylates are being used for fossils (e.g. easy to use, strong, quick setting) were reviewed by Elder et al. (1998) and recommendations on their effective use for fossils were made. The recommendations included:

- leave the cyanoacrylate to cure slowly to get deeper penetration;
- do not use accelerators;
- use accelerators and activators sparingly (e.g. to stop penetration at a particular point—never use as replacement for patience);
- do not use in damp areas;
- once opened, do not expose to wide temperature fluctuations or light that could cause curing in the container;
- unopened products can be stored frozen to prolong shelf life, but must be warmed to room temperature before use;
- evaluate every situation carefully to decide if an adhesive is really necessary since no consolidant is really removable (conserve without applying chemicals to preserve future specimen data); and
- consolidate first with an ethyl cyanoacrylate and then apply the somewhat thicker cyanoacrylates. Ethyl cyanoacrylates make more suitable consolidants than adhesives because they penetrate and have high tensile strength. The consequences of their low impact and peel strengths are minimized when used as consolidants. The more viscous ethyl cyanoacrylate/poly(methyl methacrylate) mixtures make better adhesives than consolidants because they have better consistency, and impact and peel strengths.

Cyanoacrylates are being used routinely for fossil preparation even though few scientific conservation studies have been done because many users feel that their benefits outweigh their unknowns (Elder et al. 1998). One recent conservation study followed up on the above medical research, comparing the degradation rates of different alkyl cyanoacrylates (ethyl, butyl) under differing pH conditions, but in a fossil environment (Down and Kaminska 2006). It examined the degradation of three types of cyanoacrylate adhesives—an ethyl cyanoacrylate (Loctite 420), an ethyl cyanoacrylate with added poly(methyl methacrylate) (Paleobond PB1500), and a butyl cyanoacrylate (Glustitch)—in the presence and absence of five different fossils obtained from various sites that differed in composition and pH. Hydrolytic degradation of the polymerized cyanoacrylate was monitored by quantitative determination of formaldehyde, one of the degradation products. Both in the presence and absence of a fossil, butyl cyanoacrylate was found to degrade slower than ethyl cyanoacrylate (**Table 1**). These results suggest that butyl cyanoacrylate be investigated (for cost, availability, handling properties, ease of use, penetration, strength retention and removability) for possible fossil applications. It was also shown that acidic fossil inhibits cyanoacrylate degradation, while neutral or alkaline fossils increase cyanoacrylate degradation. So, if the fossil is acidic, then the use of ethyl cyanoacrylate might not be as problematic since it degrades less than if neutral or alkaline fossil is present or no fossil is present, but butyl cyanoacrylate still degraded the least. Further study is necessary to determine how the observed cyanoacrylate degradation affects the actual fossil/cyanoacrylate bond strength, and how cyanoacrylate degradation is affected by high humidity as opposed to the immersion technique used to degrade the cyanoacrylate.

CYANOACRYLATES IN OTHER AREAS OF CONSERVATION

Ceramics

Nine different adhesives, including cyanoacrylates (Scotch, Super Glue), PVACs, a cellulose nitrate, acrylics and an epoxy, were appraised for tensile strength, yellowness index (before and after cycles of thermal aging), thickness of bond, working time, and overall hazard for Spanish medieval ceramics (Castro and Doménech Carbó 1999). Paraloid B-72 (50% in acetone) gave the best results. The Scotch cyanoacrylate showed good resistance to aging, but its other properties were rather unsuitable.

Glass

Because cyanoacrylates break down in the alkaline conditions of most glass surfaces, they are not recommended for permanent glass repair, but are used temporarily to hold glass fragments while permanent slow-curing epoxy resin adhesives are

TABLE 1. FORMALDEHYDE PRODUCED BY CYANOACRYLATE IN PRESENCE AND ABSENCE OF FOSSIL DURING HYDROLYTIC DEGRADATION (7 DAYS IMMERSION IN WATER AT 50°C)
(Down and Kaminska 2006)

Fossil present	pH of fossil	Formaldehyde (μg/g adhesive)			Comments on degradation of cyanoacrylate versus pH ↓
		Poly(ethyl cyanoacrylate)	Poly(ethyl cyanoacrylate) + poly(methyl methacrylate) (PMMA)	Poly(butyl cyanoacrylate)	
NO	5.94 (water)	160	315	7.3	← **intermediate** with *no* fossil
YES	4.53	33.7	113.9	6.6	← **lowest** in presence of acidic fossil
YES	7.43	1093	878	466.3	← **highest** in presence of neutral or alkaline fossil
YES	7.76	1273	1251	912.1	
YES	8.44	2123	1504	996.5	
YES	8.48	1223	766	765.6	
comments on degradation versus type of cyanoacrylate →		↑ poly(ethyl cyanoacrylate) shows **intermediate** degradation compared to other cyanoacrylates when no fossil is present or when fossil is acidic, but **highest** degradation when neutral or alkaline fossil is present	↑ poly(ethyl cyanoacrylate) + PMMA shows **highest** degradation compared to other cyanoacrylates when no fossil is present or when fossil is acidic, but **intermediate** degradation when neutral or alkaline fossil is present	↑ poly(butyl cyanoacrylate) shows **lowest** degradation compared to other cyanoacrylates	

introduced in the join. Studies have shown that some cyanoacrylates can alter the appearance (e.g. crizzling, opacity, colour) of some epoxy films, limiting their value as a temporary adhesive if large quantities are used (Monahan 1994; Miller 1996).

Metals

Cyanoacrylates have been known to react with brass (Horie 1987, p. 110).

HEALTH ISSUES

Human skin is an ideal substrate for curing cyanoacrylates because of the moisture and protein present and, therefore, care should be taken not to glue fingers together when handling these products (Millet and Withee 1982).

When cyanoacrylate comes in contact with skin together with fabric such as cotton or wool, it can cause a strong exothermic reaction, resulting in severe burns to the skin (Clarke 2011). Contact with cotton or wool alone may cause fire (Accumetric 2000). Do not wear cotton gloves when using cyanoacrylates.

SOLUBILITY AND/OR REMOVABILITY*

Cyanoacrylates show poor removability from geological and palaeontological specimens (Elder et al. 1997). However, they are somewhat affected by nitromethane, acetone and hot water or steam, and dissolve in dimethylformamide (Horie 2010, p. 158; Coover et al. 1990; Roff and Scott 1971, p. 97).

The removability of cyanoacrylates is dependent on getting the solvent to the adhesive. For palaeontological specimens, apply the solvent to the surface of the adhesive and wait until it becomes gummy (Elder et al. 1998). Remove the softened layer, then reapply the solvent and repeat until the adhesive has been removed.

* Disclaimer: The solvents listed in this section are ones that might dissolve the polymer or adhesive in question but are not necessarily recommended for conservation treatments. Some may be extremely toxic or may contain elements (e.g. chlorine) that may be harmful to certain materials in your object (artifact). Research the solvent before purchase or use to know its toxicity and chemical nature. It is strongly recommended to test all solvents before use to make sure that they do not adversely affect your object.

Poly(2-ethyl-2-oxazoline)

Setting mechanism	Category	Water absorption (%)	Refractive index
evaporation	thermoplastic		1.520
Glass transition temperature (°C)	**Viscosity (mPa.s)**	**pH**	**Coefficient of linear expansion /°C**
69–71[†] 55[††]		6.4	

([†]Polymer Chemistry Innovations 2010; [††]Wolbers et al. 1998)

Abbreviation

PEOX poly(2-ethyl-2-oxazoline)

Types, synonyms and/or trade names

Aquazol, poly 2-ethyloxazoline, polyethyloxazoline, poly(*n*-propionylethyleneimine)

Chemistry and/or preparation

Poly(2-ethyl-2-oxazoline) (PEOX) (**Figure 1**) is made by cationic ring-opening polymerization of the monomer 2-ethyl-2-oxazoline (Wolbers et al. 1998; Kroschwitz et al. 1985, pp. 680–739). It is available in different molecular weights (e.g. 50,000, 200,000, 500,000) (Kroschwitz et al. 1985, pp. 680–739).

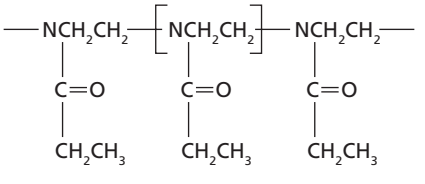


Figure 1. Primary structure of PEOX (Wolbers et al. 1998).

History and uses

The Dow Chemical Company developed PEOX and took out patents in 1977 (Wolbers et al. 1998).

PEOX has been used as a biodegradable adhesive for packaging, binder for greenware, sizing for fibreglass, binder for hot-melt and pressure-sensitive adhesives, and epoxy additive to change mechanical properties (Wolbers 2008; Arslanoglu and Tallent 2003; Wolbers et al. 1998).

In conservation, Aquazol has been used as a consolidant for glass; an adhesive, consolidant and inpainting medium for oil paintings, paintings on glass, painted furniture and watercolours; carvable fill with Paris Whiting; consolidant for deteriorated enamel and unfired clay figures; gilding material; and has been suggested as the adhesive coating for remoistenable tissues for book and paper conservation (Lechuga 2011; Knight and Borgioli 2008; Arslanoglu and Tallent 2003). It has also been suggested as a lasting plasticizer for Paraloid B-72, wheat starch paste, methylcellulose and hide glue (Wolbers 2008).

Characteristics, limitations and/or aging studies

General

PEOX is liked in conservation because it is soluble in a wide range of solvents, thermally stable and non-toxic (Arslanoglu and Tallent 2003). It also allows for re-treatment later on with other adhesives, and does not shrink, become brittle or support mould growth (Arslanoglu 2005).

When Aquazol was tested after exposure to light, it did not discolour, showed no change in the Fourier transform infrared (FTIR) spectrum, pH or re-solubilization tests, and viscosity decreased upon aging, an observation which is consistent with a decline in molecular weight (Wolbers et al. 1998). Size-exclusion chromatography data also showed the same phenomenon, which suggests that PEOX does not crosslink, but degrades by chain scission.

In the same study, the mechanical properties of Aquazol 500 were affected by relative humidity (RH), getting weaker and more extensible at high RH, although still acceptable. PEOX is thermally stable up to 220°C and perhaps even up to 320°C (Wolbers et al. 1998; Keskkula and Paul 1986). Films of Aquazol appear to be plasticized by absorbed water (Wolbers 2008).

PAINTINGS

In a study that compared Aquazol to gelatin and sturgeon glue for use in painting conservation, results showed that adhesion on canvas at elevated RH decreased with increasing RH (Arslanoglu and Tallent 2003). Also in the same study, Aquazol absorbed the most moisture at elevated RH compared to gelatin, sturgeon glue or Jade 403, but at a slower rate. However, when Aquazol films were made with isopropanol/water rather than with water alone, less moisture was absorbed. The study also showed that the Aquazol films shrank less and were more flexible (Aquazol 50>200>500) than the other adhesives. Aquazol was also found to be softer, had longer working time and better penetration. RH must be considered when using Aquazol, and it may complex with metal ions, which could cause slower re-solubilization (Arslanoglu 2005).

Aquazol 500 with or without sturgeon glue was found *not* to be strong enough for thread-by-thread tear repair for canvas (Demuth et al. 2011).

SOLUBILITY AND/OR REMOVABILITY*

PEOX is soluble in a wide range of solvents (e.g. water, acetone, methyl ethyl ketone, methanol, ethanol, isopropanol, chloroform, ethylhexanol, methyl acetate, methylene chloride, dioxane, acetonitrile, nitromethane, propylene carbonate) and slightly soluble in toluene and *n*-pentane (Wolbers et al. 2008).

* Disclaimer: The solvents listed in this section are ones that might dissolve the polymer or adhesive in question but are not necessarily recommended for conservation treatments. Some may be extremely toxic or may contain elements (e.g. chlorine) that may be harmful to certain materials in your object (artifact). Research the solvent before purchase or use to know its toxicity and chemical nature. It is strongly recommended to test all solvents before use to make sure that they do not adversely affect your object.

Epoxy Resins

Setting mechanism	Category	Water absorption (%)	Refractive index
chemical cure [room-temperature (most used in conservation) or elevated-temperature]	thermosetting	~0.1–0.2 in 24 h ~1.0 after 10 days 20–25°C	~1.56
Glass transition temperature (°C)	**Viscosity (mPa.s)**	**pH**	**Coefficient of linear expansion /°C**
typical resins (DGEBA) cured with: polyamides >50 aliphatic amines 100–150 aromatic amines 160–200	varies depending on composition but can range from 100–18,000		$70–120 \times 10^{-6}$

(Roff and Scott 1971, p. 270; Shields 1976, p. 299)

Abbreviation

EP epoxy or epoxide

Types, synonyms and/or trade names

Ablebond, Araldite, Devcon, Dur-a-glaze, Eccobond, Epojet, Epon, Eponex, Epo-Tek, Epoweld, epoxide resins, Epoxybond, Epoxyglass, ethoxyline resins, Fynebond, Homalite, Hxtal, Hysol, LePage, Maraglas, Novalac, Scotch-Weld, Tra-Bond, Tra-Cast, UHU, West System

Chemistry and/or preparation

Epoxy resin adhesives are two-part systems that employ a resin and a hardener to cure. The resin contains epoxide groups, which are three-membered heterocyclic rings. A typical epoxy resin is diglycidyl ether of bisphenol A (DGEBA) (**Figure 1**). The hardener is usually an aliphatic amine or amide compound (**Figure 2**). The resin and hardener mix together in a pre-determined ratio, which depends on the number of epoxide groups in the resin and nitrogen groups in the hardener.

The two components react together exothermically to form a three-dimensional crosslinked network. Frequently, inert fillers, diluents, flexibilizers and flame-retardants are added to adjust the properties or to lower costs (Potter 1970, p. 4). There are room-temperature-cure and elevated-temperature-cure epoxy resin adhesives. Generally, amine and amide hardeners are used in the room-temperature-cure varieties, while anhydrides are employed for the elevated-temperature-cure types (Horie 2010, p. 290). Typically, the room-temperature-cure epoxy resin adhesives are used in conservation.

History and uses

Work on epoxy resins began in 1891, but patents were not obtained until 1934 (Roff and Scott 1971, p. 272; Potter 1970, p. 5). They became commercially available in 1939 in Switzerland for dentistry, and in 1945 they were developed as adhesives by Ciba (Roff and Scott 1971, p. 272). In the United States, surface coating applications were investigated by Shell as early as 1943 (Potter 1970, p. 5). Novalacs, which contain more epoxide groups than DGEBA resins and are more brittle upon curing, and other resins began appearing in the 1950s and 1960s (Meath 1990; Potter 1970, p. 6).

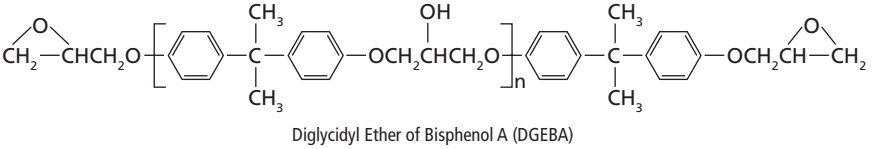


Figure 1. Typical epoxy resin

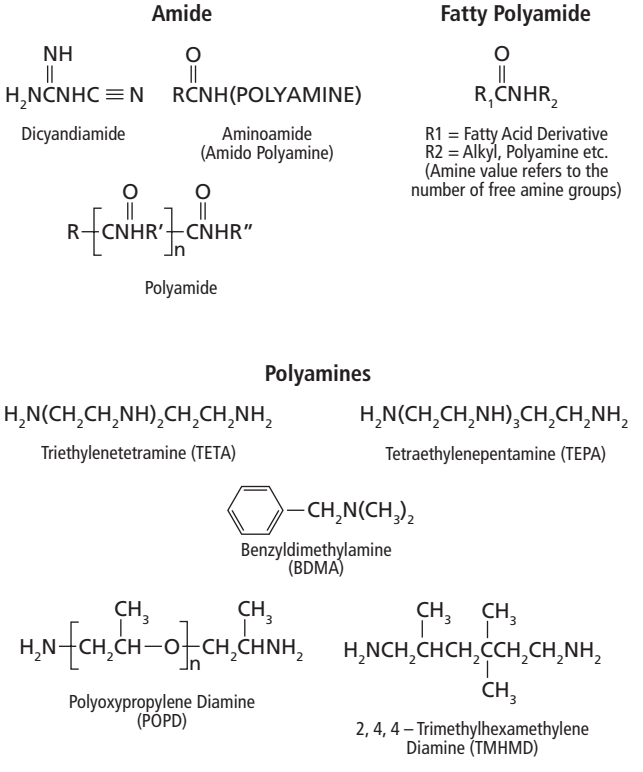


Figure 2. Typical hardeners

Epoxy resin adhesives have been used commercially for metal, stone flooring and roadway surface coatings; laminates; castings; electrical uses; caulking; sealing; and as adhesives for metals, wood, glass, ceramics, related polar materials, and fibreglass assemblies (Roff and Scott 1971, p. 272; Potter 1970, pp. 6–7).

In conservation, epoxy resins have been used as adhesives for metals, ceramic, glass, stained glass, tortoiseshell, minerals and paintings (tear repair, honey-comb support construction); adhesives and consolidants for wood and stone; surface coatings for metals and stone; and fill material for loss compensation (gap-fillers) (Horie 2010, pp. 296–297; Vidler 2006; Koob 2003, 2000; Elder et al. 1997; Munnikendam 1978).

Characteristics, limitations and/or aging studies

General

Epoxy resin adhesives are noted for outstanding toughness, versatility (adhesion to many substrates), chemical and heat resistance, high strength, low creep and high electrical resistance (Potter 1970, p. 212). Epoxy resin cohesive strength is frequently so high that failure occurs in the substrate rather than within the adhesive (Potter 1970, p. 210). While this makes epoxy resin adhesives ideal for *indirect* structural applications, they should only be used *directly* on objects with caution, especially since they are very difficult to remove. Epoxy resin adhesives have low shrinkage upon curing, so there is less stress in the glue-line, and they generally have low peel strength (Ebnesajjad 2008, pp. 80–81; Potter 1970, p. 212). Epoxy resin adhesives are solventless systems, so there is no solvent impact to worry about with the substrate; nevertheless, care must be taken to ensure that they are compatible with the substrate in question and do not harm it. They are not susceptible to microbial attack, although one study examining this for stone consolidation found growth (Domaslowski and Strzelczyk 1986).

Epoxy resin adhesives produce heat (exothermic) upon curing, especially when large quantities are mixed. Temperature rise depends on the amount used, the nature of the resin and hardener, the temperature of the reactants, and the temperature of the surroundings (Potter 1970, pp. 37–39). Heat-sensitive objects should be protected, and curing in small quantities is recommended.

Epoxy resin adhesives are noted for their good refractive index (RI) match with glass, which affords almost invisible bonds. This is one reason they are frequently used to repair glass even though difficult to remove. Glass is very polar and epoxy resins are one of the few adhesives that are polar enough to adhere well to it. Unfortunately, epoxy resin adhesives are prone to yellowing, as explained in the next section.

Yellowing

Many industrial epoxy resin adhesive studies on thermal and light stability have been done, but these were concerned with physical and mechanical properties rather than with colour change (Down 2001b). There were, however, one or two early studies that were concerned with yellowing. One stated that photo-yellowing was negatively influenced by the following hardener characteristics (Lee and Watson 1964):

- nitrogen groups adjacent to aromatic rings;
- ethylene separating nitrogen groups rather than methylene or propylene;
- high amine functionality; and
- trace impurities.

This study noted that carbonyl formation did not relate to yellowing and that the carbon nitrogen bond (C-N) was not the source of colour either. Later, other

research indicated that photochemical yellowing was caused by the formation of a conjugated structure in the bisphenol A resin (Grassie et al. 1986, 1986b, 1985, 1985b, 1985c; Allen et al. 1982; Noskov 1975; Kelleher and Gesner 1969).

One of the first conservation studies to examine yellowing compared seven epoxy resin adhesives under daylight and artificial aging (Tennent 1979). A few epoxy resin adhesives with good resistance to yellowing were identified, of which Ablebond 342-1 yellowed the least. Evidence was also presented that dibutyl phthalate plasticizer and alkyl phenol accelerator promoted photo-yellowing, and that the purity of the products and the resin/hardener ratio also had some effect. In a re-examination 25 to 35 years later of artifacts (stained glass) conserved with epoxies identified from this study as being fairly good, it was found that Araldite AY103/HY951 showed no perceptible yellowing 35 years later, which was better than predicted (Tennent 2011). Ablebond 342-1 showed just perceptible to moderate yellowing 25 years later, which agreed with predicted values from the 1979 study.

Canadian Conservation Institute study

Beginning in 1978, 55 epoxy resin adhesives were investigated at the Canadian Conservation Institute (CCI). The aim was to identify, for glass conservation, the commercial products that yellowed the least. The following summarizes the work.

Thermochemical accelerated aging

Initially, a standard technique to predict the rate of yellowing by thermal acceleration and the use of the Arrhenius equation was tested (Down 1984). Adhesives were aged at elevated temperatures, rates of yellowing were determined, and a rate at room temperature was extrapolated from a plot of log of the yellowing rate against reciprocal absolute temperature (an "Arrhenius-type" plot). The advantage of this method is that it is quicker than waiting years for the colour change to actually happen at room temperature, as yellowing occurs more rapidly at elevated temperatures. Unfortunately, this technique did not work well, as the Arrhenius predictions were in poor agreement with natural yellowing data of epoxy resin adhesives at room temperature. There are many possible explanations for this poor agreement, but one plausible theory is that a volatile component was present that catalyzed yellowing at the lower aging temperatures, but was eliminated (volatilized) at the higher aging temperatures. Because of this poor agreement with natural yellowing, thermal-accelerated aging was abandoned and natural dark aging was used.

Natural dark aging

Results of natural dark aging [22°C, ~45% relative humidity (RH)] of the 55 epoxy resin adhesives indicated that yellowing at room temperature was not necessarily a linear function with time (Down 1984b). All epoxy resin adhesives were analyzed for major components; most resins contained DGEBA, and hardeners were various amine-containing polymers (polyamines, amides, fatty polyamides). Formulations containing similar hardeners were grouped together to see if yellowing behaviour was also similar. Most epoxy resin adhesives with a modified amine-type hardener showed a greater resistance to yellowing than others tested. Epoxy resin adhesives with simple polyamines (e.g. triethelenetetramine) initially yellowed rapidly, but then slowed. Most epoxy resin adhesives with a fatty polyamide-type hardener should be avoided for glass conservation because they were highly coloured initially and rapidly discoloured more.

High-intensity light aging

The 55 epoxy resin adhesives were also exposed to high-intensity light in a xenon arc Weather-Ometer and monitored for yellowing (Down 1986). The problem with using the Weather-Ometer for light aging is that it is much more intense than typical museum lighting. The Weather-Ometer therefore accelerates light-induced reactions. Samples subjected to less intense museum lighting would require a much longer time to absorb the same total radiation as Weather-Ometer-treated samples. After such long periods of natural aging, the thermal contribution to yellowing could be significant and could not be ignored. Therefore, the light-induced yellowing value (adjusted for various lighting conditions) was added to the natural dark aging value, and life expectancies were estimated in years to unacceptable yellowing. To add the light-induced values to the natural aging value, the high-intensity rate was converted by simple proportionality to an estimated rate at a lower light intensity representative of various museum exposures. This conversion is possible only if yellowing is directly proportional to the intensity of the light, in other words, reciprocity is obeyed. This was shown to be so in a separate experiment.

Results demonstrated that light-induced yellowing also was not always a linear function of time. Most epoxy resin adhesives were almost colourless and transparent before aging (except the fatty polyamide-types). When light-induced and thermally induced yellowing was considered together, the results showed that light intensity dictates life expectancy. Under low-intensity

irradiation (museum lighting), life expectancy did not differ significantly from natural dark aging estimates.

In 1979 to 1985, when this study was done, Hxtal NYL-1 and Epo-Tek 301-2 were identified as the most resistant to yellowing of all the epoxy resin adhesives tested with Araldite AY103/HY956, Epoxyglass and Epo-Tek 301-1 performing relatively well, too. In 2012, the dark-aged samples were re-measured and re-analyzed for yellowing after 33 years and it was confirmed, happily, that these results still held true (Down 2013b). Use of epoxy resin adhesives in well-lit locations such as in the vicinity of a window is not advised.

Inhibition of yellowing

Three ways to inhibit epoxy resin adhesive yellowing were tested: addition of stabilizers, curing under an inert atmosphere and varying the ratio of the resin and hardener mixture (Down 2001b). Most of the added stabilizers that were investigated did not decrease yellowing upon aging (thermal), but some did. This reduction was dependent on the stabilizer concentration and specific to the epoxy resin adhesive chemistry. For instance, adding 2,6-di-t-butyl-4-methylphenol (BMP) stabilizer at the 1% level to EC-2216 (an epoxy with a fatty polyamide hardener) significantly decreased the rate of yellowing, but concentrations of 0.1, 2, 5 and 10% had no effect. On the other hand, when BMP was added at 1% to Araldite AY103/HY951 (an epoxy with a triethylenetetramine hardener), it significantly increased the rate of yellowing, had no effect at 2% and 10%, and significantly decreased the yellowing rate at 5%. More work on stabilizers could be useful especially since stabilizer tests were not conducted on Hxtal NYL-1 and Epo-Tek 301-2, two of the better non-yellowing adhesives. It would be interesting to see if any stabilizers could improve their performance.

None of the epoxies that were cured under an inert atmosphere of nitrogen showed a decrease in the rate of yellowing upon subsequent aging. And although certain variations in the ratio of the resin and hardener mix (usually less hardener) did reduce the rate of yellowing upon aging, it was often at the expense of a tacky, non-cured film.

Tennent and Townsend (1987) have found that some epoxy resin adhesives yellow at the surface and diminish in yellowing towards the centre of the film. For these epoxies, yellowing estimates based on film thickness could be flawed, but on a positive note, this surface yellowing could protect the bulk of the film from further yellowing. In the CCI study, it was observed that yellowing was not a surface phenomenon but constant throughout the films examined.

Effect of shelf life on yellowing

Occasionally in conservation, adhesives are used beyond their "best before" date. In order to see if this might have a negative effect on the rate of yellowing, 17 epoxy resin adhesives were stored in their separate containers of resins and hardeners for two years, made up into films every six months and aged (thermal) (Down 2001b). Generally, it was found that the rate of yellowing was increased by storing the epoxy resin adhesive components for two years. This is about one year longer than most manufacturers recommend. These tests confirmed that it is wise to use fresh epoxy resin for repairs, otherwise, joins could yellow faster if older products are used. Ordering small quantities is recommended.

Effect of adding solvents before cure

Sometimes solvents are added to epoxy resin adhesives before (and while) they cure to make them less viscous. In order to see if added solvents might have an effect on yellowing behaviour, tests with a few solvents were conducted (Down 2001b). The addition of xylene (50%) significantly increased yellowing, whereas acetone, benzene and toluene had no effect. This testing illustrates that if yellowing is important, it would be wise to carry out yellowing tests on films made from solvent-diluted epoxy resin adhesives because these solvents could increase yellowing in the cured film.

STRENGTH RETENTION

It is generally assumed that most epoxy resin adhesives will retain sufficient mechanical strength, even after extended aging, to satisfy any conservation requirement. For example, epoxy resin adhesives have been reported to be too strong for some fragile glasses (Newton and Davison 1989).

To investigate epoxy resin adhesive strength, compressive shear strength tests on glass substrates were performed before and after aging (heat, light and natural dark) on 10 products (Epoweld 3672, Tra-Cast 3012, Maraglas R658/H558, Araldite 6010/HY951, Homalite 1100, Araldite 502/HY951, Araldite 502/HY956, Hysol TE6175/HD3561, Devcon-2-ton, Canus 1000/68) (Down 1996). The results showed that the initial strength before aging was quite high (11–33 MPa). Most samples increased in strength to a maximum after aging, and then subsequently lost strength. Epoweld 3672 was the only adhesive that

showed no change under all the aging conditions used, but note that Hxtal NYL-1 and Epo-Tek 301-2, the most yellowing-resistant epoxies were not tested. It was difficult to predict with any certainty when complete strength loss under normal museum conditions would occur to any of the tested products. However, no epoxy resin adhesive tested lost an excessive amount of strength. The lowest strength observed was at least 7 MPa, supposedly adequate for normal glass bonding, according to Moser (1962).

High humidity might be the cause of some epoxy resin adhesive failures as has been suggested for old stained glass repairs in Scottish churches (Moncrieff 1975). This could depend on the type of resin and hardener used as some (e.g. aliphatic amines) are more susceptible to humidity than others (Meath 1990). Other researchers have noted curing problems and cloudy film formation in high humidity (Koob 2003; Hayes 1987; Kulikov et al. 1977). Also, according to one study, water appears to accelerate hardening and reduces adhesive strength of epoxy resin adhesives (Kulikov et al. 1977), which was also noted in another study where strength and elastic modulus of an epoxy resin adhesive (3M EC-2216) decreased upon aging in water (immersion, 35°C) (Popineau and Shanahan 2006). The degree of reduction depended on the amount of water absorbed, and time and temperature of exposure. Adhesion loss of certain epoxy resin adhesives on glass was also shown to occur when 70% RH was exceeded (Lefebvre et al. 2000). Therefore, it has been suggested that an RH of 40–55 % (at 20–25°C) is best for curing epoxy resin adhesives (Koob 2003).

HXTAL NYL-1

Besides the CCI study (Down 1986) that identified Hxtal NYL-1 as one of the most resistant epoxies to yellowing of all tested, several other studies on Hxtal NYL-1 have been conducted. One found that it had good colour stability to light exposure, but poor stability to thermal aging, variable bond strength on glass, and was partially reversible using solvents (Bradley 1990). In this study, pigmented samples were difficult to prepare and showed colour changes upon aging.

Another study compared Hxtal to other epoxy resin adhesives, ultraviolet-(UV-)reactive resins, silicone resins and cyanoacrylates after simulated winter (-20 to 15°C, 30 minutes of sunlight in a 4 hour cycle, 72 cycles) and summer (10 to 60°C, 45 minutes of sunlight in a 3 hour cycle, 96 cycles) accelerated aging, and found Hxtal to be the most suitable for edge bonding glass (Marschner 2000). However, in another study examining Hxtal NYL-1 and two photosensitive adhesives for tensile strength, stability and reversibility, the two photosensitive adhesives were stronger than the Hxtal, but discoloured more under UV exposure (Swierenga 2000). The reversibility of all three products was about equal.

When 31 adhesives (epoxy resins, polyesters, silicones, acrylics, nitrile) for glass conservation were tested (for yellowing, and short- and long-term reversibility), an epoxy resin adhesive (Eccobond 24) and a UV-cure acrylic were recommended when colour change is undesirable, but Hxtal NYL-1 was suggested for external repair where good weathering is needed (Robson 1987).

In another study, the glass transition temperature (T_g) of Hxtal was compared to two other epoxies, Fynebond and Araldite 2020 (Nunes de Silva 1998). Fynebond had the highest T_g (44–48°C) and thus might be more suitable than Araldite 2020 (40–46°C) or Hxtal (40–45°C) for use in hot countries. Choice from these three products could also depend on whether curing time or colour change was more important, as shrinkage, strength and hardness were about the same for all three (Shashoua and Ling 1998). Hxtal was best for colour, but cure time was long (96–168 h). Fynebond was best for cure time (6–24 h), but weaker than the other two and had poorer colour. Araldite was closer to Fynebond than Hxtal for cure time (12–36 h), but was not as hard and had poorer colour.

A disadvantage of Hxtal is its long curing time. Heat can be used to shorten cure, for example, in a hot room (23–30°C) or in an oven (35–40°C), but this applied heat could increase strength and yellowing of the Hxtal (Williston and Myers 1991; Williston 1983). Mixing components early and letting them cure to a desired consistency before use is an alternative. Be aware that pigments can slow cure even further (Koob 2003; Fisher 1992).

CONTROLLING REFRACTIVE INDEX

The RIs of many epoxy resin adhesives match medieval and lead glass (Tennent and Townsend 1984c). RI of epoxies can be slightly changed by adjusting the amount and type of hardener used and by adding other components such as plasticizers and diluents. RI of epoxy resin adhesives does not change upon natural aging or from batch to batch (Tennent and Townsend 1984).

In another study, the RI of Ablebond 342 and Hxtal NYL-1 was changed by using halogenated epoxide compounds and mixtures of the two resins. When results were examined, halogenated compounds marginally changed the RI, and after one year of

dark aging, the Hxtal films were unacceptably yellow, while Ablebond was acceptable (Augerson and Messinger 1993; Messinger and Lansbury 1989). Mixtures of the two resins changed the RI from 1.515 to 1.57 and, after one year of dark aging, the mixtures had not yellowed unacceptably.

ADDITIVES AND EPOXY RESIN ADHESIVES

Tennent (1979) has identified several lightfast dyes for epoxy resin adhesives. The dyes dispersed easily in the epoxies, but had lower lightfastness than pigments, which dispersed with difficulty. Also, epoxy resin adhesives have a deleterious effect on the light stability of anthraquinone disperse dyes, but radical traps help to inhibit this (Allen et al. 1983). The effect of adding dyes to resins is complex: yellowing could dominate colour change, but partial recovery after light fading is possible (Tennent and Townsend 1984b). On the other hand, adding pigments can slow the cure rate of epoxy resin and change the T_g somewhat, but in one study, the final cure state was not significantly different than if no pigment was present (Cope et al. 1995).

Some fumed silica fillers change epoxy resin adhesive colour to bright yellow, while cellulose nitrate addition changes epoxies to orange (Tennent 1995). This later finding could be important in previously glued cellulose nitrate joins that are re-glued using epoxy resin adhesives.

The effect on cure rate of adding solvents to epoxy resin adhesives is well defined (Selwitz 1992; Kotlik et al. 1981). Hydroxyl compounds (e.g. alcohols) catalyze curing, while aromatic hydrocarbons or mineral spirits have no effect on cure rate, and carbonyl compounds (e.g. acetone) retard curing.

EPOXY RESIN ADHESIVE USED WITH CYANOACRYLATES

Cyanoacrylates are often used in glass repair as temporary adhesives to hold pieces before permanent epoxy resin adhesive is introduced. However, cyanoacrylates can alter the appearance (yellowing, crizzling or change in opacity) of the epoxy upon curing. For instance, Epo-Tek 301 with Instabond showed alteration, while Hxtal NYL-1 with Krazy Glue showed little alteration (note: the amount of cyanoacrylate was very high in these experiments) (Monahan 1994). Also, when four different cyanoacrylates (Instabond, Krazy Glue, Mastercraft Superglue, The Gripper Superglue) were used with two different epoxy resin adhesives (Hxtal NYL-1, Epo-Tek 301-2), mixtures with Hxtal (except Hxtal/Gripper) remained stable and did not yellow, while mixtures with Epo-Tek yellowed under light exposure (Miller 1996).

OTHER CONSERVATION STUDIES

Paintings

When the mechanical behaviour of various adhesives with various fillers was examined for rejoining panel paintings, Araldite 2014 with and without rye-flour filler was stronger than the wood alone (Young et al. 2002).

Wood

When four barrier coatings (Butvar B-98, Paraloid B-72, Titebond Liquid Hide Glue and Paraloid B-67) were tested with an epoxy resin adhesive (Araldite 1253) on wood samples, Paraloid B-72 was the most effective barrier; shear strength was comparable to that with no barrier coating and the bond reversed easily in xylene vapours (Ellis and Heginbotham 2004). However, when Paraloid B-67 was used as a barrier coat, low shear strengths resulted and reversal was not as good. Butvar B-98 and Titebond gave fairly good strength results. The Butvar B-98 reversed in ethanol, while the Titebond reversed easily in a microwave.

Plaster

Aqueous acrylic/epoxy consolidants have been proposed for plaster consolidation (Phillips 1995). These consolidants use an alkali-soluble acrylic that helps to solubilize the cycloaliphatic epoxy. Once in place, the consolidant crosslinks with time. The system is favoured over consolidants in solvents because fire and toxicity hazards are removed. In preliminary subjective testing, hardness, toughness, degree of crosslinking, moisture resistance and strength were good, but further testing under high and changing RH is needed.

Ceramics

Nine different adhesives including an epoxy resin adhesive (Araldite AY103/HY991), poly(vinyl acetates), cellulose nitrate, acrylics and cyanoacrylates were appraised for tensile strength, yellowness index (before and after cyclic thermal aging), bond thickness, working time and overall hazard for Spanish medieval ceramics (Castro and Doménech Carbó 1999). Overall, Paraloid B-72 gave the best results, while the epoxy resin adhesive gave excellent bond strength, but other properties were rather unsuitable.

Stone

Epoxy resins have been extensively studied for the consolidation of stone and the pertinent work is summarized in this section. To begin, Domaslowski (1988b) outlined the desirable properties for stone

monument consolidation, which most of the following studies use as criteria for a good stone consolidant. These properties are that the consolidant should:

- penetrate the interior easily and allow for capillary penetration to assure uniform distribution in the stone pores;
- not migrate to the surface when the solvent evaporates (if a solvent is used);
- not obstruct the pores in the stone;
- not discolour;
- increase mechanical resistance;
- increase hydrophobicity; and
- increase resistance to water-soluble salts, micro-organisms and aging.

An early stone consolidation study showed that increases in epoxy concentration (in a solvent) increased epoxy content and penetration depth in the stone (Kotlik and Zelinger 1981). However, viscosity limited the penetration, which proceeded by diffusion rather than by capillary action. Increased immersion time and decreasing pressure in the impregnation vessel also facilitated penetration. A later study suggested that organosilane consolidants penetrated better than epoxy consolidants and allowed better gas permeability, but they did not consolidate as effectively as epoxy resins (Kotlik and Zelinger 1983). Both types of consolidants provided water repellency, but did not fix water-soluble salts in the stone. According to these researchers, to be a good stone consolidant, the epoxy resin should have a "low molecular weight of resin and hardener, a suitable type and amount of solvent, an effective impregnation method and the prevention of reverse migration" (Kotlik et al. 1983, p. 75).

Domaslowski (1988b) exploited the fact that diluting the epoxy resin component with solvent influences the curing time. He found that the best combination of solvents was alcohol (methanol) with an aromatic hydrocarbon (benzene, toluene). The more alcohol present, the better the hardening. This study further found that dampening the stone beforehand with solvent improved capillary rise rate, and best results were achieved when resins hardened in <10 days and solvents were prevented from evaporating. There was a strength increase in the stone, but a decrease in water absorption was attributed to diminished porosity.

A review by Selwitz (1992) of epoxy resins for stone consolidation described early work to improve epoxy penetration into the stone by using high temperatures (70°C) with protective covers to prevent resin oxidation, low viscosity epoxies with reactive diluents, or conventional epoxies with silanes. These treatments did not work (penetrate) very well or were either difficult to implement or left a discoloured surface crust. Also discussed in this review was the molecular rigidity of aromatic-type epoxy resins, which led to their high viscosity and low penetration into stone. When the aromatic structure is hydrogenated, the resulting epoxy resin has lower viscosity and is less likely to discolour (e.g. Eponex, EP2101). It was additionally mentioned that hardeners are generally responsible for the good adhesion to the substrate, but they can be volatile, toxic and the primary source of discolouration. More stable, less volatile and less toxic products are possible by modifying the hardener either by making derivatives or by pre-reacting parts with the resin beforehand. This can make the epoxies less viscous with good light stability, but cure time is slowed.

Also noted was epoxy penetration depth, which depends on the nature of the stone, composition and viscosity of the consolidating system, and application method. According to Selwitz's review, marble of low porosity and degraded limestone are not good candidates for epoxy consolidation. It is better to use a water repellent on these stones. On the other hand, porous sandstone (at least 14% porosity) and limestone (at least 28% porosity) benefit from epoxy consolidation, but the treatment should have careful design and testing. Degraded marble also gave good results. Other things discussed in this review that can affect consolidation are that:

- salts must be removed from the stone beforehand or they will interfere with the consolidation;
- solvents affect the epoxy/hardener polymerization rate;
- brushing is not good for low porous stone;
- recycling the consolidant (wicking it over surface in constant stream) is good; and
- wrapping the stone to prevent epoxy and solvent evaporation is also good.

In addition, Selwitz noted that, in Europe, they use a consolidation method that involves an epoxy with an alcohol and an aromatic hydrocarbon solvent and a wrapping technique (as mentioned above), while in the United States, they follow a spraying method (epoxy in acetone) and eliminate discolouration by abrading afterwards. According to this review, specifications for good epoxy consolidation are:

- all salts must be removed (sodium <0.02%);
- impregnation must be to a depth of 2.5 cm;
- compressive strength must be increased by 50%;
- water vapour permeability must be lowered by no more than 10%; and
- liquid water absorption must be no more than 5% after 48 h.

The overall conclusion from this review was that for large buildings, the US approach is more reasonable, while for smaller stone artifacts, the European method is more suitable.

Evaluation of a cycloaliphatic epoxy resin [in isopropanol (Eponex 1510 + Jeffamine T-403)] and acrylic-epoxy mixtures [in isopropanol/water (Neocryl-520 + ERL-4221 and Carboset-5141H + ERL-4221)] in various types of in-situ aging and simulated laboratory aging for hot humid tropical climates by different tests (depth of penetration, curing in high RH conditions, microabrasion resistance, water abrasion resistance, change in porosity) showed that they improved the water and particle erosion resistance of weak Maya limestone (Kumar and Ginell 1995).

For sandstone, silane derivatives work fairly well because "they penetrate deeply, improve mechanical strength and durability, and are relatively safe and easy to apply" (Ginell et al. 1995, p. 824). However, for limestone, which is most affected by environmental deterioration, epoxy resins work better than acrylics or silanes, and the epoxy resin amine group is responsible. Wrapping to minimize reverse migration before curing and exposing the stone to modest outdoor exposure afterward helps to reduce darkening, while vacuum impregnation gives deeper penetration.

In a study examining various consolidants (epoxy resin, silane, polyester, poly(vinyl chloride), polyacrylamide, ethyl silicate) on French limestone after natural aging (18 years), all treatments lost some of their consolidating effect on the surface, but not necessarily in the depth of the stone (Vallet et al. 2000). The study indicated that the silane and the epoxy resin were the best, which was attributed to the decrease in porosity and capillary properties.

When epoxy resin, Epojet, was examined for use in restoring historic buildings, it was found that full cure only occurred after four months; heating (50°C) increased the T_g and the strength (indicating that increased crosslinking had taken place); exposure to high RH (98%) decreased T_g (indicating plasticization); and not all water was desorbed from the substrate after immersion (Frigione et al. 2006).

The effectiveness of stone consolidation is directly related to the amount of consolidant absorbed. When this was studied by Pinto and Rodrigues (2008), it was found that the first brush application conveys most of the consolidant into the stone. Also, for carbonate stones, porosity must be >18% for good absorption. Epoxy resins and acrylics consolidate carbonate stones by only a few millimeters, while ethyl silicate consolidates deeper, but not as strongly.

And, finally, when thin granite was backed with fibreglass-mat using an epoxy resin adhesive, the peel strength was considerable, even after severe cycled conditions, and thus was thought to afford good support for thin veneer slabs used in historic building restoration (Roper and Poulos 1988).

Metals

Epoxy resin adhesives should not be used on copper because they can cause corrosion (Lee and Neville 1967, p. 6–52). This corrosion can be enhanced in the presence of water and is also dependent on the permeability of the epoxy to water and the presence of some fillers.

Coatings of epoxy resin have been shown to improve the stability of archaeological iron by preventing splitting, flaking, weeping or rusting, but have not been investigated thoroughly to see if the benefits outweigh the possible dangers (irreversibility, breakdown of resin, etc.) (Keene 1984).

HEALTH ISSUES

Certain glycidyl ethers may produce testicular atrophy and hemopoietic abnormalities (in lab animals) (HESIS 1989; NIOSH 1978). Chronic toxicity and skin carcinogenicity of some commercial epoxy resin adhesives have been noted in some of the earlier products but, supposedly, the newer, more modern, products with less epichlorohydrin contaminant do not cause cancer in animals (HESIS 1989; Cooper 1981). Dermatitis may also occur from contact with the resin, hardener or additive (Mathias 1981). Avoid skin contact and breathing epoxy resin adhesive fumes; wear gloves and protective glasses; mix components in well-ventilated areas; and follow clean work practices (HESIS 1989; NIOSH 1976).

SOLUBILITY AND/OR REMOVABILITY*

Epoxy resin adhesives are thermosetting resins and cannot be dissolved by solvents. The cured resins are relatively unaffected by water, hydrocarbons, alcohols and some inorganic reagents (50% sulfuric acid,

* Disclaimer: The solvents listed in this section are ones that might dissolve the polymer or adhesive in question but are not necessarily recommended for conservation treatments. Some may be extremely toxic or may contain elements (e.g. chlorine) that may be harmful to certain materials in your object (artifact). Research the solvent before purchase or use to know its toxicity and chemical nature. It is strongly recommended to test all solvents before use to make sure that they do not adversely affect your object.

10% nitric acid, concentrated hydrochloric acid, 20% sodium hydroxide) (Roff and Scott 1971, pp. 268–269). They may soften slightly in ketones and can be swollen by various solvents depending on the epoxy resin adhesive (Roff and Scott 1971, p. 269). Polyamine-cured resins tend to have poor cold water resistance (which should aid removability), while polyamide-cured resins are not as affected by cold water, but show poor resistance to hot water and alkalis (Shields 1984, pp. 44–45).

The swelling of 5 different epoxy resin films in 13 solvents was investigated by Rémillard (1989). A mixture of dimethylformamide (DMF) and tetrahydrofuran (THF) (1:1) was the most effective, followed by chloroform, methylene chloride and THF alone. The least effective solvents were alcohols and ketones. Ablebond 342 was found to be the most easily removed epoxy resin adhesive tested, followed by Epo-Tek 301-2, Epo-Tek 301-1, Araldite 502/HY956 and Epoxyglass. Different epoxies have different affinities for solvents, presumably because their chemistry differs. For instance, Araldite 502/HY956 swelled more in DMF/THF, whereas Epo-Tek 301-2 swelled more in chloroform. Acetic acid had some effect on swelling, but solutions of hydrochloric acid, sulfuric acid, sodium hydroxide, sodium borate, sodium chloride and even pineapple juice had no effect, and aging at 100°C reduced the swelling, indicating less removability.

Another study investigated swelling of the bond and time necessary to separate epoxy glass slide bonds using various chlorinated solvents (Scott 1986). The results showed that mixtures of dichloromethane with methanol and xylene (90:5:5 or 80:10:10) were most effective, aging increased time to separation, and Hxtal NYL-1 was unaffected by many chlorinated solvents. The other seven epoxy resins tested (Araldite AY103/HY956, Ablebond 342, Epo-Tek 301, Panel Fix, 5 Minute Araldite, Polystik, Epoxy Bond Putty) were more affected by the chlorinated solvents.

Acrylic barrier coating (Paraloid B-72) increased reversibility when used with an epoxy resin adhesive (Araldite AY103/HY991) for stone (Podany et al. 2001). The barrier coating must be "relatively thin, conformal, continuous and fully dried" before the epoxy resin adhesive is introduced to afford this reversibility (p. 29). It was sufficiently strong for structural joints, but long-term creep was not examined.

Some have tried unconventional means to remove epoxy resin adhesives. For instance, an investigation of enzymes to reverse UV-aged epoxy resins found that there was absolutely no effect (Harvey 1994). Another investigated diffusion of various acids (8%) into epoxy resin adhesives and found that propionic acid destroyed the epoxy resin adhesives within 10 days and that the order of diffusibility from greatest to least was propionic, formic, acetic, sulfuric and hydrochloric (Kalenda 1992). Diffusion depended on the epoxy resin adhesive structure and epoxide content.

Lasers have been evaluated for epoxy resin adhesive removal and the following has been found (Scheerer et al. 2003):

- The Nd:YAG laser was not as effective as the Er:YAG laser in removing epoxy resin adhesives; wavelength was important.
- Once irradiated, the epoxy resin adhesives were soluble in milder solvents (acetone, ethanol).
- Epoxy resin adhesive removal from porous surfaces was difficult owing to the penetration effect; however, surface removal was possible.
- Considerable heat was generated during ablation, which could affect the object and must be considered.
- Fillers in the epoxy resin adhesive could be physically stressed when ablated and this stress could be transferred to the artifact.
- Generally, a yellowed epoxy resin adhesive was more affected than a clear or colourless one.
- Discolouration of the object surrounding the irradiated area often occurred, so it is wise to pre-test on an inconspicuous spot prior to any treatment.

Soluble Nylon

Setting mechanism	Category	Water absorption (%)	Refractive index
evaporation	thermoplastic		
Glass transition temperature (°C)	**Viscosity (mPa.s)**	**pH**	**Coefficient of linear expansion /°C**
melting point (T_m) decreases with increased amide substitution (sub): 0% sub T_m 264 22% sub T_m 185 32% sub T_m 130 35% sub T_m 150–160 50% sub T_m 100–110 highly sub T_m ~22			

(Sease 1981; De Witte 1975)

Types, synonyms and/or trade names

Calaton, Elvamide, Maranyl Nylon, *n*-methoxymethyl nylon, Ultramid, Zytel

Chemistry and/or preparation

Nylon is a hard, non-elastic, high-melting, virtually insoluble polymer. These properties are attributed to extensive hydrogen bonding between the extended chains in the crystalline unit. If nylon is chemically modified by substituting alkoxy groups for the amide hydrogen, the resulting polymer, called "soluble nylon", is found to be more soluble and flexible than the parent nylon (Sease 1981; De Witte 1975). The most common type of soluble nylon is *n*-methoxymethyl nylon (**Figure 1**), which is made from heating nylon with formaldehyde, formic acid and methanol to produce short side chains (Horie 2010, p. 195).

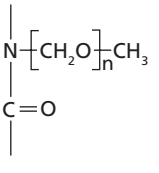


Figure 1. n-*Methoxymethyl nylon (Horie 2010, p. 195; Fromageot and Lemaire 1991).*

History and uses

The first patents for soluble nylon were applied for by Du Pont de Nemours (1939–45) (De Witte 1975).

In industry, soluble nylon has been used for stiffening and bonding fabrics, and as an adhesive and varnish for abrasion resistance or anti-slip finishes on textile threads (De Witte 1975; Roff and Scott 1971, p. 223).

In conservation, soluble nylon has been used for sizing paper; consolidation of friable stone, bone, ivory, pottery, paper, pigments, wood, wall paintings, canvas, ethnographic objects, metals and textiles; and as an adhesive for leather (Horie 2010, pp. 195–196; Sease 1981; De Witte 1975). Soluble nylon was used in palaeontology as a temporary support while washing salts out of fossils and as a consolidant for chalk fossils (Elder et al. 1997). By the late 1960s, soluble nylon use was discouraged and, by the 1980s, was strongly discouraged.

Characteristics, limitations and/or aging studies

Modification of nylon to create soluble nylon makes it more flexible, initially (Horie 2010, p. 195). As the degree of substitution (alkoxy group) increases, so does the pliability and elasticity (De Witte 1975). It was this flexibility that made soluble nylon once desirable for conservation, along with the fact that

it dries to a matte finish, allows passage of water and salts, and has good adhesive properties (Sease 1981). However, over the years, several studies have indicated that, upon aging, soluble nylon crosslinks, becomes insoluble and brittle, and shrinks (Horie 2010, p. 195; Sease 1981). The problems were so difficult that antioxidants were once suggested to extend the ability of soluble nylon to withstand exposure to light (Fromageot and Lemaire 1991). Other studies identified more drawbacks with its use such as:

- It had a fairly low glass transition temperature so it could collect dust and dirt (Buys and Oakley 1993).
- It supported microbial growth (Kyi and Drew 2000).
- Addition of soluble nylon to acidic paper initially made it fairly neutral and increased its fold endurance, but when thermally aged, it became acidic again and lost strength (Bass 1976).
- The use of soluble nylon as a temporary facing or consolidant for paper was discouraged as residues were left even after extensive soaking in various solvents (methanol/water, industrial methylated spirits/water, *n*-propanol/water, ethylene glycol, thymol/methanol, diacetone alcohol, benzyl alcohol, *n*-methyl pyrrolidone, isopropanol, isopropanol/water, *n*-butanol, *n*-propanol/thymol) (Daniels 1985).
- When objects containing vegetable fibres that had been treated with soluble nylon were inspected 10 to 15 years later, appearance, flexibility and breaking stress of the fibres were unfavourably altered (Shashoua et al. 1990b).
- Stone sculptures fixed with soluble nylon while desalting became grey-brown after two years soaking and required very aggressive removal methods (De Witte 1975).
- Tests showed that soluble nylon became very brittle, non-elastic, prone to tears and insoluble after three months soaking (De Witte 1975).

Because of these difficulties, soluble nylon is not recommended for conservation use.

SOLUBILITY AND/OR REMOVABILITY*

n-Methoxymethyl nylon is soluble (when fresh) in warm alcohols (ethanol or methanol with a little water) (Horie 2010, p. 195). As substitution increases, so does its solubility in ethanol (De Witte 1975). With time, soluble nylon becomes insoluble due to crosslinking (Sease 1981). This usually happens in the presence of mild acids and heat, but appears to happen over time even in relatively favourable conditions (Sease 1981).

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* Disclaimer: The solvents listed in this section are ones that might dissolve the polymer or adhesive in question but are not necessarily recommended for conservation treatments. Some may be extremely toxic or may contain elements (e.g. chlorine) that may be harmful to certain materials in your object (i.e. artifact). Research the solvent before purchase or use to know its toxicity and chemical nature. It is strongly recommended to test all solvents before use to make sure that they do not adversely affect your object.

Silicones (Polysiloxanes) and Silanes

Setting mechanism	Category	Water absorption (%)	Refractive index
chemical cure	thermosetting	rubbers: depends on filler – 0.4–10% after 30 days resins: ~0.03–0.2% after 24 h, 3 mm thickness	rubbers 1.404 resins 1.405–1.49
Glass transition temperature (°C)	**Viscosity (mPa.s)**	**pH**	**Coefficient of linear expansion /°C**
polydimethylsiloxane -125 polymethylphenylsiloxane -112			rubbers 50–400 $\times 10^{-6}$

(Shields 1976, p. 299; Roff and Scott 1971, pp. 462–463)

Abbreviation

SI silicone plastics (not to be confused with Si for silicon, the element)

Types, synonyms and/or trade names

alkoxysilane, alkyl silicates, Conservare, Dow Corning, Drisil, Funcosil, General Electric (GE), organosiloxane, orthosilicates, polysiloxane, Rhodorsil, Silastic, Silex, silicic acid esters, silicone esters, Silicoset, Silygutt, Tegovakon (Wheeler 2005, p. 8)

Chemistry and/or preparation

Silicone adhesives, consolidants and sealants are considered partially inorganic because they contain both inorganic (silicon) and organic (carbon) structures together in one compound (Dean 1990). They were mistakenly called "silicones" because they were originally thought to have similar structure to "ketones" in organic chemistry. However, there is a double bond between the carbon and oxygen in a "ketone" and only a single bond between the silicon and oxygen in a "silicone" (New World Encyclopedia). The correct chemical name for silicones is polymerized siloxanes or polysiloxanes.

The naming of silicon-based compounds is very similar to the naming of carbon-based compounds, for instance, silane and methane (**Figure 1**) (Charola 1988). Substitution on the silane molecule and naming is similar to substitution on the methane molecule and naming.

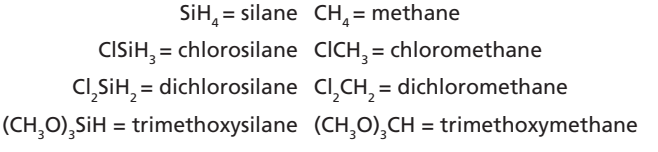

Figure 1. Naming of silicon compounds and similarity to organic-based naming

The most common reactive groups are hydrogen (H), fluorine (F), chlorine (Cl) and alkoxy [RO, where R indicates an alkyl group (e.g. methyl, ethyl, butyl) and O is oxygen] (Wheeler 2005, p. 14). The RO group is called methoxy, ethoxy, etc., depending on the alky group. The naming system for silanes with alkoxy groups follows the same standard nomenclature used for organic compounds (**Figure 2**).

The silicon-carbon bond is unreactive (**Figure 3**) and stable to hydrolysis, while the bonds with H, F, Cl and RO groups are reactive.

The silicon atom can form bonds with four neighbouring atoms. In silicone chemistry, these are usually oxygen or carbon, and the number of oxygen atoms attached to the silicon defines its functionality. These are monofunctional (M) for one oxygen present, difunctional (D) for two oxygen atoms present, trifunctional (T) for three oxygen atoms present, and tetrafunctional or quadrafunctional (Q) for four oxygen atoms present (**Figure 4**). The R groups in **Figure 4** indicate a carbon-containing structure that is unreactive, as mentioned above.

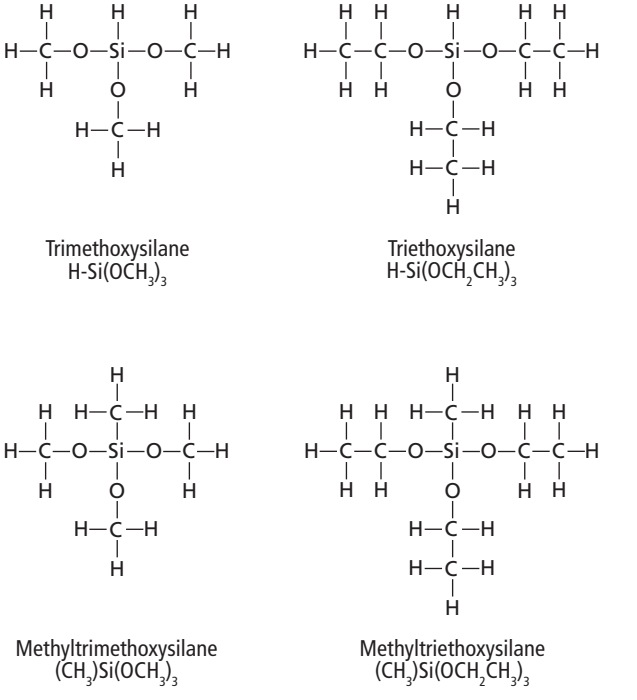

Figure 2. Naming silane compounds

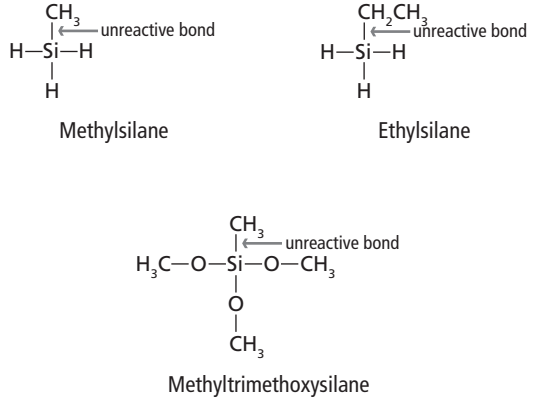

Figure 3. Examples of compounds with unreactive silicon-carbon bonds

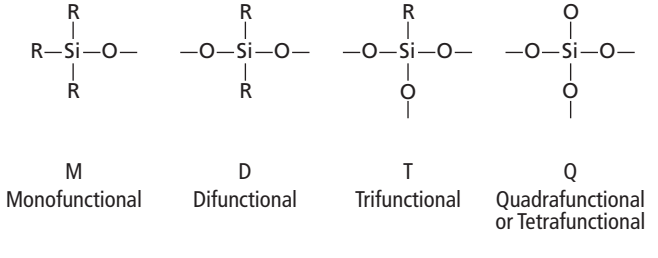

Figure 4. Different functionalities in silicone chemistry

When there is a hydroxyl (OH) substitution on the compound, it is called a silanol (**Figure 5**), very similar to alcohols in organic systems.

R_3SiOH = silanol

Figure 5. Silanol

When silanols react together, they produce a siloxane bond (**Figure 6**). Siloxane units always have one oxygen atom between two adjacent silicon atoms—the basis of a "silicone". These Si-O-Si bonds are quite stable.

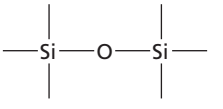

Figure 6. Siloxane unit

Figure 7 shows an example of two alkylalkoxysilanes reacting to form a siloxane bond. The reaction is initiated by water vapour either from the atmosphere or on the substrate surface. The reaction by-products can be carboxylic acids (e.g. acetic acid), alcohols or amines (Dean 1990). These by-products evaporate after cure.

M, D, T and Q units combine in different ways to produce compounds with differing properties. The compounds of interest to conservation are silicone rubbers and resins.

SILICONE RUBBERS

Silicone rubbers are linear chains of D units with M end-units (**Figure 8**) (Roff and Scott 1971, p. 459). Sometimes Q units are added to the formulation to create crosslinks between the linear D chains (Mayer 1999).

The polysiloxane rubber created with D, M and Q units can have different properties depending on the R groups (Roff and Scott 1971, p. 459). For instance, dimethylsiloxane is general purpose; methylphenylsiloxane is cold-resistant; methylvinylsiloxane is crosslinkable; those containing H, OH, alkoxy or acyloxy groups are room-temperature vulcanizates (RTVs); and those containing cyanoacryl or fluoro groups are swell-resistant. Silicone rubbers can be one- or two-part systems.

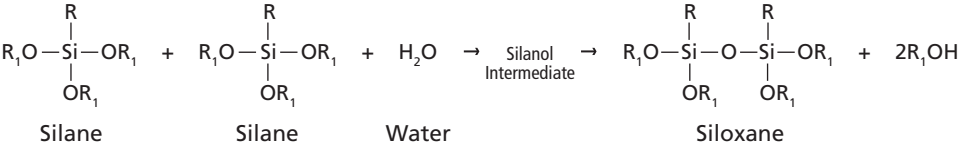

Figure 7. Alkylalkoxysilanes reacting to form siloxane bond

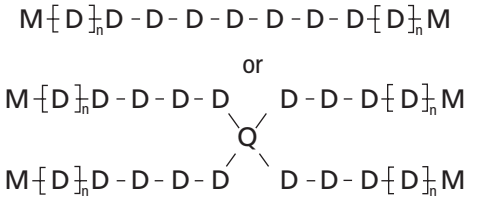

Figure 8. Composition of SI rubbers

SILICONE RESINS

Silicone resins contain more T units with more branching points (crosslinks), giving a higher degree of cure (Roff and Scott 1971, p. 459). Again, the R group imparts different properties to the resin. For instance, if the R groups were methyl, the resin would be hard and weak; if they were longer alkyl groups, the resin would be softer, less heat-resistant and more soluble; and if they were phenyl, the resin would have increased strength, heat resistance, solubility and brittleness. Sometimes, both methyl and phenyl groups are used to obtain an intermediate result.

For consolidants, especially stone consolidants, the resin of choice is one with T or Q units because these have more reactive groups to form a three-dimensional network in the stone (Wheeler 2005).

In summary, silanes are monomers that polymerize to polysiloxanes (if an alkyoxysilane is used to start) which have silicon-oxygen bonds as described above. Rubbers (typical sealants and RTV types) are polysiloxanes composed of mono- and di-functional units while resins (stone consolidants) are polysiloxanes composed of tri- and tetra-functional units.

HISTORY AND USES

Organosilicon compounds were first prepared in 1857 to 1863, but it was not until much later, after exhaustive study on them and polysiloxane chemistry, that silicone went into production (Roff and Scott 1971, pp. 468–469). In the 1940s, a chemist at General Electric US and also an independent one in Germany began working on synthesizing the silicone starting materials (Miller 2006). Silicone rubber was first commercially produced in 1946 in the United States by Dow Corning, which was formed especially to exploit them, and then production began in the United Kingdom in 1954 by Albright & Wilson Ltd., while in Russia production began in about 1947 (Roff and Scott 1971, pp. 468–469).

Silicone rubbers have been used for many items such as for RTV adhesives; for seals and gaskets to operate at low- or high-temperature conditions or to operate outdoors; for glass-fibre fabric for heater ducts; for electrical insulation; for non-stick rollers and belts; and for surgically implanted body parts (Roff and Scott 1971, pp. 467–468).

Silicone resins have been used for heat-resistant and/or water-repellent coatings and paints; impregnation of paper, fabrics and tapes; and release and non-stick agents (e.g. moulds, release papers) (Roff and Scott 1971, p. 468).

In conservation, silanes, and in particular tetraethoxysilane (TEOS, a.k.a. ethyl silicate, tetraethylorthosilicate, silicic acid ethyl ester), have been used extensively for stone consolidation (Horie 2010, pp. 276–280; Wheeler 2005, p. 8; Shashoua 1995, pp. 322–324). Silane coupling agents have been added to adhesives to increase adhesion to glass (Horie 2010, p. 277). Silicone oils have been suggested for metal and fossil protective coatings and as release agents. Silicone RTV rubbers have been used for repairing stained glass and making moulds, as well as in caulking for display cases. Also, rubbers have been used in a pressure-sensitive relining fabric for paintings (Fabri-Sil), and have been suggested for use as a picture varnish (Vani-sil) (Horie 2010, p. 281; Fieux 1984).

CHARACTERISTICS, LIMITATIONS AND/OR AGING STUDIES

GENERAL

Vulcanized silicone rubbers retain their properties over a wide temperature range (low and high) and have exceptional resistance to prolonged heating, to oxidative degradation, and to ozone and ultraviolet radiation attack (Dean 1990; Roff and

Scott 1971, pp. 467–468). They have good chemical resistance, excellent water repellency and useful electrical properties, due to low polarity (Miller 2006; Dean 1990; Roff and Scott 1971, pp. 467–468). Low, medium and high strength RTV formulations are manufactured, but, generally, they have inferior performance compared to organic rubbers at room temperature; however, at elevated temperatures, the silicones have equal or better performance (Dean 1990; Roff and Scott 1971, p. 458). Silicones have excellent resistance to weathering and do not shrink or crack in long-term outdoor use, which makes them good construction sealants (Park et al. 2003; Oldfield and Symes 1996; Dean 1990).

The rubbers have very low glass transition temperatures and thus can pick up dust (Horie 2010, p. 276). Also, silicone rubbers sometimes contain un-reacted silicone oils that can migrate into porous materials or creep in joints (Horie 2010, p. 281; Shashoua 1995, pp. 322–324). Acetic-acid-liberating rubbers are usually high strength adhesives and sealants, but have corrosion potential (Romig and Bush 1979). Alcohol- and amine-liberating types pose less corrosion risk, but may have lower strength and adhesion, and a slower cure (Romig and Bush 1979). The alcohol curing types are frequently used to seal glass in museum display cases (Shashoua 1995, pp. 322–324).

Moulds

Silicone rubbers are frequently used for making moulds of objects because they are easy to use and give good reproductions. The rubber should have low tear strength to reduce stress on the object, high elongation at break to pull out of the mould without damage, and good hardness to retain its shape after (Horie 2010, p. 281). Silicone rubbers contain oils, so a mould barrier is not necessary unless the substrate is porous (e.g. stone), in which case an impermeable barrier should be used (Shashoua 1995, pp. 322–324). This was illustrated in a study that tested two silicone rubbers (Elite Double, Silicoset 105) and found them suitable to mould ceramics because of their good replication, no measurable shrinkage upon curing, and no curing inhibition to a modeling compound used; however, there was migration of oils into the substrate, which did pose problems (Ecclestone and Daniels 1999).

Microbial resistance

One study showed that various silanes had a range from good to poor resistance to microbial attack when tested: Tegovakon V had good resistance, GE Silicone 1048, Conservare H, and Conservare OH had medium resistance, while Conservare H40 had the least resistance (Koestler 2000).

Paper

Aminopropylmethyldiethoxysilane (AMDES) was shown to deacidify, create an alkaline reserve and improve the mechanical properties of paper (Dupont et al. 2011). After hygrothermal aging (100°C for 2, 5 or 10 days in a closed glass vessel) these properties were partly preserved but less so on highly degraded papers. It was suggested that the yellowing on the highly degraded papers might be caused from the reaction of the amine in the AMDES with the carbonyl groups on the cellulose in paper.

Photographs

One-part-acetic-acid-cure silicones have been used to face-mount colour photographs onto Plexiglas. This technique was developed in the 1970s (called Diasec) to heighten colour contrast in photos. The silicone cures in an air-tight space between the photo and Plexiglas using moisture from the gelatin emulsion. Silicone was used because of its stability, inertness and resistance to temperature and relative humidity (RH). Some problems can develop if residual chemicals from poorly processed prints are trapped. Also, the process is not reversible, so the Plexiglas becomes part of the artifact and is subject to scratching from dusting (Plexiglas picks up dust easily). In one study, it was shown that the process slowed yellow staining, but increased colour shifts in the print, which depended on the photographic materials, print process, mounting materials and/or storage environment employed (Jürgens 2001). Other studies have shown that the photographs mounted with this process discolour faster than unmounted photographs and can delaminate, while the yellow and magenta colourants in the photos visibly degrade and the acetic acid released from the silicone seems to accelerate degradation (Zorn and Dobrusskin 2011; Wei 2008).

Paintings

In the early 1980s, a silicone pressure-sensitive adhesive (PSA) product (Fabri-Sil) was suggested for relining paintings (Fieux 1984). It had low reactivity, was chemically neutral, did not migrate into porous materials, could be applied without heat or pressure and was removable with naphtha. In a study of various lining procedures that did not employ heat or moisture, the application of Fabri-Sil with light manual pressure produced relatively weak bonds; however, when activated with petroleum spirit, tack and bond strength increased and appeared at the time to give good results (Phenix 1985). It also appeared to have some resistance to creep and deformation under constant stress. Despite its advantages, it did not become a viable treatment option for conservators because of its unpredictability

with loss of bond strength and delamination in lining applications over time (Belloli 1993).

WOOD

In a comprehensive study of 33 different gap-fillers for wood, silicone rubber with microballoons was recommended as the best gap-filler because of its low compression modulus, in comparison to the other materials studied (Grattan and Barclay 1998).

POLYURETHANE FOAM

The resistance to compression of polyurethane ester foams consolidated with AMDES was improved on aged and unaged samples. The deposition is larger on aged foams due to the reaction with carboxyl groups that form in the foam during the thermal degradation process. The colour of the foam was somewhat affected by AMDES consolidation on the unaged samples, but on the aged samples AMDES reduced the colour change. The natural texture of the foam was preserved when AMDES was used.

GLASS

Various silicones (Seurasil 1503, Silygutt, M.S.W.2A, Rhodorsil 3B) were found suitable for stained glass repair because they remained supple and translucent after weathering tests (Bettembourg 1975, 1972).

FOSSILS

A neutral-cure silicone RTV (Silastic RTV 1080) was tested and selected for gluing fractured pieces of trackways in mudstone because, upon cyclic boiling and heating, it took longer to fail than the other epoxy resin adhesives that were also tested, was easily reversible with peeling, was flexible, and did not discolour (Agnew 1984).

STONE AND MASONRY

Depending on type, alkoxysilanes tend to evaporate before the hydrolysis reaction can happen, especially since this reaction is usually very slow (Mayer 1999). Consolidation can be improved either by constantly replenishing the consolidant on the surface, by covering the surface with an impermeable membrane and/or by adding slow evaporating solvents (Horie 1987, p. 159).

In a study of silicone resins [Sandsteinverfestiger OH (non-hydrophobic), X 54-802 (hydrophobic), Drisil 773 (water repellent)] for stone conservation, it was shown that when RH is increased, cure polymerization is accelerated, and when film thickness is increased, polymerization is slowed down because cure proceeds from the outside to the inside of the bond (Mavrov 1983). Also, this study showed that the films these products produced had many defects (e.g. holes, tears, cracks, strains) when viewed using electron microscopy. In addition, the hydrophobic product played a protective role against the action of water and a salt solution, but it aged more quickly in sulfur dioxide under high RH. For all products, the stone pores were not continuously covered by a film, but formed bridges that improved the mechanical properties. One of the most important findings was the inability of the products to block hygroscopic salts in the stone pores, so treatment with these types of products was not recommended for salty or insufficiently desalted stones that might be further exposed to moisture.

RH plays an important role in curing methyltrimethoxysilane (Charola et al. 1984). Cure should take place between 30 to 50% RH; otherwise, it will be too rapid if RH is >50%, causing stress to develop; or too much resin will evaporate if RH is <30%. The consolidation of fine silica powder with this silane was better than the consolidation of coarse silica, but marble powder was not consolidated as there was no chemical affinity for it. Observed consolidation of deteriorated marble with this resin, therefore, must be due to a physical "locking in" rather than a chemical phenomenon. This was seen as an advantage as the substrate was less disrupted than with some forms of chemical interaction.

In a study of silicone coatings for limestone, the coating disappeared quickly when it was exposed outdoors and this was due to poor adhesion (Zádor et al. 1993). This finding was corroborated in another systematic test of water-repellent organosilicon-based agents; water repellency depended on the type of substrate (e.g. higher on brick than on limestone) and concentration of the agent, but did not depend on diluent, as water and organic solvents seemed to give the same result (De Witte et al. 1996).

Two-part methylsilicone resins with white spirits and a crosslinking agent have been used as a consolidant (Ahydrosil Z) and as a binding agent (Adhesil K1) for masonry materials in Poland (Zielecka and Rościszewski 1988). The products imparted water repellency and resistance to frost, aggressive salts and other chemical hazards (e.g. acid rain). Plus, the colour and surface masonry appearance was not changed. Furthermore, solvent penetration of the masonry remained the same, so further future impregnation was possible.

Another study suggested that organosilane consolidants penetrated better than epoxy resin

consolidants and gave better permeability to gases and vapours, but did not consolidate as effectively as the epoxy resins (Kotlik and Zelinger 1983). Both consolidants provided water repellency, but did not fix water-soluble salts in the stone.

Twenty-five consolidants, including what the study calls silicones and silanes, epoxy resins, acrylics, fluorides and a polyurethane, were tested for their consolidating abilities on sandstone and limestone (tensile and compressive strength) (Clifton 1984). Only some of the consolidants passed this test. These were further evaluated after an acid drip and for freeze-thaw stability and thermal expansion testing, for depth of penetration, appearance, depth of erosion and colour change. The silanes and silicones did not fare well. Generally, the silanes failed to consolidate the stone, but did penetrate well and had a good appearance, while the silicones consolidated very weakly. The best results were obtained with an epoxy resin. Interestingly, in another study, silane (methyltriethoxysilane) consolidation was shown to make porous stone resistant to disintegration by freeze-thaw cycles, while acrylic and epoxy resins gave little protection (Nishiura et al. 1984).

Evaluation of various consolidants for weak Maya limestone, including Conservare H [a mixture of ethyl silicate and methyltriethoxysilane (Stein et al. 2000)], showed that the Conservare H did not provide protection from erosion (Kumar and Ginell 1995). An epoxy resin and an acrylic/epoxy resin mixture were better. Further, it was concluded that, for sandstone, silanes work fairly well because "they penetrate deeply, improve mechanical strength and durability, and are relatively safe and easy to apply" (Ginell et al. 1995, p. 824). However, for deteriorated limestone, epoxy resins work better than acrylics or silanes.

In a study of various consolidants [silane (type not specified), ethyl silicate, epoxy resin, polyester, poly(vinyl chloride) (PVC), and polyacrylamide] on French limestone after natural aging (18 years), all treatments lost some of their surface consolidating effect, but not necessarily within the interior of the stone (Vallet et al. 2000). The silane and epoxy resin were the best in retaining superficial consolidation, while PVC, polyester and polyacrylamide were not so effective. The success of silane and epoxy resin was attributed to a resultant decrease in porosity and capillarity. The ethyl silicate was sensitive to the acidic environment and partially hydrolyzed.

Three organosilicon-based stone treatments [Paraloid B-72 with Dri-Film 104 (methylsilicone), Rhodorsil R893 (methylphenylsiloxane) and Strengthener H (ethylsilicate with alkylalkoxysilane)] were compared after 20 years in the field for water absorption, and the Fourier transform infrared (FTIR) spectra of the original versus the aged consolidants were also compared (Rossi-Manaresi et al. 1995). The Paraloid B-72/ Dri-Film 104 was still an effective and durable preservative for the sandstone tomb; however, there was considerable chemical alteration of the product after 20 years. The Rhodorsil R893 had less chemical alteration, kept its hydrophobicity and still had a good aggregating effect. The Strengthener H was less effective and less durable than the other two; its water repellency was lower and it was brittle.

When consolidation of blocks of quartz-sand/ marble-dust was performed in a sequenced treatment—ethyl silicate, followed by Paraloid B-72, followed by methyltrimethoxysilane—and compared to consolidation with a mixture of the three products, the differences in properties (depth of penetration, water absorption, bulk specific gravity, water vapour transmission and salt crystallization) were not that great (Matero and Oliver 1997). However, it was noted that consolidation with the mixture was somewhat more effective than the sequenced treatment. Addition of acrylic resin to the silane acts as a wetting agent, increases viscosity, reduces evaporation, and allows for deeper pore penetration (Shashoua 1995, pp. 322–324).

When five different polymer coatings [Bayer LF (siloxane/acrylic copolymer), Rhodorsil 224 (polymethylsiloxane), Wacker 290 (a mixture of silanes and siloxanes), Akeogard P (perfluorinated polyether), Florsam 39 (fluoro-organosilane copolymer)] were tested for contact angle, capillary water absorption, vapour permeability, and colour change on five different stones (marble from Drama and Thassos, porous stone from Kilkis, sandstone from Turkey and brick), the siloxane/acrylic copolymer displayed the best results overall, except on the brick (Tsakalof et al. 2007).

In 2005, a review of alkoxysilanes for stone consolidation appeared that gave a summary of the history and state of the art at that time (some of which is reviewed above in this section) (Wheeler 2005). The review stated that TEOS or ethyl silicate was proposed as early as 1861 for stone consolidation, while alkylalkoxysilanes started to be used in the late 1940s; however, by the 1970s, there were conflicting reports on effectiveness. Wheeler noted that alkoxysilanes proved to be a popular stone consolidant because their starting materials had low viscosity and their end cured products had thermal, oxidative and light stability. Their

performance is rather complex, but, simply put, the composition and structure of the stone can influence the catalysis, gelation, ripening and drying of this type of consolidant. It was further noted that stones that darken from these treatments were usually ones that contain certain minerals and those with fine pores or matrices. Generally (as mentioned above), strength benefits for silicate stones (sandstones) are larger than for carbonate stones (limestones). Laboratory experiments using freeze-thaw and wet-dry cycling showed failures in only a few cycles, indicating that frequent examinations and re-treating of stones in the field are necessary. Wheeler also stated that often stone scaling and flaking is not stabilized by consolidation with alkoxysilanes, but if the scales and flakes are small enough, then treatment can be successful. Likewise, if the intergranular spaces in the structure of the stone are small enough, consolidation can be successful. In general, success of an alkoxysilane consolidation depends on the original composition of the stone, its condition, and whether the causes for the deterioration can be removed.

SOLUBILITY AND/OR REMOVABILITY*

Silicones have very low surface energy and thus will wet nearly any surface they contact (Cagle 1973, p. 1–13). Thus, a silicone-contaminated surface will have a very low surface energy, which adhesives, paints and cleaning agents find hard to wet. As silicones are very chemically and heat stable, they are difficult to remove from surfaces and thus should never be used unless other "less-permanent possibilities have been explored" (Shashoua 1995, pp. 322–324; Cagle 1973, p. 1-13). There are now "paintable" silicones, which presumably afford a higher energy surface.

The un-crosslinked silicone rubber is soluble in hydrocarbons, chlorinated hydrocarbons and some ketones, esters and ethers, while the uncured resins are soluble in various organic liquids, such as toluene, xylene, petroleum spirit and *n*-butyl acetate (Roff and Scott 1971, p. 461).

The vulcanized rubber is swollen by non-polar liquids (e.g. hydrocarbons), chlorinated hydrocarbons, esters, methyl ethyl ketone, some silicone fluids, carbon disulfide and warm water (>70°C) (Roff and Scott 1971, p. 461). The cured resins are swollen by toluene and some other hydrocarbons, carbon tetrachloride, methyl chloride, acetone, methyl ethyl ketone, liquid ammonia, liquid sulfur dioxide and glacial acetic acid (Roff and Scott 1971, p. 461).

A very aggressive mixture of alkylbenzenesulfonic acid and dichloromethane, toluene and xylene was suggested to remove old silicone from glass objects, but this mixture was not recommended for various metal surfaces, some glasses or low-fired ceramics that contain carbonates, and porous acid-sensitive materials like paper or textiles (Bradshaw 1992; Erhardt 1983). Rinsing must follow removal treatment. Plexiglas, Lexan, Mylar and polyethylene showed no effects after contact, but could craze later due to exposure to these solvents.

A pre-polymerized silicone resin is more removable from stone than a silicone resin cured from a monomer because it is deposited on the stone with little bonding to it, while the cured monomer forms an irreversible network around the stone particles (Charola et al. 1984b).

* Disclaimer: The solvents listed in this section are ones that might dissolve the polymer or adhesive in question but are not necessarily recommended for conservation treatments. Some may be extremely toxic or may contain elements (e.g. chlorine) that may be harmful to certain materials in your object (artifact). Research the solvent before purchase or use to know its toxicity and chemical nature. It is strongly recommended to test all solvents before use to make sure that they do not adversely affect your object.

7 Choosing Adhesives and/or Consolidants for Conservation Treatments

Introduction

by Carole Dignard, Canadian Conservation Institute

While the previous chapter described the technical properties, characteristics, limitations and aging studies of the different adhesive classes, this chapter deals with adhesive selection criteria in conservation applications. The selection and use of adhesives in conservation is, of course, but one part of the decision-making process for the preserving and restoring of cultural property (**Figure 1**). The objectives of this chapter are to consider possible options and typical solutions, and to illustrate examples of the decision-making process when choosing an adhesive for specific treatments. As many similar adhesives are used across conservation subdisciplines, the properties and application methods that are common form the introduction to this chapter. The remainder of the chapter covers adhesive selection for each specific subdiscipline.

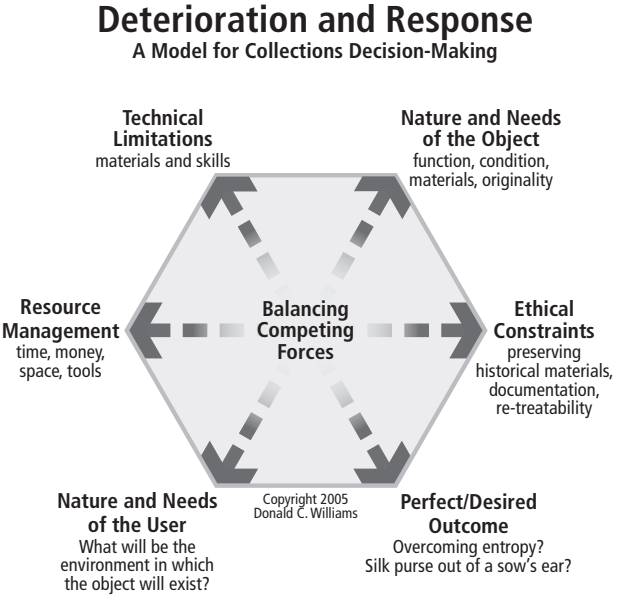

Figure 1. Decision-making processes for preserving and restoring cultural property (Williams 2011).

General principles

Conservation ethics provide basic axioms which a practitioner uses to assess the suitability of an adhesive:

- The adhesive and treatment must not adversely affect the object.
- The adhesive must be chemically stable and have good aging properties.
- The adhesive should remain removable and/or the treatment reversible (exceptions must be justified).
- Minimum intervention that addresses conservation needs is preferred over an invasive treatment (stitching or non-adhesive mounting are possible alternatives to an adhesive solution).
- Normally, the adhesive repair should be weaker than the material it is repairing so that under stress the repair will fail rather than the original material.
- The adhesive must be compatible with the rest of the treatment sequence and should not prevent future re-treatment of the object.

Beyond these general principles, adhesive selection for all conservation subdisciplines is always dependent on the nature and condition of the object, the type of treatment required and the object's end use (purpose, place). Pre-treatment testing is performed to assist in determining options.

Common considerations: Adhesive properties

In all conservation subdisciplines, defining adhesive treatments involves assessing adhesive properties relevant to the outcome. Some of these considerations, common to many subdisciplines, are described below.

STRENGTH
Assessment of the strength needed in the repair is essential; for instance, whether the bond is load-bearing or not, and what stresses are present or expected. Adhesive repairs have to overcome stresses exerted at the damage site during handling, display, transit and possible changes in relative humidity and temperature. If the adhesive fails in this respect, then the old damage will either recur or become increasingly visible.

FLEXIBILITY
Flexibility of an adhesive film is related to its glass transition temperature (T_g) (see Chapter 4): polymers with lower T_gs are more flexible and may be more desirable than brittle polymers having a higher T_g. However, those with a low T_g around or below room temperature remain tacky and can attract dirt or stick to adjacent materials. The flexibility in the repaired area will depend not only on the adhesive polymer's T_g, but on adhesive thickness, solvent retention and adhesive penetration into the substrate. If a backing material is used, its thickness and inherent flexibility will also have a large impact on overall flexibility of the repair.

VISCOSITY AND PENETRATION
It is often desirable to select a certain adhesive/consolidant viscosity for a treatment. For thermosetting adhesives, such as epoxy resins, the viscosity is usually set by the selection of the product itself, as dilution with solvents or addition of modifiers can change the curing behaviour and film properties. On the other hand, for thermoplastic adhesives, whether solvent or water-based, the viscosity is most often controlled by the choice of solvent(s) or dispersing agent(s), the adhesive's concentration (dilution), or use of additives (thickeners). Substrate porosity and extent of adhesive penetration are often important treatment considerations; in some instances, a surface (nap) bond is required and, in others, a deeper penetration within the substrate is desired. Factors affecting penetration, besides the nature of the adhesive, include the application method and—for wet (liquid) applications—choice of solvent, viscosity and drying time.

ADHESIVE SURFACE DEPOSITS AND GLOSS
There should be no adhesive left on the surface to impart gloss nor should the adhesive alter the original surface appearance (e.g. shift in refractive index). Surface characteristics should not be altered by any solution or solvent used to remove excess adhesive.

DRYING TIME
Quick drying times may be desirable, for example, if extended clamping or weighting time is not possible. Longer drying times may be most suitable for complex alignments so as to provide time for exact positioning. Adhesives that can be applied using different application methods and with different parameters (e.g. different solvents) are the most versatile in terms of range of working and drying times.

COMMON CONSIDERATIONS: APPLICATION METHODS
Thermosetting adhesives, such as epoxy resins, are usually only applied wet, for example, by brush or capillary action. However, in the case of thermoplastic adhesives, there are often a number of application methods possible which have a large impact on the treatment and, notably, on the adhesive working properties (drying time, strength of resulting bond and penetration). These adhesives may be applied wet (as a solution or dispersion) by brush, syringe, tube, spray or ultrasonic mister, or they may be applied dry as pre-formed films that can be reactivated by heat, solvents or solvent vapours. These methods have many advantages and disadvantages, which are discussed below.

WET APPLICATION OF ADHESIVE
Application of an adhesive wet (in a solution or dispersion), usually by brush, is the most common practice. The solvent or the dispersing liquid (usually water) generally has a large impact. It can affect bond strength, flexibility, working time, drying time, viscosity and wetting (Hansen 1995). Choosing between a polar (e.g. water, ethanol) or a non-polar solvent can significantly affect the outcome for both organic and inorganic substrates. Solvents must be tested to ensure that they do not cause any harmful or undesired effects. Good contact should be maintained during drying. Advantages and disadvantages are summarized in **Table 1**.

REACTIVATION BY HEAT, SOLVENTS OR SOLVENT VAPOURS
Reactivation techniques involve using a dry adhesive film either alone or on a backing material. The dry adhesive or adhesive-backing composite is positioned on the object to be treated, and then reactivated by heat, solvents or vapours to form the bond. While only a few conservation-quality, ready-made adhesive films, such as BEVA 371 Film, are commercially available, most adhesives can be cast into films [e.g. acrylics, poly(vinyl acetates), vinyl acetate/ethylene copolymers, ethylene/vinyl acetate copolymers and mixtures with other adhesives (e.g. cellulose ethers)]. Heat-set tissues are available commercially, but most have not yet been rigorously tested. Advantages to reactivation of pre-set adhesives include reduced or no wetness, less risk of penetration or staining, less risk of accidental smearing or smudging during

positioning, and—depending on the reactivation method—short clamping time (especially in the case of heat reactivation). Although not widely applied in conservation, remoistenable tissues, if stable, are an option for paper and photographs, allowing reduced amounts of moisture and therefore limiting problems such as dimensional swelling and shrinking.

Heat reactivation

Heat reactivation is a method that employs heat, using hand tools (e.g. hot spatulas) or vacuum hot tables (mainly in painting treatments), to soften the adhesive and form a bond upon cooling while pressure is applied to maintain contact. To achieve strong bonds, the adhesive must become sufficiently tacky to form a new bond. Activation temperatures typically range between 60–80°C, depending on the adhesive. Strict temperature control is required. Some materials are heat sensitive and should not be exposed to heat; prior testing is required. Advantages and disadvantages are summarized in **Table 2**.

Table 1. Wet application of adhesive (in solution or dispersion) – advantages/disadvantages

Advantages	Disadvantages
Water-based adhesives (solutions or dispersions)	
- water can soften stiff or distorted organic materials and improve contact - fast to apply, minimal preparation required - avoids heat, solvents - minimal health concerns - minimal tools and equipment required - range of products offering a variety of adhesive properties available - wide range of viscosities possible (viscosity can be tailored to help control penetration) - dispersion repairs remain largely unaffected if water is subsequently applied to treat adjacent areas	- water concerns for water-sensitive substrates, components (colourants, binders) or extraneous compounds (dirt, stains): e.g. dissolution, migration, stains - organic substrates may absorb water—may swell, shrink or become misaligned - unwanted penetration or staining possible - positioning and handling may be difficult while wet or tacky (risk of smudging) - wetting difficult if oils are present - clamping or weights required for good contact to produce the bond - dispersions: additives may affect stability (research needed)
Non-aqueous solutions	
- avoids water and heat - fast to apply, minimal preparation required - variety of adhesives and properties possible - variety of solvents possible, providing control of properties such as evaporation rate, viscosity, etc.	- unwanted penetration, stiffening, staining possible - possible softening of solvent-sensitive materials (certain paint media) - positioning and handling may be difficult while adhesive is wet - limited working time depending on solvent evaporation rate - clamping or weights required for good contact to produce the bond - fume extraction required—health concerns

Table 2. Heat reactivation – advantages/disadvantages

Advantages	Disadvantages
- easy positioning (avoids wet adhesive during cramped positioning; working time unlimited) - can work vertically (no dripping) - no clamping needed, hand-controlled pressure exerted from spatula - quick setting time (seconds) - penetration controlled by temperature and dwell time - minimal wicking into backing material possible (depends on adhesive) - adhesive film or backing prepared beforehand (easy for in-situ work) - avoids solvents, extraction requirements, health concerns - no water involved	- heat (potentially harmful for some materials) - pressure (potentially harmful for some materials or surfaces) - film preparation time consuming compared to applying the wet adhesive directly by brush - usually requires access to reverse of object (applying heat through support material preferable)

Solvent and solvent vapour reactivation

Solvent and solvent vapour reactivation involves using solvents or solvent vapours to soften the adhesive film or coating, allowing for a 'cold set' application. Evaporation usually needs to be controlled, often using a plastic sheeting cover. Solvent is usually delivered by brush or syringe, often in-situ after positioning the area under repair and an adhesive-backed reinforcing material. Spraying or misting the solvent is also possible. Another option for delivering solvent is to use an absorbent material (blotters, cotton wadding) in contact with the adhesive backing, itself positioned against the area under repair; this allows application of small amounts of solvent to reactivate the adhesive film. If it is important to prevent liquid solvent from reaching the object by wicking and capillary action, a barrier layer such as Gore-Tex is used. Reactivation using solvent vapours, on the other hand, can be achieved by carrying out the repair within a vapour chamber. Or, small localized repairs can be done using a glass jar containing solvent-dampened blotting paper wedged at the bottom of the jar and overturned over the area under repair to create a 'mini vapour chamber'. Vapours take more time to soften the adhesive film and form a bond than liquid solvent. All of the above methods require pressure (weights, clamps, vacuum, etc.) applied during reactivation and drying.

Table 3 provides general advantages and disadvantages regarding the use of solvent and solvent vapour reactivation. Factors affecting outcome include type and quantity of solvent used, controlling evaporation so that the adhesive may sufficiently soften, and applying adequate pressure to ensure good contact.

SPRAYING

Spraying is generally used to apply low-viscous adhesive solutions to form an even surface layer, for example, for surface consolidants (**Table 4**).

ULTRASONIC MISTING/NEBULIZING

Ultrasonic misting and nebulizing are useful techniques to apply low-viscous solutions to objects, and particularly for consolidation of powdery pigments (Michalski and Dignard 1997) (**Table 5**).

TABLE 3. SOLVENT AND SOLVENT VAPOUR REACTIVATION – ADVANTAGES/DISADVANTAGES

Advantages	Disadvantages
- avoids water and heat - no swelling or shrinking of organic substrates (using non-polar solvents) - fine control of solvent applied (quantity, location) - surface (or nap) bonds are possible - can work vertically (no dripping) - short clamping time possible depending on tack, solvent evaporation time, etc. - low pressures possible	- film preparation is time-consuming - fume extraction required—health concerns - excessive solvent may stain object or cause unwanted adhesive penetration - pressure required - sufficient time is required for adhesive to soften - weak bond if adhesive does not soften enough or if contact poor - blotters or barriers hide bond site while adhesive dries

TABLE 4. SPRAYING (PNEUMATIC) – ADVANTAGES/DISADVANTAGES

Advantages	Disadvantages
- even application over large surfaces is usually possible - no brush strokes - fast - can apply moderately viscous surface coating	- velocity needed to deliver spray on surface—may be too strong for delicate surfaces - solution evaporation occurs during delivery (travel from nozzle to surface) - large distance required between nozzle and object to reduce impact, thus difficult to apply in precise locations or to small surfaces (masking required) - adhesive solution may dry before achieving intended result (sufficient areas of contact, sufficiently deep penetration, etc.) - irregular size of adhesive droplets or irregularity of spray may not allow for even spread of adhesive

Table 5. Ultrasonic misting/nebulizing – advantages/disadvantages

Advantages	Disadvantages
- mist delivered with tiny force of impact—very delicate means of delivery - controlled application due to small hand-held nozzle (~1 cm from object and 1 cm diameter precise locations on object) - mist droplets 10–100 times smaller than spray droplets - less risk of smudging or dislodging surface elements (powdery pigments)	- needs non-viscous (and hence, often very dilute) solutions to mist; dispersions can be difficult to mist - generally several coats are required so treatment is slow - delivery area is small (~1 cm diameter) so treatment is slow - suitable for particulate consolidation only (not flakes)

Paper artifacts

by Sherry Guild, Canadian Conservation Institute, and Anne F. Maheux, Library and Archives Canada

The nature of the paper artifact can be defined by several variables: the composition, nature and method of manufacture of the paper support; and the materials, technique and manufacture of the image and/or text. Adhesive conservation treatments will be informed by these variables, along with artifact condition and treatment objectives.

A variety of naturally occurring aqueous-based adhesives have traditionally been available to the paper conservator, ranging from starches and gums, like wheat starch, *funori* and gum arabic, to proteins, like gelatin, parchment size and isinglass (Fuchs 2008). Synthetic adhesives and cellulose derivatives are appreciated for their unique properties, including solubility in water and organic solvents, resistance to biological degradation and pH stability (Feller and Wilt 1990; Down et al. 1996). These natural and synthetic adhesives have been used alone or in mixtures in conservation treatments.

Properties and condition of the object that influence adhesive choice

The physical characteristics of paper include strength, density, texture, colour and surface finish. These attributes affect adhesive choice and auxiliary materials used to perform a given conservation operation. The following describe some of these considerations:

Strength

Strength of the sheet is affected by type of fibre, degree of pulping and beating, presence of impurities, chemical processing and additives. Exceptionally strong papers, such as heavyweight art paper, Bristol board or card stock, may require an adhesive with good bonding characteristics. The adhesive should bond well to the paper substrate yet not be so strong as to cause damage, or so weak that it endangers the object.

Porosity

Porosity will affect the absorption of adhesives, which may cause tide lines and staining in porous sheets with the movement of soluble deterioration components. Less porous papers may resist adherence, resulting in a weaker bond of substrate to repair, lining or hinging materials.

Flexibility

Flexibility can be dictated by the quality of the papermaking fibre and by the presence of lignin and other impurities. Frequently handled flexible supports require flexible adhesives, like a combination of wheat starch paste (WSP) and methylcellulose (MC). Degraded, brittle papers may require a more sturdy support to minimize further damage, so adhesive flexibility is not an issue.

Colour and surface finish

Colour and surface finish have an effect on the substrate's sensitivity to water or solvents. Adhesive application on highly calendared and coated surfaces can result in tide lines and staining. The use of solvent-reactivated adhesive with dyed papers might be avoided for similar reasons.

Media and unique characteristics

Moisture, solvents, heat, weight and pressure—all or some of which may be used in adhesive treatments—may have an undesired effect on media. For example, the surface quality of media (e.g. glossy or matte) can be adversely affected by an inappropriate adhesive.

Flaking and friable media may pose challenges, whether in the repair or lining of the substrate, or in media consolidation. Unfixed pastel is easily disturbed by surface manipulation, and flaking gouache layers can be disrupted and damaged further by inappropriate treatment methods and materials. Aqueous adhesives can leave irreversible tide lines in matte media, like gouache and tempera.

Characteristics inherent in various media may also pose problems during conservation treatment. Oil-based media may impregnate and degrade paper, making adhesion of repairs, infills and linings problematic. Surface texture, embossings and platemarks can be altered with the application of heat or pressure during adhesive activation and drying.

Heavily embossed substrates may not withstand the pressure and weight necessary to produce an effective bond with aqueous adhesives. Pressure-sensitive or solvent-reactivated adhesives requiring little pressure may be more appropriate.

On some artifacts, the presence of iron gall ink has resulted in ink corrosion with subsequent degradation

to the paper substrate (Neevel and Mensch 1999). This degradation process is the result of two substances in the iron gall ink: iron(II) ions and acids. In the presence of moisture or high relative humidity, the iron(II) ions and acids may migrate from the ink into the surrounding paper, thereby increasing the area of ink corrosion (Eusman and Mensch 2000). Areas of iron gall ink corrosion may be in poor condition, with cracks and losses at the ink lines, and may require physical stabilization with local repairs or lining. In these instances, adhesive choice is critical, as an adhesive that is too wet may expand the area of corrosion.

Many inks and pigments used on historic paper documents contain copper, and studies have shown that copper ions are active in catalyzing oxidation of the paper (Daniels 2002). As a result, the paper substrate may be physically compromised and in need of repairs or mechanical stabilization, and the choice of adhesive and application method must be carefully considered.

OBJECT FORM AND SIZE

Treatment of works on paper that are large, unusually shaped or on heavyweight paper can be challenging, requiring the choice of an adhesive with good bond strength to achieve satisfactory results. For example, hinging large works of art on heavyweight paper may require the use of a non-conventional hinging material like a non-woven polyester fabric, coupled with an acrylic dispersion adhesive (e.g. Lascaux 360 HV). A thick almost dry WSP may be an appropriate adhesive choice for repairs or infills executed on thicker, more rigid supports, such as artist's board or illustration board. Three-dimensional paper objects, such as fans, screens or papier-mâché objects, may require adhesives with adequate bond strength and minimal moisture, such as a thick, almost dry WSP, to conform to the delicate manipulations required to treat such fragile objects.

OVERALL CONDITION OF THE OBJECT

Strength and overall physical condition of the object may influence adhesive choice and treatment application. For example, heat-set or solvent-reactivated adhesives may be appropriate for lining fragile paper or fragmented archival paper objects that cannot withstand the manipulation required for wet adhesives. Alternatively, wet adhesive like WSP can be modified with MC to give more slip and working time to minimize manipulation of the fragmented object.

ADHESIVE PROPERTIES CONSIDERED

Factors to consider when choosing an adhesive include suitability of the adhesive to the object and to the proposed treatment protocol and outcome. Attributes of the adhesive to consider include stability, removability, bond strength, penetration into the paper substrate, flexibility, pH, moisture content and surface gloss. Application method is an important consideration and is determined by the nature and condition of the substrate and media.

Adhesive application can be broadly divided into three systems: wet application (e.g. starch pastes), heat-activated [e.g. acrylic dispersions like the Lascaux products, poly(vinyl acetates), and proprietary solvent adhesive mixtures, like BEVA 371] and solvent-reactivated adhesives (Lascaux products).

ADHESIVES FREQUENTLY USED

Adhesives and consolidants typically used for paper conservation are given in **Table 1**.

TABLE 1. TYPICAL ADHESIVES USED FOR PAPER CONSERVATION

Type of treatment	Adhesive frequently used
hinging	starch paste, cellulose ethers, synthetic polymer dispersions
tear repair	starch paste, cellulose ethers
reinforcements	starch paste, cellulose ethers
infilling	starch paste, cellulose ethers
lining	starch paste, cellulose ethers, synthetic polymer dispersions
poultice	cellulose ethers
sizing	cellulose ethers, gelatin proteins
consolidant	cellulose ethers, gelatin proteins, acrylic resins
inpainting	cellulose ethers

CONSERVATION OPERATIONS REQUIRING THE USE OF ADHESIVES

HINGING

WSP has proven to be suitable for direct long-term application to most paper artifacts; it is one of the most popular adhesives in paper conservation and is frequently used for hinging paper objects. The cooked paste can be diluted to the appropriate thickness, determined by the object and treatment application, and will retain a proportional degree

of strength. This characteristic renders it possible to produce a number of adhesive strengths from one material and makes WSP a very versatile adhesive. Once dry, it produces a strong reliable bond that is reversible. When possible, WSP is the preferred adhesive for hinging works on paper. Thinned WSP can be used for lightweight papers where a stronger adhesive might cause distortion of the paper support. Thicker WSP can be used if hinging a heavyweight watercolour paper. To reduce moisture, the hinging material may be pasted out on a blotter to absorb excess moisture.

Remoistenable, or reactivated, adhesive systems are used for hinging, repair and lining of paper objects that are sensitive to moisture (e.g. tracing papers). The adhesive is applied in a smooth, even coat to polyester film (Mylar) and then the repair tissue is applied to the adhesive. Once dry, the tissue is peeled off the polyester film, and can be stored until needed. The adhesive is reactivated by a misting of water in the case of water-based adhesives, or solvent in the case of synthetic polymer dispersions (**Figure 1**). Starches, gelatins, isinglass, cellulose ethers and synthetic polymer dispersions are all suitable adhesives for reactivation films (Pataki 2009).

WSP can be used on many, but not all, paper artifacts. It is appropriate for most porous cellulose-based papers, but will not adhere to non-porous substrates such as coated papers, tracing paper or imitation vellum paper. Synthetic adhesives, like Lascaux 360 HV, Lascaux 498 HV or Lascaux 498 20-X, may be more suitable for these papers. These adhesives can be thinned with water to control strength and are insoluble in water once dry. They can be reactivated with heat or solvents to produce a strong, flexible bond (Maheux 1992).

When adhesive bond strength is not critical, MC can be used for hinging. For example, it can be used when applying hinges to small, thin Japanese paper objects. MC is available in several viscosities, with the mid-range viscosity (1500 mPa.s) the most appropriate for hinging. Because of its weaker bond, MC is sometimes used to adhere hinges to the bottom corners of works on paper that are float-mounted, to prevent contact with the glazing when framed.

TEAR REPAIR

WSP is frequently used for tear repair of paper artifacts. WSP can be used at the desired dilution (e.g. thin for lightweight paper, thick for heavyweight paper). If the paper object is highly reactive to moisture (e.g. tracing papers), remoistenable WSP repairs are an option. Similarly, synthetic polymer dispersions avoid the use of moisture and are reactivated with heat or solvents.

Treatment options for paper with iron gall ink that has extensive physical damage and loss of substrate may be limited. Local repairs should be executed with as little moisture as possible, as too much humidity may cause the iron(II) ions and acids in the ink to migrate to the surrounding paper (Jacobi et al. 2011). With caution, solvent-reactivated or remoistenable tissue can be used to repair these paper artifacts. Repair tissues coated with a suitable adhesive, such as Klucel G (2% in water), can be reactivated with solvent (Ubbink and Partridge 2003) and may lower the risk of ink migration. Research indicates that gelatin retards

Figure 1. Mounting of an oversize mixed-media drawing to a canvas support. Oversize works on paper often require robust hinging systems, such as strong Japanese papers, that are pulled through slits made in the mount and adhered to the verso for added strength. In the case of a long mixed-media drawing executed on heavyweight paper, a non-woven polyester fabric (Hollytex) and a water-based acrylic dispersion adhesive (Lascaux 360 HV) were chosen to mount the drawing to an auxiliary stretched canvas support. The Hollytex hinges were prepared with a coating of Lascaux 360 HV. Once dry, the adhesive film on the hinges was solvent-reactivated with acetone. The resulting hinges were strong and well-adhered to the canvas auxiliary support.

iron gall ink corrosion (Kolbe 2004) and it may be the most suitable adhesive for remoistenable tissue repairs. Gelatin, re-activated with a water and ethanol mixture (25:75), can be used to prepare remoistenable tissue for repairs (Titus et al. 2009; Pataki 2009). Gelatin-coated remoistenable tissues (Pataki 2009), modified gelatin and recombinant proteins (Meyer and Neumann 2009) were investigated and found to be potentially effective in protecting paper against corrosion caused by copper pigments. Further studies should lead to more definitive treatment recommendations for these artifacts.

REINFORCEMENTS

Using the same method found under *Tear Repair*, weakened areas in paper objects, such as creases, folds, skinned areas or paper weakened by deleterious media, can be reinforced with Japanese papers of appropriate weight with WSP. Moisture-sensitive objects that have a tendency to develop planar deformations can be reinforced with tissues prepared with remoistenable, heat-set or solvent-reactivated adhesives, if the media are not adversely affected.

INFILLING

Compensation for losses in paper artifacts are often executed with paper inserts, pulp fills or full linings to restore aesthetic integrity and reinstate physical stability. Adhesives are not only used to attach infill material, but are added to pulp fill slurry to improve the dispersion of fibres and increase bond strength (Paper Conservation Catalog 1990). Moreover, the surface of infill materials can be coated with diluted adhesive to control absorbance of compensation media and provide a particular surface finish or quality.

The criteria for choosing an adhesive to attach infills include flexibility, strength, non-yellowing aging characteristics and reversibility. Starch pastes are a frequent choice because they are strong, relatively colourless and have smooth consistency and good tack even when diluted. Cellulose ethers are also commonly used and possess the same qualities, but provide a weaker bond than starch pastes (Paper Conservation Catalog 1990).

LINING

An auxiliary support of paper or non-woven polyester fabrics can be solidly adhered to the verso of an object that requires added strength, flexibility, repair or compensation for losses. The type of lining chosen is informed by the physical characteristics of the object and its condition. Generally, for a primary lining, a more lightweight material than the object is chosen. Multiple lining layers may be applied, sometimes with different adhesives. For example, if a rigid support is needed for poster mounting, an interleaf lining of Japanese paper adhered with WSP may be followed by attachment to a thicker card with a solvent-reactivated synthetic adhesive, in order to avoid the use of moisture that may cause planar deformations in the lined poster.

The type and strength of lining adhesive also depends on the physical properties and condition of the substrate and media. The choice of adhesive diluent (water or solvent) must take into account the solubility of the media. Adhesive viscosity is another factor that must be considered. Thin paste is more suitable for delicate substrates; it will dry more quickly with minimal adhesion. Thicker pastes provide improved adhesion to heavy, porous substrates. Remoistenable lining systems are also suitable for fragile substrates and water-sensitive media, providing the lightest bond.

Starch paste is most frequently used as a lining adhesive. MC is another adhesive used for lining, both alone and in combination with starch paste. The addition of MC can moderate the strength, viscosity and flexibility of WSP, and can increase the slip and working time, allowing pieces and tears to be properly aligned. Rates of distortion and contraction are markedly less in MC linings, and such linings can be easily reversed with only a moderate application of moisture (Brückle 1996).

For oversized and heavy objects, stronger adhesives are recommended. Synthetic polymer dispersions, like the Lascaux adhesives, can be applied as a film to hinging materials that can be reactivated by heat or with solvents. Synthetic adhesives are used in the application of secondary linings, but may also be employed in direct contact with objects that are moisture sensitive or non-traditional in nature (Maheux 1992; Jirat-Wasiutynski 1980). These adhesives are not only strong and flexible, but are transparent and can be used with non-traditional substrates like polyester drafting films.

POULTICE

Cellulose ethers are frequently used as water-holding poultices in paper conservation to remove adhesives. For example, MC (4000 mPa.s) (2.0–5.0% in water) can be applied to soften old adhesive layers after a backing has been removed. It is especially useful as a poultice with enzymes (Schwarz et al. 1999).

SURFACE SIZING

During washing treatments, it is assumed that the extant size is removed, or partially removed,

and the application of a new surface size may be considered. The purpose of the size is to give the paper mechanical strength and flexibility and to protect the surface of the paper. Size for paper objects is usually protein- or cellulose-ether-based and can be applied by brush or spray. Low- to mid-range MCs (400–1500 mPa.s) (0.5% in water) are frequently used to resize paper. Gelatin is recommended as a sizing agent for works with iron gall ink, as it can block iron corrosion (Kolbe 2004).

CONSOLIDATION

Consolidation in paper conservation is the application of a binding medium to friable or flaking media to reattach it to the support. The working properties and characteristics of the consolidant must be evaluated in light of the media and the support, application technique and treatment objective. The consolidant choice depends on colour change, gloss, cohesion and adhesion of the media, and the physical and chemical compatibility of the consolidant with the object. The condition of the media determines the application method. Stable paint layers with large flakes may be consolidated with a brush, while friable media may best be consolidated with ultrasonic misting. Some techniques have limitations. For example, if powdery paint is to be consolidated with ultrasonic misting, consolidant choice is limited to those that are known to mist, usually at low concentrations (**Figure 2**) (Dignard et al. 1997). Typical polymers used for consolidation for media on paper are gelatin or MC (400 mPa.s), although there are times when *funori*, isinglass, parchment size, Klucel G or Paraloid B-72 may be options.

INPAINTING

Inpainting is the addition of suitable media to fills, repairs and other areas of loss in order to visually integrate the damaged area with the object. Inpainting is restricted to the area of loss and, although few inpainting materials are completely reversible, the application of an isolating layer on the original substrate may improve reversibility. Low- to mid-range MCs (400–1500 mPa.s) (0.5% in water) are frequently used as an isolating layer over paper losses prior to inpainting and facilitates its removal by minimizing penetration into the paper fibres.

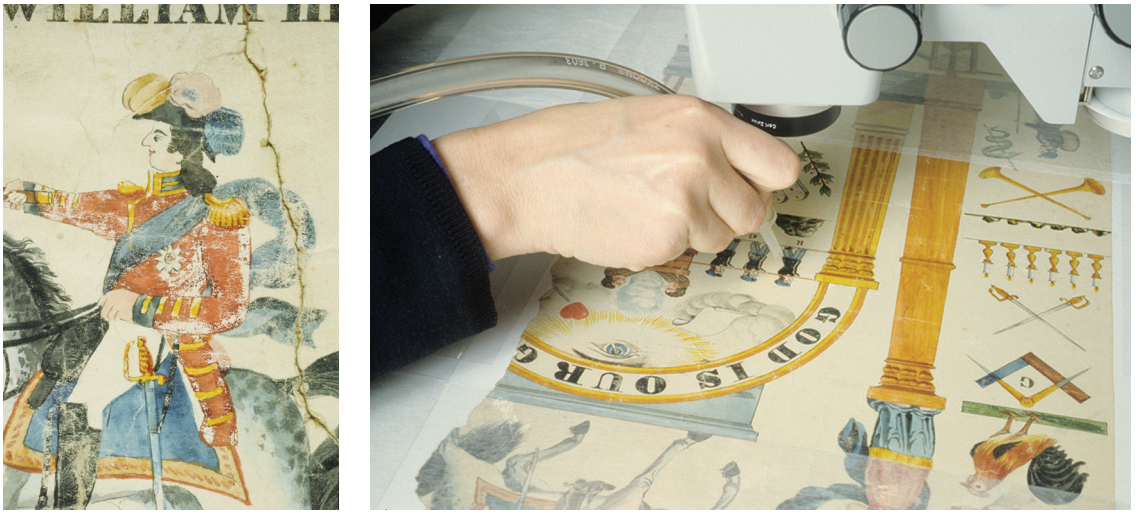

Figure 2. Consolidation of friable and flaking watercolour on an ink drawing. Areas with friable, powdery pigment were consolidated using Methocel A4C (0.25% in water) applied with an ultrasonic misting device. Methocel A4C was selected for this treatment because, among the consolidants known to mist, it has good stability and is close to neutral pH. To encourage penetration of the consolidant, small areas of the drawing were held under light vacuum pressure on the paper suction table while the mist was applied. A microscope was used to direct the mist to a specific site and to determine when the area was adequately saturated with consolidant. The ultrasonic misting device was used for this treatment because the small mist droplets did not smear the friable pigment and the dilute concentration of the consolidant, necessary for misting, allowed the treatment to be monitored and halted before discernable colour change or gloss occurred. Areas with cupped and flaking pigment were secured with two applications of Methocel A4C (0.5% in water) applied by brush (Dignard et al. 1997).

Photographic Materials

by Gregory Hill, Canadian Conservation Institute

Photographs, both negatives and positives, are typically multi-layered composite materials incorporating a substrate (support layer), a light-sensitive emulsion (image-forming layer) and a subbing layer that holds the emulsion to the substrate. The materials employed vary depending on the process. The image-forming substance of conventional black and white photographic materials is metallic silver that exploits the light-sensitive properties of certain silver salts (halides). Most colour emulsions also rely on silver halides to produce the image, though the final image is composed of synthetic dyes. Other light-sensitive, non-silver-based materials were used at various times throughout the history of photography to create images on paper, including platinum, iron, various gums and pigments, and all are routinely found in fine art and archival print collections.

Adhesives are traditionally employed in the remediation of physical damage, including the results of biological attack and in the mounting of photographs for display purposes. As photographic materials are largely paper-based, photograph conservators have traditionally borrowed heavily from the field of paper conservation for the repair, handling and display of photographic prints. However, there are unique properties that need to be addressed.

Properties and condition of the object that influence adhesive choice

Materials and structure of photographs

Negatives

Negatives can be differentiated by both the substrate and the binder (emulsion type). The first viable substrate employed was paper followed by glass and finally flexible plastic films. Paper negatives generally contained no binding material for the metallic silver image that sits directly on the paper surface. However, the paper was routinely oiled or waxed, rendering it more translucent.

By the late 1840s, glass replaced paper due to its complete transparency. A binder was required to adhere the image silver to the surface of the glass and, after considerable experimentation, collodion was adopted and used throughout the latter half of the 19th century. Natural resin varnishes were applied as a protective layer following processing. Collodion was also used as the binder for ambrotypes, tintypes and collodio-chloride papers. By the end of the 19th century, gelatin replaced collodion with pre-sensitized gelatin dry plates, commercially available by the 1880s (Valverde 2003).

With the invention of thin flexible plastic roll films in the late 1880s came the *naissance* of the motion picture and the amateur still photographic industries. Plastics used included cellulose nitrate, cellulose acetate and polyester. Albumen was occasionally used as the subbing layer for collodion plates, switching to gelatin with additives such as albumen and India rubber for gelatin dry glass. Early plastic films used dilute cellulose nitrate as the subbing layer, later changing to gelatin with polymer additives (Valverde 2003).

All of the commercially available negative materials produced from this point until the advent of electronic, magnetic and digital media were predicated on the silver gelatin emulsion on a substrate.

Photographic positive prints

Photographic positive prints can also be differentiated by their support and binding media, generally containing the same emulsions as negatives. In its most basic form, as exemplified by the salted paper print, the print consists of a metallic silver image embedded in the top layers of the paper fibre. There are no discrete layers. The same basic structure is found in most non-silver images including platinotypes, palladiotypes, cyanotypes, Van Dyke prints, argentotypes and chrystalotypes, to name a few.

Early on, paper was discovered to be a potential source of oxidizing agents for image silver. Consequently, the paper quality has always been extremely high.

Binders were introduced in the late 1840s, raising the image silver off of the paper surface and providing greater physical and chemical protection. The most widely used binder of the 19th century (1850–1890s) was egg albumen and these prints were called albumen prints. Separately, gelatin and collodion (collodio-chloride) were adopted as binders by

the late 1880s, both incorporating another innovation, the baryta layer—barium sulfate in gelatin. Layered between the paper base and the binder/emulsion, the baryta provided a smooth white background for the image. Collodio-chloride papers ceased production in the early 20th century, leaving gelatin as the binder for all commercially available materials to the present day. In the late 1960s, resin-coated (RC) papers were introduced, inserting a layer of polyethylene on either side of the paper substrate. This sealed the fibres against contact with processing chemicals, greatly reducing required wash times. The vast majority of conventional colour print material (non-digital) after the early 1970s is a chromogenic emulsion on RC paper. However, both RC and fibre-based (non-RC) papers continue to be produced.

Throughout the history of photography, hand colouring and other design elements have been applied to the surface of photographs and both negatives and prints have been retouched to enhance image quality. As a result, watercolours, inks, dry pigments, pastels, charcoal and graphite are commonly found. Also, mounting for both protection and decorative presentation has been done almost from the beginning.

CONDITION AND SENSITIVITIES OF PHOTOGRAPHS

When considering treatment and adhesive use, the nature and diversity of the materials, the sensitivity of the image-forming substance (whether silver, dyes or pigments), the condition of the object, and the intended use of the object following treatment must be factored into the decision-making process.

Oxidation of image silver

Image silver is very susceptible to oxidation, therefore adhesives, repair tissues, mount boards, etc. must be non-reactive and conform to (pass) the photographic activity test (PAT), which is the international standard test for evaluating photo storage and display products (ISO 18916:2007; Schenck and McCabe 1989).

Non-porous surfaces

Repairing glass plates for copying requires an adhesive that bonds to non-absorbent surfaces, provides very high strength, and has a matching refractive index to that of glass.

pH

Some photographic image-forming substances are pH sensitive. Cyanotypes, for example, change colour when placed in alkaline environments.

Translucency

Repairs to paper negatives must maintain translucency and be applied to the non-image surface. This generally precludes the use of repair tissues. Adhesives must be flexible with little shrinkage. Emulsion consolidation on negatives of all types must be colourless and completely transparent.

Strength and weight of paper

Many 20th-century photographic print papers are very dense and robust. Repairs to tears and losses require a strong adhesive bond like wheat starch paste (WSP) in combination with a strong repair tissue. Other papers can be very lightweight, requiring fine tissue and a weaker bonding adhesive such as a dilute aged WSP or methylcellulose (MC). Brittle acidic boards are prone to edge damage and breakage. Aligning edges can be problematic, requiring humidification in combination with strong adhesive and repair tissue.

Substrate to emulsion bond

Emulsions can peel from glass and plastic substrates, which is the result of fluctuating humidity levels compromising the bond between the subbing layer and the substrate. Deteriorating unstable plastics, including cellulose nitrate and cellulose acetate, can result in lifting, channeling, flaking emulsions and often complete destruction. Consolidants must be colourless and completely transparent.

Fracturing of emulsion layer

All emulsion layers are susceptible to cracking, creasing, lifting and flaking, which are the results of mishandling, desiccation of gelatin, and differential expansion and contraction rates of the separate layers of the print. Consolidants must be colourless, strong and compatible with the binder.

Solubility of emulsion layers

Colour photographic dyes can be sensitive to solvents, which is important to consider when choosing solvent-based adhesives or solubilizing residual mounting adhesives. Applied media, retouch and design elements are all potentially moisture-sensitive, limiting aqueous adhesive use. Other media can become friable, requiring consolidation. Collodion and most lacquer protective coats for emulsions are soluble in standard solvents used in conservation.

Planar deformations

Curling of unmounted prints is common. Lining adhesives must be strong yet flexible and any shrinkage must work to counteract the natural tendency of the print to curl emulsion inward. Aqueous treatments may exacerbate this problem, in which case non-aqueous adhesives could be considered.

RC papers distort locally (e.g. pucker, lift) when hinges are applied to the verso using aqueous adhesives. In this case, non-aqueous heat-set or solvent-activated adhesives are required.

Flexibility
Print and negative materials without rigid auxiliary supports are flexible materials requiring flexible adhesives. Repairs to cracks in the emulsion should maintain a high degree of flexibility to ensure that gap-fills, for example, do not crack during handling.

Surface characteristics – refractive index
The surface characteristics of photographic paper can be extremely sensitive to touch and to water and solvent contact. For example, the refractive index can change as the result of inappropriate aqueous and solvent treatment accompanied by physical manipulation. The cracking of albumen binders is part of the natural aging process and can be greatly enhanced by aqueous treatments (Messier and Vitale 1993, 1994). Maintaining the homogeneity of the emulsion surface is critical.

Gelatin deterioration
Photographic gelatin emulsions are generally strong, durable structures that can be compromised by elevated temperatures, high humidity, light damage and mould/mildew attack. The results can be increased solubility of the gelatin, necessitating a judicious use of aqueous-based adhesives. Even attempting to consolidate deteriorated emulsion can result in enhanced flaking.

Object form and size
Many very large oversized prints on RC papers or polyester bases are meant to be mounted perfectly flat or with minimal undulations to the surface. Most often the surfaces are smooth, glossy and easily damaged. Mounting generally requires a fully mechanized non-aqueous system.

Porosity
Thin or degraded papers can be highly porous, particularly if previous mounts were highly acidic. Migration of degradation products within the paper during wet treatment can result in staining and tide lines within the image (often impossible to remove), limiting the use of aqueous adhesives.

Overall condition of the object
Strength and overall physical condition of the object may influence the choice of adhesive and treatment application. For example, heat-set or solvent-activated adhesives may be appropriate for lining fragile or fragmented photographs that cannot withstand the manipulation required for wet adhesive applications. Alternatively, wet adhesive such as WSP can be modified with MC to give more working time and slip to facilitate manipulation of the fragmented object.

Optical brighteners
Optical brightening agents (e.g. stilbene compounds) began appearing in photographic fibre-based print material in 1950. They are subject to degradation over time and to alteration from aqueous and solvent treatments.

Adhesive properties considered

Factors to consider when choosing an adhesive for paper-based photographs are the same as for paper (see *Paper Artifacts* section).

Adhesives frequently used

Adhesives and consolidants typically used for photographic materials are given in **Table 1**.

Table 1. Typical adhesives used for photograph conservation
[Only those adhesives that pass the PAT are recommended for photographic materials (ISO 18916:2007). Be aware that some acrylic dispersions do not pass the PAT (see *Hinging* below).]

Type of treatment	Adhesives frequently used
hinging	WSP, cellulose ethers, acrylic dispersions
mount repair	WSP, acrylic dispersions
glass plate repair	epoxy resins, acrylic resins (Paraloid B-72)
emulsion repair	gelatin, cellulose ethers
paper repair	WSP, cellulose ethers, acrylic dispersions
lining	WSP, cellulose ethers, acrylic dispersions, ethylene/vinyl acetate copolymer
heat-set tissue	acrylic dispersions
consolidation	gelatin, cellulose ethers
surface coatings	acrylic resins
poultice	WSP, cellulose ethers

Mounting of photographs

Hinging
Hinging of fibre-based photographic prints into mats is traditionally done using WSP or MC with Japanese tissue hinges. Both adhesives can provide strong attachment, are easily reversible and are the

materials of choice for T and V hinges. However, due to potential planar distortions, aqueous adhesives cannot be used on RC papers (**Figure 1**). Polymer-coated ink jet papers can also prove problematic in this regard. Until recently, the preferred non-aqueous adhesives were synthetic films made with acrylic dispersions (e.g. Rhoplex N-580, Lascaux 498 HV, Lascaux 360 HV, Plextol B500), which can be heat-set or solvent-activated and do not distort the paper (Watkins 2004). Lascaux 360 HV applied to Japanese tissue was also used as a pressure-sensitive hinge when a very weak bond is required, and is easily removed mechanically or with gentle heat. Used individually or in combination, Lascaux 498 HV and Lascaux 360 HV provide a range of properties depending on the needs of the object. These have proven particularly useful in the mounting of large format prints on RC paper or pigmented polyester supports where planar distortions would not be tolerated. Applied to either Japanese tissue or non-woven polyester webbing, such as Hollytex, the strips can be applied as an edge lining and can be solvent-activated in cases when heating could cause damage, followed by being wrapped around a rigid support mount (Chen et al. 2007; Morrison 2007; Valverde 2007). Unfortunately, these materials have not passed recent PATs conducted at the Canadian Conservation Institute (CCI), and suitable replacement adhesives have not yet been identified (Down et al. 2013). Also, Lascaux 360 HV has been recently discontinued and, according to the Lascaux company, replaced with Lascaux 303 HV, a 2-ethylhexyl acrylate/ethyl acrylate copolymer, a very different formulation. The Lascaux 303 HV passed the PAT and has a neutral initial pH (7.77 ± 0.06), but, as there is no information on its long-term stability nor working properties, its use cannot be fully endorsed by CCI at this time (Down et al. 2013; Down 2013). BEVA 371 Film (original formula) passed the PAT and might be a viable option for some hinging requirements; however, the new BEVA 371b (solution) did not pass the PAT (Down et al. 2013).

Wet mounting

The mounting of photographic materials onto solid supports has been practiced from a very early stage for protection from physical damage, ease of presentation and aesthetic considerations. Albumen prints, traditionally produced on very thin paper with a propensity to curl, are often found wet mounted onto heavy paper board supports in all formats: *carte-de-visites*, cabinet cards, *minettes*, etc. Early gelatin and collodio-chloride prints were mounted in a similar fashion. WSP and purified gelatins were commonly used along with natural resins and gums, dextrine and albumen. WSP and MC are both routinely used today and occasionally combined to alter the working properties.

Dry mounting

By the beginning of the 20th century, thin paper sheets impregnated with thermoplastic natural resins called dry mount tissues were used to adhere prints to mount boards (Watkins 1993). Overheating during the mounting procedure weakened the emulsion layer and the natural resins discoloured over time. Mount boards tended to be highly acidic wood pulp laminates—many becoming very brittle over time and subject to damage

Figure 1. Example of aqueous adhesive applied to resin-coated paper causing planar distortion. Cibachromes and pigmented polyester bases can distort slightly upon drying after aqueous adhesives are applied, causing puckering on the recto. Here, the hinges are visible from the recto. Non-aqueous heat-set or solvent-activated adhesives applied to a Japanese paper hinge are recommended.

and loss. Later, dry mount tissues and pressure-sensitive mounting systems were developed using a range of synthetic adhesives, some proving unstable and deleterious to the photographic print (Reinhold 1991). However, many contemporary photographers working in very large formats demand absolute planar integrity and resort to dry mounting onto laminated aluminum sheets (e.g. Dibond) and acrylic sheets [poly(methyl methacrylate) (PMMA), e.g. Plexiglas]. Though mount adhesive formulations change frequently and little analytical information is available about them, many products exhibit good stability.

Face mounting

Face mounting of prints onto PMMA or Plexiglas sheets has become popular in the fine art market for large format prints, whether they are chromogenic or digital. The reasons are both aesthetic and practical. Protection is provided against fingerprints and mechanical damage, and ultraviolet absorbers in the Plexiglas limit photochemical degradation. Aesthetically, the colours tend to be more saturated and clear. The Diasec process uses a silicone rubber-based adhesive layer between the print and the PMMA, the chemistry of which is proprietary, though there are many non-licensed practitioners and little research has been done on its long-term stability (Penichon and Juergens 2001).

Glass plate repair

Glass plate repair requires a strong adhesive that bonds well to non-porous substrates, with a refractive index matching glass. Epo-Tek 301-2 and Hxtal NYL-1 are two recommended epoxy resin adhesives (they have good yellowing resistance), the former being more viscous and having better working properties (Down 1986; Hendriks et al. 1991). Alignment is difficult as glass plates are rarely perfectly flat. More recently, an improved procedure was developed whereby the plate is assembled vertically and the pieces are held in place with Vigor sticky wax (Koob 2000; Whitman and Wiegandt 2007) (**Figure 2**). This process is aided by the use of a light beam directed along the surface of the plate, illuminating the fracture interfaces. Paraloid B-72 (20% in toluene) is wicked into the cracks from the glass side of the plate. Once set, it is left to harden and is then supported overall with a layer of clear silicone (Clear P-4 Silicone from Silicones Inc.) on the verso. Unlike epoxy resins, the lower viscosity of the Paraloid B-72 solution allows it to wick easily into the fractures and, like epoxy resins, its refractive index matches that of glass. Paraloid B-72 passed the PAT carried out at CCI (Down et al. 2013).

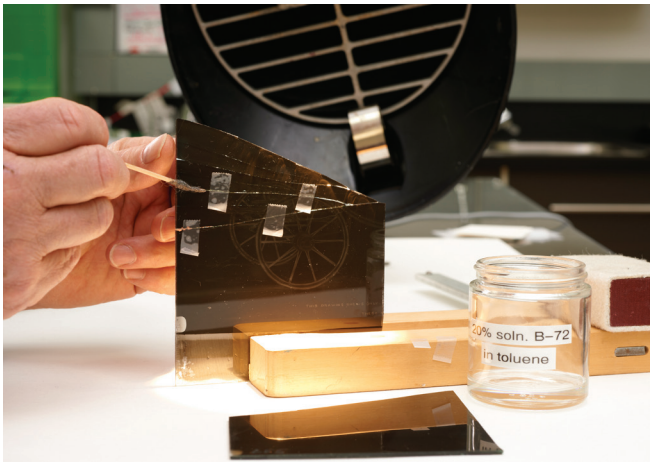

Figure 2. Example of a glass plate being repaired by vertical assembly. The base piece of a broken glass plate is clamped and stood vertically. Pieces are then assembled rising vertically and gently taped in place on the glass side. The adhesive, Paraloid B-72 (20% in toluene), is applied to the break edges, using a steel wool swab.

Repair

Adhesive choice for the repair of photographic prints varies according to the paper, emulsion type and media solubility. The preferred procedure is to apply a small amount of adhesive, most often WSP, to overlapping paper fibres along a break edge, bringing the two edges together, placing it between non-woven polyester sheeting and blotters, and then placing the entire assembly under pressure until dry. Most often, repair tissues are required and, when repairing heavyweight fibre-based silver gelatin prints, cracks to the emulsion must also be closed up with applications of warm gelatin to the crack edges, followed by light burnishing and drying under pressure (Hendriks et al. 1991). Cracks in rolled panoramas have a tendency to run vertically, cutting across the panorama, opening up on the recto. They tenaciously cup even following proper flattening procedures. Often, bringing these cracks/breaks into plane requires several layers of repair tissue applied to the verso of the crack.

Albumen prints react significantly to aqueous adhesives, necessitating a very thin dry starch paste or a very thin dry coat of MC on thin Japanese tissue. These must then be placed under weight for extended periods of time to ensure against planar distortions. Extremely fragile and reactive papers can also be repaired with remoistenable tissues using Klucel G activated with alcohol.

WSP, Japanese tissue and Western papers also provide the strongest bond for the repair and consolidation of deteriorated mount boards. Paste can be introduced between the layers of the board and dried under pressure to minimize planar deformations. Missing corners can be built up using layers of Western paper—with water-cut edges meshing the paper and board fibres—and contoured to the break edge. Moisture content of paste must be controlled to avoid staining from water wicking into the board.

LINING

Physical damage and paper degradation can necessitate overall lining of a print, preceded by tear repair. Generally, linings are done with an appropriate weight Japanese tissue or high quality Western paper and WSP. One must always keep in mind the potential for the adhesive to cause staining in the paper and image layer. Dacron or modified Dacron linings and drop linings can be applied directly to the verso of the humidified photograph (Wagner 1991).

The lining of damaged albumen prints requires special consideration due to the damaging effect of moisture on albumen binders (Messier and Vitale 1994). Humidification is required to swell both the paper and binder prior to lining (Vischi and Hill 2008). The print is then immediately placed face down on polyester, repaired, lined and moisture reduced on a suction table. While still damp, the lined print is then placed in a "hard soft sandwich" blotter stack to reduce curl (Homburger and Korbel 1999; Vischi and Hill 2008). Remoistenable lining tissues with MC and WSP mixtures can be very effective in lining and repairing albumen papers and objects with aqueous sensitivities as they require little moisture (Brückle 1997; Wagner 1997).

The repair of photographs on non-traditional substrates such as leather and textiles can necessitate the use of lining materials not traditionally used in paper or photograph conservation. For instance, a large 1913 photographic panorama on cloth was lined onto new fabric using BEVA 371 Film (Baker and Hill 1987).

CONSOLIDATION OF LIFTING AND FLAKING EMULSIONS

When the bond between the emulsion and a glass substrate is compromised, the emulsion is extremely vulnerable to mechanical damage and requires reattachment. Gelatin expands significantly in contact with moisture, making realignment of pieces difficult or impossible with aqueous adhesives. Klucel G (2% in ethanol, applied by brush) wicks under loose fragments, providing a relatively weak bond. The gelatin is lightly humidified with the ultrasonic mister prior to adhesion (**Figures 3a** and **b**). Warm gelatin (2% in water) can be used for very small areas of consolidation along the edges of both flaking negatives and prints. Paraloid B-72 (5% w/v in xylene) with humidification of the gelatin has been tried, but full testing has not been carried out (Albright et al. 2003).

Edges of tears can be brought together and adhered using warm gelatin. Generally, it is advisable to first repair tears from the verso using Japanese tissue and WSP. Warm gelatin (2%) is then applied to the edges of the emulsion, lightly burnished and allowed to dry under pressure. Gelatin applied to the verso of a crack can also help to pull the cupping emulsion into plane, depending on the thickness of the paper (Hofmann 1991; Long 1989).

Degraded and friable gelatin emulsions can be consolidated using very controlled applications

Figures 3a and b. Flaking gelatin being re-adhered to a glass substrate using Klucel G (1.5–2% solution in ethanol). The flakes are lifted slightly; the adhesive applied; and then the gelatin is humidified using the ultrasonic mister, prior to being laid down and manipulated into place. Light pressure should be applied until the adhesive is set.

of warm gelatin (2%) applied locally with a fine brush or ultrasonic mister. Careful testing must be done first as overall reticulation of the gelatin is possible. The resulting bond is generally not strong and any flexing can easily dislodge the consolidated surfaces.

MC is also an option for these applications as it is a more flexible material. Friable paint surfaces found on crayon enlargements (large format over-painted photographs) can be consolidated using ultrasonic misting techniques (see *Paper Artifacts* section).

Books

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A book is a collection of printed, manuscript (handwritten) or blank pages sewn or glued together along one side and bound between rigid boards or flexible covers. This section will focus primarily on the binding of a book rather than the actual pages. In book conservation, a multitude of variables can be encountered, including historic and modern binding structures, cover attachments, multi-media inclusions and the remains of previous conservation interventions. While traditional materials in book manufacture include parchment, paper, textiles, wood, millboard, leather, inks and pigments, materials such as plastics and metals are also currently used. Traditional adhesives for bookbinding and book repair/conservation are starch-based and animal glues. Since the 1940s, bookbinding practice has included the use of synthetic adhesives such as poly(vinyl acetates) (PVACs), acrylic dispersions and hot-melts (ethylene/vinyl acetate copolymers, styrene block copolymers and component reactive polyurethanes).

As the book ages, its materials, adhesives and structures deteriorate chemically and physically. The practice of conservation strives to reinforce or replace the working parts in order for a book to serve its function of allowing visual access to its contents. It is the continuing usability of books that gives them value. Depending on the frequency of handling, the storage environment and properties of the bound material, it can be expected that books of enduring value will require conservation intervention periodically over their lifetime. Minimal conservation intervention or non-adhesive treatments should be considered when possible to prevent loss of binding components and/or information contained within the book.

Properties of the object and factors that influence adhesive choice

Ethical considerations

The choice of adhesives and repair materials should respect the binding style, historic period, manufacturing techniques, materials and tradition. Adhesives and repair materials should be chosen with respect to any relevant religious or cultural significance of the object regarding the use of animal-based products. The cause of the current deterioration and the objectives of the conservation treatment in light of anticipated future use and preservation should be carefully considered.

Previous adhesives or coatings

Successive layers of adhesives and repair materials cause stiffness or lack of flexibility in the book that can lead to structural breakdown, thus necessitating the removal of these added materials. The removal of previous adhesives can be problematic because of their irreversibility, limited reversibility, lack of access to the area, and porous or fragile materials in contact with the adhesive. New adhesive bonds will be affected if the previous adhesives are not completely removed (e.g. starch adhesives do not bond well to synthetic adhesives). Adhesive bonds will be unreliable if surface coatings such as wax or leather dressings are present.

Strength

The original materials and structural components of book bindings should be assessed for condition and strength. The movable structure of books requires a moderate adhesive bond and a flexible repair material. Components that are completely detached may require stronger bonds than components that are fragile and at risk of detachment.

Porosity and penetration

The porosity of materials affects the absorption and penetration of adhesives. Porous or degraded leather can stain, darken, shrink and harden in contact with water-based adhesives (**Figure 1**). A fragile paper spine may stiffen or stain with a thick application of adhesive. A porous fibre-board will draw in adhesive rapidly, reducing the adhesive film and bond strength. Penetration can be controlled by the application method, by adjusting viscosity, by layering adhesive applications, and/or by limiting the amount of adhesive.

Flexibility

In general, a high degree of flexibility is desirable for hinge repair, lining and rebacking. Mobility of the structural components depends upon the flexibility of original materials and the choice of repair materials and adhesive, which must all work in conjunction.

Figure 1. Darkening of leather due to water-based adhesive.

Solubility

The components of book bindings contain compounds that can solubilize or migrate when in contact with water- or solvent-based adhesives. Sizing agents, dyes and pigments in textile and paper coverings may bleed or cause staining. Migration of tannins in degraded vegetable-tanned leathers can cause darkening. Migration of acids in paper-based materials can occur. Old adhesives can cause staining and stiffening of materials.

Access to bond site

When there is limited access to the bond site, adaptation of adhesives and/or application method is the key to success. For example, access to a deteriorated book spine can be very limited if the cover material is intact. An adhesive with a longer working time or application of adhesive using a syringe may be useful. To adhere new hinge material under fragile aged leather, the adhesive can be off-set from a polyester strip to the underside of the fragile leather in a two-step process: one thin adhesive layer is employed to seal the leather or board, and then a second adhesive layer attaches the hinge.

Clamping

To ensure bonding of adhesive repairs, customized clamping, weighting, pressing or tying-up will be required during drying. Too much weight or pressure during drying of aqueous adhesive repairs can result in staining or darkening of the leather, paper and textile covering material. Underlying imperfections or unevenness will be visible through covering material that has been pressed too long or under too much pressure. Adhesives can ooze out or be forced into unwanted areas when under too much pressure, resulting in weakening of the bond, hardened adhesive lumps and unwanted adhesion or staining of materials. Conversely, not enough weight or pressure can lead to poor adhesion.

Adhesive properties considered

In general, a good adhesive for book conservation is versatile in application method, easy to prepare, not subject to insect or biological attack or to discolouration, has a reasonable shelf life, remains readily soluble or removable, is acid-free, and retains sufficient bond performance and function (Metzger et al. 2003). Typical adhesives used for book conservation are given in **Table 1**.

Table 1. Typical adhesives used for book conservation

Type of treatment	Adhesive frequently used
lining	collagen (gelatin, animal glue), starch (wheat, rice, corn), cellulose ether [methylcellulose, hydroxypropylcellulose (Klucel G)], vinyl acetate/ethylene copolymer dispersion (VAE) (Jade 403, Elvace 45675), acrylic dispersion (Lascaux 360 HV, Lascaux 498 HV), mixture of water-based adhesives (starch/VAE; methylcellulose/VAE)
rebacking	starch (wheat, rice, corn), VAE dispersion (Jade 403, Elvace 45675), acrylic dispersion (Lascaux 360 HV, Lascaux 498 HV), mixture of water-based adhesives (starch/VAE; methylcellulose/VAE)
hinging	collagen (gelatin, animal glue), starch (wheat, rice, corn), VAE dispersion (Jade 403, Elvace 45675), acrylic dispersion (Lascaux 360 HV, Lascaux 498 HV), mixture of water-based adhesives (starch/VAE; methylcellulose/VAE)
poultice	cellulose ether [methylcellulose, hydroxypropylcellulose (Klucel G)]
consolidant	collagen (gelatin, animal glue), cellulose ether [methylcellulose, hydroxypropylcellulose (Klucel G)], acrylic dispersion (Lascaux 360 HV, Lascaux 498 HV), acrylic resin (Paraloid B-72, Plextol series)

Choosing adhesives for conservation treatments

Lining

Adhesives and materials used as spine linings are selected based on a number of factors such as the characteristics of the binding, the anticipated use of the object and the experiences or traditions of the binder or conservator. A certain amount of vertical rigidity is required to support the spine structure and prevent deformation. Flexibility across the width of the spine is necessary to ensure mobility of the structure. Combinations of adhesives, materials and techniques are employed to create this essential balance between rigidity and flexibility.

Primary spine lining

Primary spine lining includes an adhesive and may or may not incorporate a lining material that is applied directly to the spine of the text block. The bond strength is not required to be exceedingly strong since the main stresses on the moving spine are across its width and do not bear on the adhesive or lining at right angles to the stresses (Conroy 1987). As this may be the only lining on a spine, it must hold the spine in the desired functional shape. Starch-based adhesives are a traditional adhesive in bookbinding and conservation due to their chemical stability, water-based nature and reliable bond, which can be adjusted to the required strength. They generally have high moisture content and are slow to dry. Starch pastes can be applied in a diluted state as a 'paste wash', with or without a lining paper, to seal the folded paper signatures making up the spine, and can act as a barrier to subsequent adhesive layers (**Figure 2**). Lining between the sewing supports (transverse lining) helps to prevent build-up of adhesives and to prevent linings that result in the loss of flexibility and the potential damage of supports and thread through excess humidity.

Collagen-based adhesives (e.g. gelatin or animal glues) have also been used traditionally for primary and secondary linings (see below for more on secondary linings). These characteristically strong adhesives are chemically stable and reversible in water; however, they tend to be somewhat brittle when dry. Additives such as sorbitol and glycerin have been used to improve flexibility. Collagen adhesives have high tack, are quick to set and contain less moisture than starch pastes, resulting in less warping of materials. They must be used in a heated state, and the heat should be customized to the materials being bonded. Traditional heavy applications of gelatin as a lining adhesive on parchment text blocks is thought to be inappropriate to the material and the cause of inflexibility and breakdown of spine structures. It is however a strong, reversible adhesive to which buffering agents and softeners can be added. Parchment text blocks may benefit from a non-adhesive primary lining (Spitzmueller 2004).

Secondary spine linings

Secondary spine linings include adhesives and lining materials that provide structural support or physical

Figure 2. Primary spine lining. Diluted wheat starch paste and Kozo fibre tissue.

means to reattach boards or covers, such as a hollow tube, cloth hinges or parchment tabs. The adhesives used in secondary linings generally provide the bond between new materials, such as the primary lining tissue and a cloth lining.

Starch and collagen adhesives traditionally provide reliable results (as described in the previous section). Starch pastes are often chosen for use on older, rare and fragile bindings due to their reversibility. Vinyl acetate/ethylene copolymer (VAE) dispersions are selected if a stronger bond is required, when quicker drying time is desired to inhibit staining or swelling, or if reversibility is not a concern. VAE dispersions used with cotton cambric lining or pre-washed aero linen as a support for new sewing adhered over primary lining offer better anchoring for the liner, greater structural support, and flexibility for the spine (McPherson 2000). If iron-containing staples are present, the use of water-based adhesives may cause the iron to rust and damage the surrounding cellulose materials. In this case, a VAE dispersion may supply less humidity and a quicker drying time than starch paste, inhibiting further degradation of the staples (Doyle 2004). In preparation for a cloth hinge, it is important to work the VAE well into the cloth, as the strength of the board attachment depends in part upon a strong bond between the cloth and the text block spine. Aero linen and cotton liners can be dampened slightly during application to achieve greater adhesion to the spine (Brock 2001). A reversible VAE dispersion (e.g. Jade R) can be applied directly to the paste-washed spine in order to adhere aero linen to new bindings (Funazaki 2007).

Various aqueous adhesives can be custom mixed to manipulate drying time, penetration, tack, slip, clamping time, strength or flexibility. Aging of mixed adhesives containing synthetic glues may contribute to irreversibility (Fuchs 2008). According to a survey, 13% of book conservators used mixtures of PVAC/starch paste and PVAC/methylcellulose (MC) (St. John 2000). VAE dispersion moderated with MC (25%) applied sparingly can control concavity of the spine without inhibiting the opening of the text block (McPherson and McPherson 2000). A secondary parchment spine liner adhered with a mixture of VAE dispersion and starch paste to speed drying time will provide a strong bond to the primary lining (Kellar 1988).

Rebacking

Rebacking is the removal and replacement of spine covering materials and adhesives that are usually in a state of deterioration. It can also be done to preserve the mobility of the spine and/or provide cover attachment. The goal is to provide a durable, flexible bond between old and new materials while controlling humidity and drying time. Reversibility versus durability of the repair is an important factor to consider; the requirement for a special collections item will be different than that of a non-rare circulation volume. Depending upon the desired results and materials involved, variations can be achieved by adjusting adhesive recipes and concentrations, or by combining various aqueous adhesives. Controlled application of viscous starch paste applied to the underside of aged leather acts as a barrier to prevent moisture wicking through to the surface as subsequent starch paste layers are applied (**Figure 3**). To reduce

Figure 3. Controlled application of adhesive under aged leather.

potential discolouration, shrinkage and stiffening of aged leather, VAE dispersion is preferable to starch paste. Lascaux 360 HV and Lascaux 498 HV, either alone or in mixture, can be used to adhere aged leather to a new substrate because their solubility in certain solvents may improve removability, as compared to VAE dispersions. In a treatment by Frederick and Hellman (2001), Lascaux 360 HV used to adhere a Japanese tissue lining to an aged leather spine remained very flexible and did not inhibit the movement of the spine.

HINGING

The traditional board attachments that enable hinging of the book covers are the sewing supports, covering materials and endpapers—all of which are subject to physical damage from repetitive folding action, potentially resulting in detachment of the boards. To reinforce loose hinges that are still intact, a long pointed tool or syringe can be used to insert diluted starch or a VAE dispersion into the tight space between the hinge and the spine.

Three groups of techniques used to replace broken hinges are:

Extended sewing supports

The first group of techniques generally relies on the physical connection of the new hinge material to attach the board to the text block; however, adhesives can be used to attach new materials and to reattach original materials as part of the hinging process (see *Rebacking*).

Spine lining extensions

The second group of techniques relies on adhesive bonding of new hinge materials to the spine or shoulder of the text block and the board.

Cloth flange

This technique employs a cloth spine lining with extensions that can be adhered to either the recto or verso of the cover board and acts as a hinged board attachment. A few different types of adhesives have been used for this application. For instance, on a 13th-century book, rice starch paste was used to adhere a cloth flange to oak boards and provided a reversible attachment consistent with original construction materials (Quandt 1991). Also, washed and pre-shrunk aero linen adhered to 17th-century pulp boards with starch paste provided a sympathetic, reversible attachment (Honey 2003). In addition, PVAC has been used during a cloth flange board reattachment process designed for either large or small tight-backed books sewn on raised bands, requiring a very strong bond (Brock 2001).

Board-slotting

PVACs (including VAE dispersions) or gelatin will provide a stronger bond than paste for attaching the repair cloth into the slot when using a board-slotting technique (Fredericks and Hellman 2001). Both the PVACs and the starch paste have a longer working time than gelatin, which is helpful when inserting the cloth into the slot. Starch paste may be preferable, however, due to its well-known permanence and sufficient adhesive power to hold the fabric inside the slot. Cotton in general has a better fold endurance than linen and may be a more durable hinge material (Zimmern 2000).

Strip repair

The third group of techniques relies on adhesive bonding of new hinge materials to the shoulder or endleaves of the text block and the board.

Japanese paper hinge

Depending on the size, weight, rarity and condition of the book, starch paste may form an adequate bond between the Japanese paper hinge and the board or the surface material on the board (Primanis 2000). Etherington (2007) suggests that PVAC or a PVAC/starch paste mix forms a stronger, more reliable bond between repair tissue and old leather than starch paste alone. In a study by Dorning (2005), fold endurance testing after eight months of aging and 1000 folds revealed that the strongest hinging samples were adhered with a wheat starch paste (WSP)/Evacon mix with surface coatings of Liquitex and SC6000. Evacon is a VAE dispersion while SC6000 is a blend of waxes with an acrylic resin emulsified in aqueous isopropanol. As well, Lascaux 498 HV applied to Japanese tissue and employed as a solvent-activated repair tissue can be used with the following advantages: quick application, improved strength and stability of the repair material, ease in colour matching repair tissue to binding substrate and reversibility of the adhesive. Unlike Lascaux 360 HV, Lascaux 498 HV is not tacky at room temperature. As a final note on possible adhesives for this technique, a solvent-based application method is less likely to darken or damage aged leather, as is common with direct application of water-based adhesives (Puglia and Anderson 2007).

POULTICE

Crystallized and crumbling adhesives on the text block spine may need to be removed before

the application of new adhesives and linings. To avoid excessive dampening of spine folds of signatures and joint areas, dry scraping of old adhesives and/or minimal poultice application can be effective to soften water-soluble adhesives and cellulose linings. WSP, MC and Laponite RD are suitable for poultice preparations (**Figure 4**). The viscosity and pH can be adjusted. Application can be controlled by avoiding areas where humidity could cause damage, such as textile sewing supports and thread, or by application through a synthetic or cellulose release paper to prevent build-up of poultice in spine folds and the creation of tide lines and stains in the text block.

Consolidant

The surfaces of bookbinding leathers often require consolidation due to red rot or deterioration in the grain layer. Consolidation should be considered to be irreversible because of penetration of the consolidant into degraded (or deteriorating) porous materials. Consolidation can be a final step in a conservation treatment or it may also facilitate further steps, as in the consolidation of a leather tight-back spine before its removal from the text block (Cains 1994). Gelatin can provide a strong bond to the flaking grain layer of leather enabling gentle flexing without further loss. However, some discolouration of leather can occur (Cains 2007). Flaking or detached grain layer can also be reinforced with an application of Paraloid B-72 in acetone which does not darken leather, but can leave it hard and inflexible (Puissant 1994).

Klucel G (hydroxypropylcellulose) is a versatile surface consolidant for flaking and powdering leather bindings because it can be dissolved in a variety of solvents at various concentrations in such a way that it imparts strength, gives adequate penetration, minimal darkening and a matte finish. Klucel G (1–2% in ethanol or isopropanol) can be an effective consolidant and can be applied in multiple thin layers by brush or airbrush (Primanis 2000). However, it may not penetrate past the surface layer nor improve cohesion between the grain layer of leather and the layers beneath (Fredericks and Hellman 2001). The film formed by Klucel G is known to break down in the presence of sulfuric acid, a by-product of leather deterioration. Klucel G film is vulnerable to stress fracture and therefore is most effective when applied to semi-rigid materials such as book covers and may not be as effective as a film on leather joints or flexible spines. Klucel G should be stored in a tightly closed container and in a dry atmosphere to prevent any increase in moisture content that could lead to darkening of leather (Cains 2007). Consolidation of red-rotted leather with Klucel G seals the powdering surface, making it an acceptable base for the aqueous adhesives required for a Japanese paper hinge board attachment (Fredericks and Hellman 2001). Klucel G can be used to adhere a reinforcing facing tissue to brittle leather prior to lifting and can be subsequently removed after completion

Figure 4. Detail showing the complete removal of multiple layers of adhesives and spine linings using a poultice of Laponite (5% in water).

(Espinosa 1991) (**Figure 5**). Following consolidation with Klucel G, WSP can be used to adhere facing tissue without causing discolouration of the leather (Levy 1987).

For strength, flexibility, penetration and a surface finish more appropriate for aged leather bindings, a mixture of equal parts (1:1:1) ethanol, SC6000 and a Klucel G solution (2% in ethanol) provides a good level of protection (Ruzicka et al. 2007). Lascaux 498 HV in isopropanol (1:6) yields a clear, slightly viscous mixture that requires only one coat to consolidate most leathers. It leaves the leather flexible and provides a strong bond between the aged leather and the solvent-activated repair tissue (Puglia and Anderson 2007).

Figure 5. Adhesion of facing tissue to spine leather using Klucel G (3% in ethanol).

Leather and Skin

by Carole Dignard and Janet Mason, Canadian Conservation Institute

Adhesive treatments for leather and skin are generally used to repair tears, reinforce weak areas, fill gaps or consolidate. The type of treatment and adhesive will vary depending on the object's purpose or use, as well as on the type of skin or leather and its properties and condition.

Properties and condition of objects that influence adhesive choice

There are many types of leather and skin in collections. How a skin is processed and tanned influences its characteristics, deterioration and options for treatment.

- *Vegetable-tanned leathers* of various thicknesses, flexibilities and surface finishes are common in museum collections, as found in furniture and automobile upholstery, wall hangings, saddlery, etc. These leathers can stiffen with disuse or can become more acidic and weaken with time. In some cases, they become extremely weak and powdery—a condition known as 'red rot'—which makes them more vulnerable to tearing and losses.
- *Iron-tanned* or *black-tanned skins* can suffer from 'black rot', characterized by extreme weakness and brittleness.
- Native *semi-tanned skins* and *fur-skins*, also known as brain-tanned skins or as smoked-tanned skins, are typically soft, flexible and plump, as seen in clothing items like buckskin vests, moccasins and parkas. Other items such as tents are made of thicker, stiffer hides. Semi-tanned skins can become less flexible upon disuse or if exposed to water.
- *Oil-tanned (chamois) leathers* are generally soft and flexible. They are often used to make soft bags or pieces of clothing, including fur clothing. Chamois leather is strong, resistant to water and very absorbent.
- *Alum-tawed skins*, used for example to make gloves and other fine accessories, are not as stable in water as tanned leathers; higher fat contents improve water resistance.
- *Rawhides* are tough, stiff skins of variable thickness with multiple uses, such as for shadow puppets, bags, etc. Some rawhide objects found in collections may have been originally oiled, like kayaks and sealskin floats, and this oiled surface presents additional challenges.
- Thin translucent *gut-skins* used by the Inuit to make parkas are challenging due to their stiffness, thinness and oily surface.
- *Stretched* or *restrained skins*, such as those found covering wooden chests and shields or on drums, may pose additional challenges with respect to access to the skins, clamping and anisotropic environmental stresses.
- *Archaeological leather* is often found in fragmentary condition and can be quite weak, friable, brittle and delaminating.

Adhesive properties considered

Discussions on which adhesives to choose for treatments of different skins and leathers have been published, for example, by Fenn (1984), Wouters (1989), Calnan et al. (1991), and Kite and Thomson (2006). When choosing an adhesive treatment, all treatment components, such as use of heat, water or solvents, clamping method, etc., need to be assessed in relation to the skin or leather characteristics and condition.

Use of heat

With skins of low hydrothermal stability (shrinkage temperature <40°C), use of water should be avoided because it may cause irreversible fibrillar shrinkage, resulting in hardened, distorted or darkened skin, or even gelatinization, thinning or holes. Heat and water together are especially to be avoided because water swells the collagen macromolecule (the main polymer in skin and leather), which then becomes vulnerable to heat denaturation. Applying heat alone to dry degraded skin is also a concern, although unswollen collagen is more resistant to heat denaturation. Experience has shown that dry heat in the range of 60–75°C is usually safe, but testing is necessary. Minimizing temperature and time of exposure remain important.

Use of water and solvents

The effects of solvents must also be assessed. Some types of skins or leathers are definitely harmed by water (e.g. alum-tawed skins). Water and polar solvents can cause staining, stiffening and/or hardening, or surface migration of tannins. Generally, less polar solvents have less effect. In addition, water, and to a lesser extent polar solvents, can cause

dimensional swelling and shrinkage, which may lead to unwanted movement in aligned tear edges during the repair stage. Water and solvent fastness of dyes must also be checked.

PENETRATION

Penetration of the adhesive or solvent into a skin depends on condition and porosity. Semi-tanned skins, glove leathers, fur-skin clothing and vegetable-tanned leathers in particular can wick-up adhesive or solvent, possibly causing stiffening, stains or darkening. Dry adhesive films can minimize penetration because the activation methods can create a tacky but not runny adhesive and a surface (nap) bond. Also, high viscosity adhesives provide reduced penetration. Conversely, adhesive penetration may be needed to impart strength to weak substrates, or to consolidate brittle or powdery skins. Penetration can usually be controlled by the solvent, adhesive viscosity, application method and quantity of adhesive applied.

REMOVABILITY

The ease and extent of adhesive removability is relative to the type of adhesive, the quantity applied, the solvent/carrier, the skin porosity, the degree of penetration, and the application method used. If water or solvents are not appropriate for the skin, this limits removability options. Surface (nap) bonds with minimal penetration are more easily removed than bonds with deeper penetration. Mechanical means like peeling or prying with small tools, spatulas and scalpels may work well, if necessary, combined with various methods for swelling or dissolving the adhesive (e.g. use of small amounts of solvents, solvent vapours, poultices or solvent gels). Treatments requiring deeper capillary penetration (e.g. consolidation) are virtually impossible to remove completely. The stability of the adhesive upon aging is of prime importance and is critical for treatments involving penetration.

BOND STRENGTH

Adhesive bond strength must be sufficient for the needs of the object and its intended use, and vary depending on whether the skin or leather must withstand certain stresses (e.g. if the repair is load-bearing or is exposed to fluctuating climates). The application method and solvent used can affect strength. Good contact is essential to create a strong bond. Surface oils will repel a water-based or polar adhesive, while surfaces that are powdery, dusty, or covered in other deposits may also interfere. Uneven surfaces that need to be joined back in plane also pose challenges; reshaping treatments are usually carried out as a separate step prior to adhesive treatments. If areas undergoing repair remain to some extent deformed or misaligned, use of a water-based or polar adhesive can have the additional benefit of helping to soften and reform small distortions, thereby improving contact and alignment.

FLEXIBILITY

Flexibility of the repair should generally match the flexibility of the skin or leather. The flexibility of any backing material used will also have a large overall effect.

pH

The pH of the adhesive should be in the range of the normal isoelectric point of the skin or leather or towards a neutral range. The pH of leather or skin depends on their processing and tanning (e.g. rawhide has a pH between 5 and 6; vegetable-tanning shifts the pH towards the acidic side).

COMPATIBILITY AND CLAMPING

Compatibility of the adhesive with other treatments, such as cleaning or impregnation, or the extent of access to the skin for positioning and clamping are other aspects influencing adhesive choice.

ADHESIVES FREQUENTLY USED

Adhesives typically used for skins and leathers are given in **Table 1**.

TABLE 1. TYPICAL ADHESIVES USED FOR SKIN AND LEATHER CONSERVATION

Type of treatment	Adhesives commonly used
backing/lining	BEVA 371, BEVA 371 Film, Lascaux 498 HV, Lascaux 498 HV + Lascaux 360 HV, Paraloid B-72, PVAC resins (e.g. AYAA), cellulose ethers (e.g. Klucel G), wheat starch paste (alone or mixed with other adhesives)
tear edge repairs	BEVA 371 Film, Lascaux 498 HV
fill	BEVA 371, BEVA 371 Film, Paraloid B-72, PVAC resins (e.g. AYAA), cellulose ethers (e.g. Klucel G)
consolidation	cellulose ethers (e.g. Klucel G), BEVA 371, Paraloid B-72, Paraloid F-10, PVAC resins (e.g. AYAA)

BACKING OR LINING

Backing or lining treatments are common to strengthen weak skins and leathers or to rejoin tears. Good, overall contact is required to provide even support. In localized repairs (patches), the material bridging each side of the tear must be wide enough because this affects the strength of the repair. Possible backing materials include:

- various leathers and skins;
- parchment or vellum;
- skin membranes [e.g. goldbeater's skin, fish skin, cow gut, regenerated (patent) sausage casings, lambskin prophylactics];
- Japanese papers;
- woven fabrics [e.g. silk crepeline, gauze, cotton, linen, polyesters such as Tetex (formerly Stabiltex)]; and
- non-wovens (e.g. polyesters such as Hollytex, Reemay, Bondina T1780; nylon gossamer such as Cerex; and viscose such as Tootal).

Properties considered when choosing a backing material include thickness, strength, flexibility, texture, weave, stability, and how well it sticks and conforms to the object. The composition, texture and surface finish of the backing material can affect adhesive wetting and repair strength. Porous backings like woven fabrics may not be suitable for tacky adhesives. Because of their weave, woven fabrics react differently than skin and leather to relative humidity (RH) fluctuations. If the backing repair is visible, ease of dyeing or colouring needs to be considered.

BEVA 371 and BEVA 371 Film

BEVA 371 and BEVA 371 Film are useful as backing adhesives for a variety of skin and leather objects; see Kronthal et al. (2003) for a discussion. Advantages include known aging characteristics, removability, strong bonds, low reactivation temperature, flexibility and good tack. Their application by heat-reactivation (**Figures 1** and **2**) is fast, precise, simplifies clamping and avoids solvent problems. Examples discussed in the literature include treatment of upholstery leather (Calnan 1991), gilt leather (Tsu et al. 1999; Calnan 1992), a saddle (Sturge 2000), semi-tanned skins (Richardson 2002; Dumka 2006), fur-lined capes (Kite 1990; Dignard and Gordon 1999), suede gloves (Kite 1996), shadow puppets (Falcao 2007; Kronthal 2001), and both a drum-skin and a lizard specimen (Nieuwenhuizen 1998). When heated, BEVA 371 turns fluid fairly suddenly; penetration is controlled by managing the quantity of adhesive used, temperature, dwell time and pressure. Treatments can also be done using BEVA 371 brushed wet or using BEVA 371 Film (or BEVA 371 cast as a film) reactivated with solvents or vapours. These bonds are weaker than when heat-activated. Applications avoiding heat have mainly been used for untanned and semi-tanned skins (Heine 2003; Kronthal 2001; Boulton 1986; Fenn 1984).

Water-based polymer dispersions

Water-based dispersions such as those made of vinyl acetate/acrylic copolymers, vinyl acetate/ethylene copolymers, and acrylic polymers have attractive properties as backing adhesives for skins and leathers: strong flexible bonds can be achieved, the moisture helps soften stiff uneven joins, their relatively high viscosity can reduce the risk of unwanted penetration and darkening, and their health risk is minimal. Some commercial products have questionable stability (e.g. yellowing, acidity, acid emissions) and removability; therefore, testing is required to determine suitability. Residual tackiness in the case of those that have a low glass transition temperature (T_g) may also be a concern. Cellulose ethers and/or wheat starch paste (WSP) have been used to improve reversibility or to control other properties, such as bond strength, drying time, aging properties or flexibility (Raphael 2003; Morrison 1988).

In particular, the acrylic dispersion Lascaux 498 HV is frequently used for backing or lining leathers and skins, sometimes in combination with Lascaux 360 HV. These Lascaux dispersions have a thick whipped texture that avoids drip problems and reduces risks of excessive penetration, yet, unlike a highly viscous adhesive, they can be easily spread to a thin film. Advantages include a strong nap bond, good stability, long-term solubility (in acetone or toluene) and removability (solvent swelling and/or mechanical peeling). Lascaux 498 HV produces a flexible film with light tack, while Lascaux 360 HV produces a softer film that may be too tacky for many applications but which can slip if tensions occur. Mixtures of the two produce intermediate degrees of tackiness and flexibility. They are applied by brush or can be cast into films or onto a backing material and reactivated using heat, solvents or solvent vapours. They are useful for a large range of objects. Examples of backing repair applications for the Lascaux products include:

- Lascaux 498 HV used on buckskin (**Figure 3**), on rawhide (Dicus 1995), and on parts of a black-dyed skin pouch (Cruickshank et al. 2009);
- Lascaux 360 HV used on a drum-skin (Bonnot-Diconne 2000); and
- mixtures of Lascaux 360 HV and Lascaux 498 HV used on archaeological leather (Lafrance 2010) (**Figure 4**), on gloves (Sturge 2006a) and on various vegetable-tanned leather and/or gilt leather (Calnan 1991; Sturge 2000; Kite and Thomson 2006, pp. 268–324).

Figure 1. An 1876 leather upholstered chair with losses and tears (Mason 2010). Left: Before treatment. Right, top: An old leather fill adhered with hide glue had darkened and hardened, causing new damage. Right, bottom: During treatment, the area of loss was backed with cotton or with a non-woven coated with BEVA 371 Film, then filled with a leather skiver attached by heat-reactivating the BEVA 371 Film. The leather skiver was later toned. Using BEVA 371 Film facilitated backing and placement, aided by spot-tacking. Exposed fibres were consolidated where necessary with Klucel G (2% in ethanol).

Figure 2. Treatment of a 19th-century fur-trimmed black velvet cape (Dignard and Gordon 1999). Top: The fur trim during its removal from the cape. The fur was extremely weak and torn throughout. Bottom, left to right: Backing repairs using Tetex (Stabiltex) polyester net flocked with BEVA 371, heat reactivated onto the fur-skin. Individual fragments were pieced together locally using small backing repair strips, then five larger Tetex-BEVA backings were applied overall, which provided additional strength and allowed stitching the fur back onto the cape. Bottom far right: After treatment.

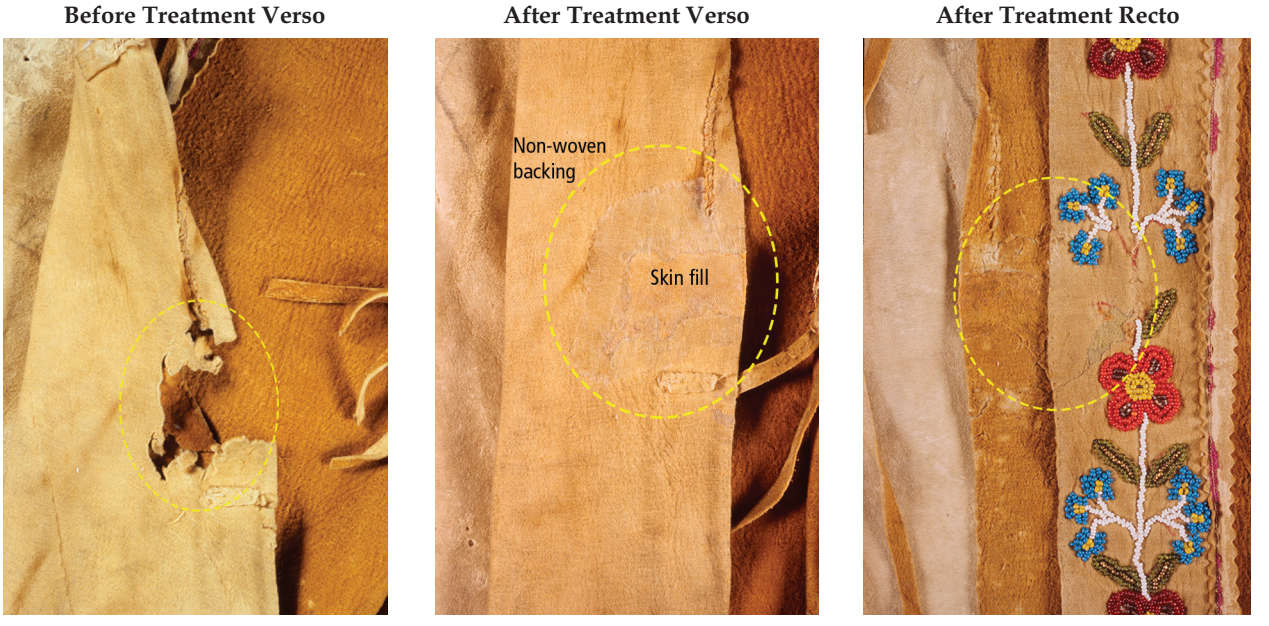

Figure 3. Backing treatment of a Native-tanned skin coat (Mason 1996). Left: Detail of the front flap of a beaded Native-tanned skin coat before treatment (verso), showing tears and loss. Centre and right: Detail after treatment, respectively of verso and recto. Tear edges were aligned by applying strips of non-woven polyester coated with Lascaux 498 HV. After drying, a larger non-woven backing was applied over the area of damage. A Native-tanned skin fill was cut to match losses and adhered on top of the backing using the same adhesive, and the edges of the fill and perimeter of the original skin were also butt joined. Weights were applied until the adhesive was dry.

Figure 4. Backing treatment of an archaeological leather boot (Lafrance 2010). Left: Leather boot after treatment. Right: Detail after treatment of heel. Areas where leather was friable or fibres were exposed were consolidated with Klucel G (2% w/v in ethanol) applied by brush or syringe. Damaged area at heel was reinforced by backing with strips or patches of Reemay covered with a dry layer of Lascaux 498 HV and Lascaux 360 HV (50:50 w/w) and reactivated using acetone. The repair was efficient, visually appealing, easily identifiable and reversible. The adhesive strength was enough to hold the pieces together while allowing flexibility.

Other water-based polymer dispersions that practitioners have used include:

- Mowilith DM5 on 'Russian leather' boots (Volken and Volken 1995);
- Mowilith DMC2 on gilt leather (Bonnot-Diconne and Fournet 2007; Chiotasso and Sarnelli 2007), bird-skin parkas (Rae and Wills 2002), sealskins (Ruppel 2002) and archaeological leather (Montembault 2001);
- Vinamul 3254 on upholstery (Angus and Joram 1995; Calnan 1991) or gilt leather (Calnan 1992);
- Hewitt Reversible M218 on a python skin chair (Sturge 2000) and leather upholstery (Beaumont and Sturge 2006); and
- Jade 403 on upholstery (Trupin 2003) and semi-tanned skins (Long 2001).

Solvent-based resins

Solvent-based acrylic resins, in particular Paraloid B-72, and poly(vinyl acetate) (PVAC) resins, such as AYAA, AYAC and Mowilith 50, are also possible choices in leather and skin repairs. They are valued for their good stability and reversibility, and provide options when water or heat must be avoided. They are fairly to moderately stiff, and are used to repair stiff to moderately flexible leathers and skins. They are used typically at high viscosities, which may cause handling difficulties such as stringiness. They are generally applied by brush. Care must be taken to avoid unwanted penetration into porous skins, leading to stiffness or staining. They can be quick drying depending on the solvent (e.g. acetone). They can also be cast as a film and reactivated. They are glossy when dry.

Examples of the use of solvent-based resins include:

- Paraloid B-72 to repair ethnographic skins (Kaminitz and Levinson 1988), a kayak (Marchand 1998), military straps, helmets and scabbards (Storch 1989), a saddle (Selm 1989), and chair upholstery and a leather-covered coffer (Spicer 1995). Paraloid B72 can also be mixed with cellulose ethers to increase viscosity, tack or flexibility (Vuori 1985). Examples include mixtures with ethylhydroxyethylcellulose to repair a kayak (Dignard 1992) (**Figure 5**) and with Klucel G to repair gut-skin parkas (Dumka 1991);
- Paraloid B-48N on a bird necklace (Kaminitz and Levinson 1988);
- Mowilith 50 on a bird-skin cloak (Rae and Wills 2002), a model kayak (Cruickshank 1987) and on polyethylene-glycol-treated seal gut-skins (Hill 1986); and
- AYAA on a skin membrane bag (Kaminitz and Levinson 1988).

Cellulose ethers

Cellulose ethers generally produce weak bonds that may not withstand flexing, but which may be

Figure 5. Treatment of a large tear in a sealskin kayak. Left, top: Detail of a large tear in the sealskins of a kayak. Left, bottom: Adhesive backing repair, using Japanese paper backings adhered with Paraloid B-72 mixed with ethylhydroxyethylcellulose. Parafilm and styrofoam wedges were used to clamp the skins onto their backings, using the kayak's wood structure behind as a support. Right: The gap was filled with a mixture of paper pulp fibres in a methylcellulose binder, and inpainted with acrylic paints.

adequate in some cases. Klucel G is predominantly used; it is soluble in water or in various solvents. Examples of uses include backing treatments on fans (Hermans 1992), moccasins (Chapman 1987), parkas (White and Sully 1992; Fenn 1984) and black-dyed skin (Cruickshank et al. 2009). Cellulose ethers are also commonly used as modifiers, with dispersions or with acrylic resins (see *Water-based polymer dispersions* and *Solvent-based resins*, above).

Wheat starch paste (WSP)

WSP is reversible in water and gives good but stiff bonds that may not be appropriate if flexing is expected. Disadvantages are linked to high water content, which can lead to staining, puckering or shrinkage. Using WSP 'almost dry' helps avoid these problems. It does not gap-fill, therefore intimate contact is required. It does not adhere well to oily surfaces or some synthetics. Examples of use include treatments of gilt leather (Paris et al. 2008; Angus et al. 2006), a leather-covered box (Sturge 2006b), alum-tawed gloves (Beaumont 2006), fans (Hermans 1992) and an archaeological bag (Kaminitz and Levinson 1988). Adding a PVAC or an acrylic dispersion to WSP produces a mixture which has increased strength and tack, as compared to WSP alone. The WSP in such mixtures helps to slow the drying and improves the reversibility as compared to the dispersions alone (Raphael 2003). Sometimes a cellulose ether is added to these mixtures as well. Mixtures of WSP with sodium alginate have also been used, for example, on a fur-lined robe (Kite 1991).

Collagen-based adhesives

Collagen-based adhesives, such as parchment size, gelatin, isinglass and animal glues, are traditional materials, with parchment size historically used to repair vellum and parchment. They are stable and removable in water. Disadvantages include potential harm of using both water and heat on deteriorated leather, strong reactions to RH changes, significant shrinkage upon drying and fairly brittle films. Examples of use include cooled isinglass to repair a salmon-skin robe (Kite 1999) and parchment size adhering paper backings to gilt leather panels (van Kempen 2007).

TEAR EDGE REPAIRS

Direct tear edge repair—butt, lap or scarf joint repairs—brings torn edges together as close as possible and in plane. This type of repair may be deemed useful to provide extra strength when carried out in combination with a backing repair, or with fills (**Figure 3**). As a treatment on its own, the small contact area usually produces a weak repair, especially in butt joins. However, if the bond is strong enough, a tear edge repair alone has the advantage of being less invasive than a backing repair. It is useful for small rips, cuts or when a backing is not possible due to aesthetic requirements or lack of access. Strength, flexibility and high viscosity are usually important adhesive characteristics for tear edge repairs. BEVA 371 Film, as used by Richardson (2002) to repair Native-tanned skins, and Lascaux 498 HV are possible adhesive options for tear edge repairs.

FILLS

Broader discussions on gap-filling are found in Calnan et al. (1991), Raphael (1985) and Nieuwenhuizen (1998). Common types of fills are those made of new skin or leather similar to the original (see **Figures 1** and **3**) adhered to a gap perimeter using a lap, scarf or butt join, and most often are combined with a backing reinforcement. Adhesives employed are typically the same as those discussed under *Backing or lining*. Other solid sheet materials that can mimic the appearance of the leather or skin surface under treatment are possible, for example, some non-wovens, or films of resin mixtures cast to replicate the grain of adjacent leather surfaces [e.g. Paraloid F-10 mixed with microspheres (Nieuwenhuizen 1998)].

Fills can also be made by applying a paste consisting of an adhesive with a filler into the gap previously lined with a backing material and letting it dry. After drying, the process is repeated to build up the area to the right thickness. BEVA 371 and BEVA 371 Film are commonly used in this way as a paste fill material. Other adhesive polymers that can be used include acrylic resins (e.g. Paraloid B-72, Paraloid F-10) and PVAC resins (e.g. AYAA, AYAC or equivalent, as these are no longer available). Glass microspheres, cellulose powder, shredded leather, powdered parchment or paper fibres are used to increase bulk and reduce shrinkage. Dry pigments are often added, also providing bulk and reducing shrinkage. Fumed silica can improve modeling properties. Other types of paste fills can also be made of paper fibres mixed with small amounts of cellulose ether (Dignard 1992; see **Figure 5**) or WSP (Boulton 1986). Typically, polymer paste or fibrous paste fills do not reproduce a skin or leather appearance as well as fills made of new skin or leather, or fills made of cast films. They are useful when those other options are impractical because the gap is small, narrow or irregular (e.g. along a crack). Desired properties of paste fills include thick consistency, fast drying and minimal shrinkage upon drying. Using a solid insert made of new skin or a non-woven to fill most of the gap and then surface-finishing it with polymer paste is a variant that reduces shrinkage when building up layers. Other desired properties include good

aging and reversibility, flexibility, strength, abrasion-resistance, long-term dimensional stability and ability to match the original.

CONSOLIDATION

In-depth consolidation is aimed at increasing internal cohesive strength; this is considered mainly for powdery surfaces (red rot), for extreme brittleness (black rot) or for weak archaeological finds. Long-term stability and good aging characteristics of the consolidant are essential since the treatment is virtually irreversible. Typically, the consolidant must penetrate well and impart strength and flexibility without altering appearance. A high T_g is advisable and is required in hot climates (Wills 2001). Water-based consolidants, as well as those dissolved in polar solvents, can darken and harden degraded skins. Klucel G in non-aqueous solutions is used for consolidation of skins and leathers (see **Figure 4**). It can penetrate degraded leather without causing darkening or staining and it provides strength. Another option is Paraloid B-72 (Wills 2001; Fenn 1984). A retrospective discussion on past use of Pliantex (which is no longer available) is found in O'Donnell (1997). Consolidants for archaeological leather, such as acrylic resins, cellulose ethers, poly(vinyl butyrals) and ethylene/vinyl acetate copolymers, are discussed in Cameron et al. (2006) and Wills (2001).

Surface consolidation may be needed for worn areas showing loss of grain. Adhesives used for such applications have included Klucel G or Paraloid F-10 (Mason 2010; Bonnot-Diconne and Fournet 2007). Delamination between the corium and grain layer may require consolidation, as was described for wall hangings treated with BEVA 371 (Tsu et al. 1999). Lifting epidermal layers may need securing, for example, by using Paraloid B-72 (Heine 2003) or WSP (Sturge 2000, p. 15). Surface consolidants do not require deep penetration as bulk consolidants do. Useful properties of surface consolidants include good control of penetration where needed, good wetting, no residual tackiness and good appearance (no staining or darkening).

Textiles

by Irene F. Karsten and Jan Vuori, Canadian Conservation Institute

Adhesive treatments for textiles are complex. Research has confirmed what conservators have observed through practice, namely that achieving an adhesive bond that will effectively support a textile artifact is not due to adhesive choice alone (Karsten 2003). Many factors are involved, including characteristics of the textile artifact, the support, materials and processes required to adhere that support to the artifact, future use of the artifact, the conservator's experience, and the availability of materials, equipment and required safety measures (e.g. fume extraction). The textile artifact, its fibres, dyes, applied decoration, construction, condition, etc., will dictate which adhesive, solvent or procedure may *not* be applied to it. Within these limits, it is possible that one of several adhesives and methodologies *can* be implemented to produce an effective and acceptable treatment. As textile conservators confront new challenges and continue to learn from evaluating past treatments (Lewis 1998; Hillyer 1998; Hillyer et al. 1997; Hillyer 2010), treatment methods and rationales will continue to be refined.

The primary use for adhesives—and the focus of this section—is for attaching a fragile or fragmenting textile artifact to a support fabric (backing). Other applications include reattaching textile components that were originally adhered as part of composite objects; reattaching decorative elements on textiles, particularly those that were originally adhered; and attaching facings to support a weak or fragmented textile temporarily during treatment (Cruickshank et al. 1999; Hackett and Szuhay 2003). Adhesives are also used for consolidating paint on painted textiles, a treatment that requires the participation of a paintings conservator (AIC Textile Specialty Group 2011).

Adhesive use remains far less frequent in textile conservation than in most other conservation disciplines. Conservators generally acknowledge that adhesive treatments on textiles are never totally reversible. Even if the adhesive should remain soluble, complete removal from a porous, fibrous and weakened surface without damage is impossible. The risks, benefits and feasibility of an adhesive treatment must, therefore, be evaluated against other treatment options such as stitching or pressure mounting. Most applications involving adhesives are restricted to textiles that cannot be treated by these means, a small percentage of the work carried out in most textile conservation labs.

Moreover, successful adhesive treatments depend on technical skill and judgment that can only be gained by experience. Even with formal instruction in the techniques involved, which is highly recommended, conservators may find it difficult to achieve and maintain the in-depth expertise they need to carry out such treatments. Inappropriate or unskillful adhesive treatments can cause serious and permanent damage to textiles.

Textile object characteristics that influence adhesive use

Flexibility

Flexibility, or drape, a fundamental characteristic of textiles, is reduced whenever a textile is attached to a support material whether by stitching or adhesive. Flexibility of an adhered textile is retained primarily in two ways: by limiting adhesive contact with, and penetration into, the textile through the use of adhesive reactivation techniques, and by using an adhesive with a polymer glass transition temperature (T_g) around or below room temperature. Adhesives with a lower polymer T_g have a slight tack that can be helpful in positioning the support on the artifact prior to reactivation but remain tacky and can attract dirt or stick to adjacent materials. Adhesive flexibility is not usually important for textiles that will remain flat on a rigid support after treatment.

Composition and construction

The chemical and physical properties of a textile artifact will determine if, and limit how, an adhesive treatment is carried out. Treatment materials and methods, particularly adhesives and solvents, must not react adversely with any component of the artifact. Fabric construction is also influential. Adhesive backing treatments best support smooth, closely woven textiles and may be appropriate for fragile textiles that would be visually disturbed by stitching. An exception is the obverse of a fragile warp-faced satin, which will not benefit from a support fabric adhered to the reverse (Lord 1997). Complex or thick, heavy textiles, such as tapestries, are not properly supported by adhesive backing. Highly textured textiles, such as embroideries with floats and knots on the reverse, may not bond well using reactivation techniques. Such textiles have been successfully backed onto paper using starch (Kite and Webber 1995).

Adhered supports are most effective if the adhering surface, normally the reverse, is fully accessible. Multiple layers, three-dimensionality, puckers, pleats and gathers (common on costume), and fabric distortion can limit such access. These textiles may require an adhesive that forms a stronger, more flexible bond and a support material that is more conforming to the artifact. In some cases, disassembly can provide this access (Hartog and Tinker 1998); however, even partial disassembly is a major intervention and would never be carried out unless the benefits outweigh the loss of original construction details. When disassembly is not possible, sheer supports have been applied to the obverse of textiles (Gill and Boersma 1997; Lord 1997). Moreover, complex structures make it difficult to ensure that the grain of the artifact and the coated support are aligned, which is believed to maximize the flexibility of the adhered laminate (Bede 1993). Cutting the prepared support into pattern pieces that match the artifact structure facilitates alignment, although perfect matching is not always possible (Hartog and Tinker 1998).

CONDITION

Although most textiles treated with adhesives are in poor condition, certain conditions argue against the use of adhesives. Powdering textiles are usually not good candidates for adhesive backing because, with time and handling, the support will no longer be attached to the textile but only to the powdering fibres. Extremely brittle textiles may not be able to endure the handling and pressure involved in the treatment. Some forms of deterioration, such as shattering caused by weighting silk with metallic salts or tendering caused by dyeing or printing with iron mordants, are progressive and thus a full adhered support should be considered even if current damage is localized (AIC Textile Specialty Group 2011).

Loose particulate soiling, stains, and residue from previous adhesive treatment can adversely affect the strength and effectiveness of an adhesive bond. Cleaning is, therefore, often carried out before a textile artifact is adhered to a support fabric. In addition, attempts are made to align the textile's weave structure. This allows the weight of the textile to be more evenly distributed over, and borne by, the support fabric (Keyserlingk 1993). The nature and condition of the textile will dictate the extent of cleaning and alignment.

FUTURE USE

How the textile will be displayed, studied and stored will influence choices regarding adhesive treatment. A large flat textile that will be rolled for storage requires an adhesive that produces a stronger, flexible bond, as does a costume that will be placed on and off a mannequin. For textiles destined for open display, adhesives with a polymer T_g >25ºC are considered desirable, while those with a polymer T_g <10°C are not (Pretzel 1997). Adhesive treatment often obscures one surface of the textile, thereby hindering future structural analysis. Adhesive residue may interfere with future instrumental analysis. The possible future need to clean the treated textile using water or solvents is also problematic.

ADHESIVE PROPERTIES CONSIDERED

Adhesives for textile conservation must fulfill criteria of physical and chemical stability, compatibility with the substrate, adequate adhesive strength, a colourless and non-yellowing appearance, and reversibility. Equally, the solvents and methods used to create the adhesive bond and undo it should not adversely affect the textile (e.g. fibres, dyes, applied decorations).

ADHESIVES USED IN TEXTILE CONSERVATION

Textile conservators have used many different adhesives for stabilization treatments (**Table 1**) (Hillyer et al. 1997; Karsten 1998). Synthetic polymers—primarily vinyl acetate copolymers and acrylics—are used if heat reactivation is chosen as the attachment method. Starch and other carbohydrates are used if heat or non-aqueous solvent reactivation is inappropriate. Solvent reactivation techniques may be used with synthetic polymers and carbohydrate adhesives, although the specific methods vary. Some adhesives, such as Klucel G (Haldane 2000) and mixtures of Lascaux 360 HV and Lascaux 498 HV (Thompson 2001), can also be cast as unsupported films which can be solvent-reactivated in-situ.

The particular properties of adhesives affect their performance in textile backing treatments, although this is often difficult to distinguish from the effects of the treatment method. Adhesives with low polymer T_g or modulus, such as BEVA 371, Lascaux 360 HV (alone or combined 1:1 with Lascaux 498 HV) or Vinamul 3252 will give more flexible adhesive coatings that are likely to stretch in response to stress resulting in bonds that resist failure when flexed. On the other hand, if peeling is desired for future reversal of the treatment, such adhesives are more likely to fail cohesively, leaving residue on the artifact surface. This particularly occurs when these adhesives coat only the yarns, are applied as a flocked coating (**Figure 1**), or anchor poorly to the

yarns as do mixtures of Lascaux 360 HV with Lascaux 498 HV (1:1) and coatings on monofilament nylon net (**Figure 2**) (Karsten and Kerr 2003). Stiffer adhesives like Clariant T1601 (a.k.a. Mowilith DMC2) or Paraloid F-10 tend to fail at the adhesive/artifact interface with almost no transfer of adhesive unless a thin, weak coating extends over large interstices in support fabrics like nylon net (**Figure 3**) (Karsten and Kerr 2003). The bonds formed by less flexible adhesives tend to be weaker (Karsten 2003; Karsten and Kerr 2011).

BEVA 371, which contains wax, can impregnate and stain textiles if reactivated with too high a temperature. When used for backing painted textiles, it is sometimes used only on the reverse of painted areas and the unpainted textile is backed using another adhesive (e.g. Mowilith DMC2) (Lochhead 1995). BEVA 371 is known to yellow under light but this may not be a problem on the reverse of textile artifacts. When peeled, brushed BEVA 371 coatings leave less residue on the textile than do sprayed coatings (Karsten and Kerr 2003). BEVA 371 Film (0.03 mm or 1.0 mil) is too thick for many textiles.

Klucel G is a relatively weak adhesive that is soluble in both water and organic solvents (e.g. ethanol and acetone). Low molecular weight grades are considered to have "intermediate" stability (Feller and Wilt 1990, p. 94). Dried coatings on sheer fabric have been solvent-reactivated as backings (Lord and Sutcliffe 2000), overlays (Petschek 1995) and as temporary facings (Harrison et al. 2005).

Table 1. Adhesives commonly used in textile conservation

Adhesive	Type of treatment
Synthetic polymers	
BEVA 371 (mixture of resins and wax in solvent)	backing: heat-reactivated, solvent-reactivated
Lascaux 360 HV and/or Lascaux 498 HV (aqueous dispersions)	backing: heat-reactivated, solvent-reactivated, unsupported film
Lascaux P 550-40 TB (resin in solvent)	backing: heat-reactivated
Clariant T1601 (formerly Mowilith DMC2) (aqueous dispersion)	backing: heat-reactivated
Vinamul 3252 (aqueous dispersion)	backing: heat-reactivated
Starch and carbohydrate adhesives	
mixture of arrowroot starch and carboxymethylcellulose	backing: cold lining, solvent-reactivated
hydroxypropylcellulose (Klucel G)	backing, overlay, facing: solvent-reactivated, unsupported film
methylcellulose	facing: solvent-reactivated, wet application
wheat starch paste	backing, facing: wet application

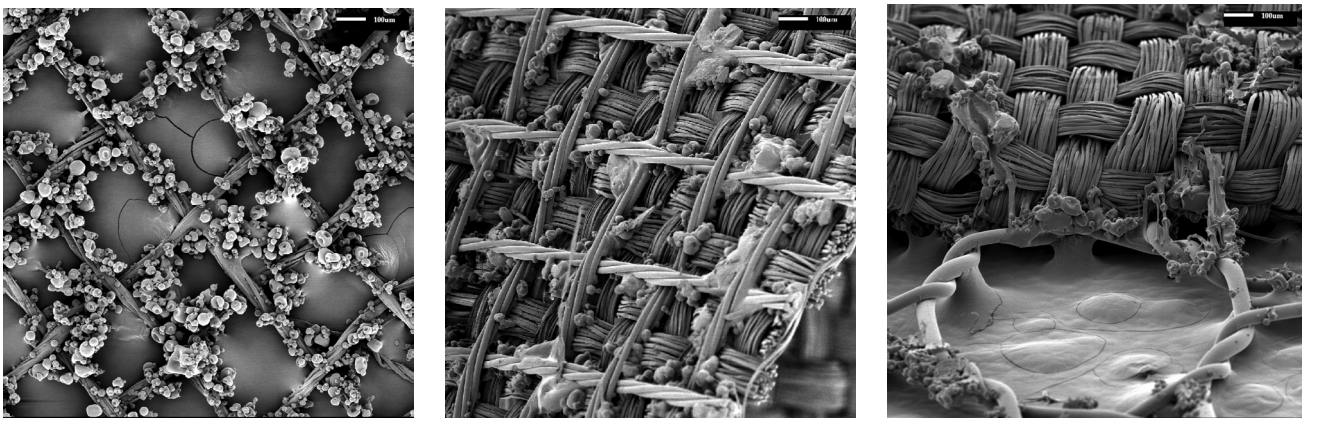

Figure 1. Flocked coatings of BEVA 371. The SEM micrographs show coatings of BEVA 371 (1:1 by volume in toluene) prepared by spraying on silk crepeline (left), polyester crepeline (centre, after heat-sealing at 65°C for 20 s to silk habutae) and nylon net (right, after heat-sealing and peeling).

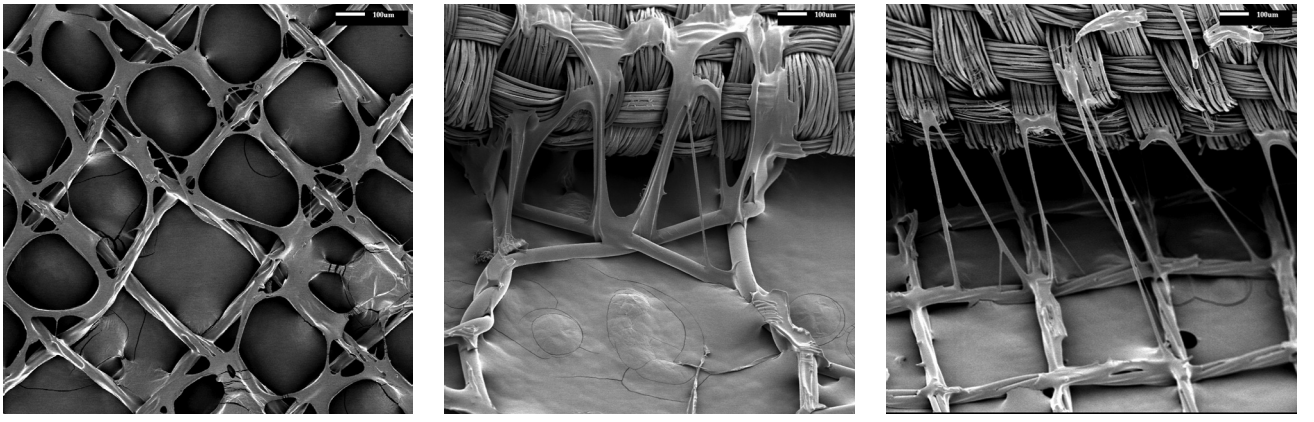

Figure 2. Transfer of poorly anchored adhesive coatings. The SEM micrograph (left) shows a poorly anchored coating typical of Lascaux 360 HV and Lascaux 498 HV (1:1 mixture applied by brush as a 1:5 solution by volume in water). After heat-sealing at 60°C for 20 s and peeling, this adhesive exhibits more transfer to silk habutae from nylon net (centre) than from polyester crepeline (right). Transfer from silk or polyester crepeline is not always visible to the naked eye.

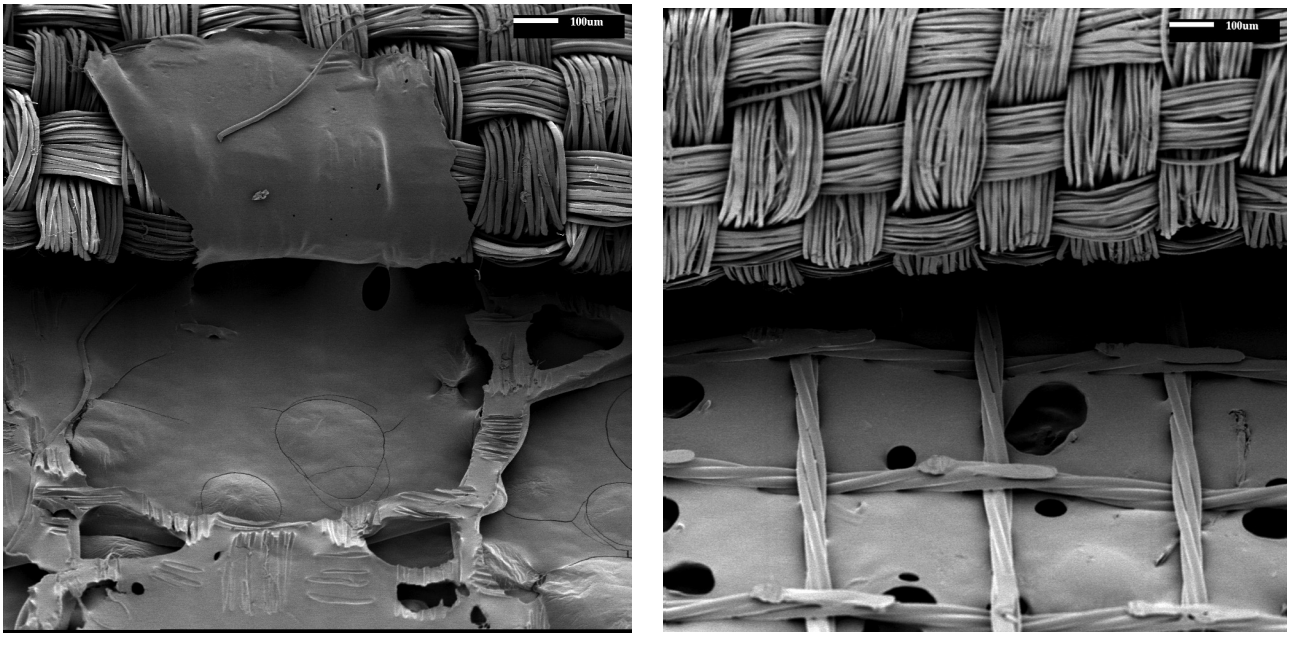

Figure 3. Peeling behaviour of stiffer adhesives. SEM micrographs of silk habutae samples adhered with Clariant T1601 (left, 1:5 solution by volume in water, brushed onto nylon net, heat-sealed at 95°C for 20 s) and Paraloid F-10 (right, 1:8 solution by volume in toluene brushed onto polyester crepeline, heat-sealed at 65°C for 20 s).

Methylcellulose is another relatively weak adhesive. It has been used in combination with cooked wheat starch paste to prepare remoistenable tissues used as temporary facings on textiles (Hackett and Szuhay 2003).

TREATMENT DESIGN

Research (Karsten 2003) and practice suggests that successful adhesive textile stabilization depends less on adhesive choice than on the skillful combining of materials and methods to achieve an adhesive bond that is just strong enough to meet treatment goals. An evaluation matrix has been developed that evaluates pertinent physical properties (pH, T_g, heat-sealing temperature, peel strength, tear strength) of selected adhesives together with working properties (ease of application to support, ease of handling the adhesive coated support, requirement for fume extraction, staining, adhesive residue on artifact after peeling) (Pretzel 1997). A scoring and weighting system enables the conservator to calculate

scores for the adhesives in the context of the particular artifact being treated. Although useful, this matrix is limited by results that are specific to the treatment methods used to create the samples. If different conservation techniques are used, the same adhesives can produce different results (Karsten 2003). A fuller understanding of how treatment design affects the behaviour of the adhesive combined with pre-testing is probably a more useful approach.

SUPPORT

Silk crepeline, polyester crepeline (Tetex TR, Stabiltex) and nylon net (20 denier, Dukeries #N8000) are the most commonly used support materials in adhesive treatments for textiles (**Table 2**). Sheer, lightweight yet adequately strong, they create a flexible laminate that still provides visual access to the artifact. Non-wovens such as papers, tissues and nylon gossamer (lightweight nylon Cerex) and spun-bonded and calendered polyester (Hollytex) have also been used.

In some cases, localized support in the form of patches may be used. However, patches can distort textiles, become more visible over time and cause splitting at the edges of the patch. Care must be taken with their size and placement to minimize stress at this juncture. Techniques include using thin patches with feathered edges. Sheer support fabrics, partially unraveled to create "band-aids" or "eyelashes" (AIC Textile Specialty Group 2011), and individual threads can be coated with adhesive to make very delicate repairs (Landi 1992).

COATING TECHNIQUE

Adhesives are almost always applied and allowed to dry on the support before bonding to textile artifacts. The grain of woven fabrics must remain properly aligned because once the adhesive has dried it cannot be corrected. When applied by brush or roller to a sheer support over a non-stick casting substrate, the dilute adhesive solution shrinks to coat only the underside of the yarns as it dries. Increasing the adhesive solution concentration or applying multiple coats results in a heavier coating or a continuous film over the fabric (**Figure 4**) which, in turn, gives stronger bonds (Karsten 2003). The interaction of the adhesive solution with the casting substrate

TABLE 2. SUPPORT MATERIALS

Fabric	Advantages	Disadvantages
silk crepeline	- highest thread count—retains more adhesive - conforms well to textile	- irregular yarns—less uniform coating
polyester crepeline (Tetex TR, Stabilitex)	- uniform yarns and weave—uniform coating	- does not conform well to textile - stiffer laminate
nylon net (20 denier, Dukeries #N8000)	- conforms to textile better than lower quality net or polyester crepeline - flexible laminate	- monofilament, lowest thread count—retains less adhesive - possible imprint of net pattern onto textile - distorts when adhesive applied

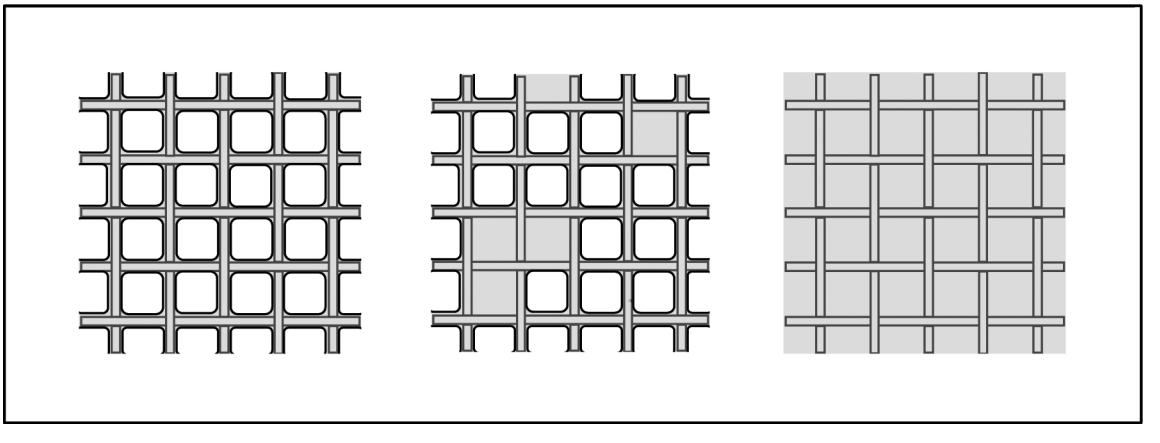

Figure 4. Effect of adhesive application on the continuity of the coating. The adhesive solution and the manner in which it is applied determines whether the adhesive coats only the yarns (left), fills some interstices (centre) or produces a continuous film over yarns and interstices (right).

(Teflon-coated glass cloth, polyethylene sheeting or polyester film) also affects the coating. Smoother substrates produce shinier coatings (Hillyer et al. 1997). Creases and bubbles in the substrate result in bare spots or uneven adhesive deposit. How easily the adhesive-coated fabric peels off the casting substrate is important for handling and varies depending on the adhesive and casting substrate. The goal is to produce a uniform coating of adhesive that covers only the yarns or both yarns and interstices (**Figure 4**, left and right).

Spray application onto a support fabric aligned and stretched on a stretcher frame is often used with resins like BEVA 371 dissolved in non-aqueous solvents. Several factors (e.g. adhesive concentration, pressure, distance from the sprayer to the support, solvent) affect the adhesive deposit. If still in a liquid phase when it reaches the support, the adhesive solution will coat both sides of the yarns. If the solvent evaporates before reaching the support, dry adhesive threads or particles form a "flocked" layer on one side of the support. Several spray coats are required. Templates can be used to mask off areas so that adhesive is not applied to corresponding areas of loss on the artifact.

Wet application of adhesive is sometimes used with starch and carbohydrate pastes (Masschelein-Kleiner 1980; Van Steen and Masschelein-Kleiner 1980; Thomsen 1984; Cruickshank and Tinker 1995; Kite and Webber 1995). The paste is applied to a paper or tissue, allowed to partially dry, and then applied to the pre-wet or humidified textile. Gentle tamping with a soft brush helps to mold the non-woven paper to the textile (Kite and Webber 1995). For this technique, collaboration with experienced paper conservators is helpful.

REACTIVATION TECHNIQUE

Heat and/or an appropriate solvent can be used to soften the adhesive sufficiently to permit bonding to a textile artifact. Bond strength depends on the amount of adhesive applied to the support, temperature or solvent chosen, reactivation time and pressure (Karsten and Down 2005). A variety of methods have been described: hand-held irons and vacuum hot tables for heat reactivation (Karsten 1998); misting with a portable compressor and nebulizer (Kiefer 2005), direct application or controlled delivery through a solvent-permeable barrier (e.g. blotting paper, GORE-TEX or Sympatex) (Medina and Rogerson 2006; Takami and Eastop 2002) for solvent reactivation; and cold-lining using humidification for reactivation of a carbohydrate paste (Cruickshank et al. 1999). Comparative testing using a mixture of Lascaux 360 HV and Lascaux 498 HV (1:2) has shown that solvent reactivation produces stronger bonds than heat reactivation under identical conditions of time and pressure (Karsten and Down 2005). Pre-testing is required to determine appropriate parameters since reactivation tools and techniques differ from lab to lab and the adhesive coating varies according to materials and techniques used. A simple method for comparing the bond strength of samples that uses clips and weights has been developed by Karsten and Down (2005) for labs that do not possess commercial mechanical testing machines.

Whatever the technique chosen, overlapping reactivated areas should be avoided. Applying the reactivating agent and pressure more than once creates unnecessarily strong bonds and could impregnate the textile with adhesive. In heat reactivation, constant movement of the spatula or iron is sometimes used to avoid this. Nevertheless, uniform bond strength over the entire surface of the textile is difficult to obtain.

AUXILIARY SUPPORTS

Adhering textile artifacts to sheer support fabrics affords stabilization and safe handling during subsequent attachment to another firmer auxiliary support fabric or mount. Firmer fabrics in auxiliary supports help prevent layers sticking together, provide an aesthetically pleasing fill for losses in the artifact and provide a solid support for stitching (Landi 1992). Supplementary stitching can contribute significantly to the durability of an adhesive treatment. Conversely, the adhesive treatment reduces the amount of stitching required for a treatment. Flat textiles immobilized on a rigid support can be treated with a weaker adhesive.

ADHESIVE REMOVAL FROM SUPPORT

The method for removing adhesive from exposed areas of the support should be considered when the adhesive is chosen. Adhesive exposed through losses in the textile may be shiny or attract dust; therefore, it is removed locally using solvents in conjunction with absorbent materials or suction devices. In some instances, exposed adhesive may be covered with a cosmetic infill.

RE-TREATMENT OF ADHERED TEXTILES

Adhesive-treated textiles may require re-treatment for a number of reasons; for example, the adhesive bond may be failing or the support material may be deteriorated and no longer providing adequate

support. The current condition of the textile as well as the adhesive treatment must be carefully assessed as it may be preferable to retain the original adhesive treatment and supplement it with stitching, where required, than to attempt to undo it (Tinker 2011).

If the adhesive treatment is to be reversed, trying to dissolve the adhesive in the bond may cause it to penetrate into the textile artifact. Therefore, swelling or softening the adhesive to permit mechanical removal is often preferable (Ballard 1987). This can be achieved using appropriate solvents or heat. Water can be effective for poly(vinyl acetate) dispersion adhesives and carbohydrate pastes. Mechanical action, such as peeling, is required to remove the support. In most instances, adhesive residues may be left behind. In cases where starch paste has impregnated a textile, enzyme removal may be required.

Basketry

by Ruth E. Norton, Field Museum of Natural History

Properties and condition of the object that influence adhesive choice

Basketry in general comprises objects made using interworked element construction where elements are more robust than those used in textiles. Construction methods include the knotted, looped, linked, twined and woven structures commonly found in baskets, mats, containers, costume, headdresses, hammocks and a wide range of utilitarian objects or composite objects (Florian et al. 1990; Seiler-Baldinger 1994; Emery 1966). Because they do not fit in with other object categories, barkcloth and objects made of non-woody plant materials will also be included here. Basketry composition, morphology, cultural context (including non-tangible qualities), pre- and post-collection use, and location and type of damage or degradation are highly variable (Florian et al. 1990; Mellor 1992; Bernstein 1992; Clavir 2002). General guidelines for adhesive applications can be made, but these variables must be understood in each specific application.

Basketry materials are frequently altered in manufacture and use through processing, such as soaking, scraping, pounding or cutting; application of decoration, such as dyeing, painting or oiling; and application of materials in cultural use, such as oils, blood or poisons. These alterations affect the morphology, structure and composition of the object and thus affect adhesive choice. The adhesive properties referred to below are detailed in the *Introduction* to this chapter. Density affects the weight on a join (adhesive strength) and penetration of adhesive (solvent choices, molecular weight). Surface textures, such as matte and gloss, affect adhesive ability. The epidermis, with its dense cellular layers and waxy cutin, has greater resistance to penetration and adhesion than surfaces comprising the more porous sub-epidermal morphology. The flexibility of the object material will dictate adhesive choice and application. The tightness of a construction (open versus close knotting, twining or weaving) will affect requirements for flexibility, and need and type of reinforcement. Solvent choice may be affected by the composition of the plant material, such as the presence of extractives like resin, wax, oil, tannins or starch, and by the composition of culturally applied materials. The location of a break or weakness in an object often affects what tensile and shear stresses must be met by an adhesive and thus determines cohesive and adhesive strength requirements.

Adhesive properties considered

The properties discussed in the *Introduction* to this chapter apply and should be considered in adhesive choices for basketry. Other specific notes follow.

Adhesive solubility in application and removal

The solvent used for the application or removal of an adhesive should not degrade, alter or cause migration of natural extractives, such as oils, waxes, resins or tannin, or culturally applied materials, such as oils, paints, waxes or poisons in the basketry.

Rate of solvent evaporation

The rate of solvent evaporation affects migration and alteration of the materials mentioned above. The speed of adhesive setting may be a factor where the application of pressure may be limited by object shape and location of damage.

Shrinkage on setting

Plant materials and basketry objects are composite or fibrous structures, so surface layers, fibres and applied decoration can be pulled away by an adhesive that is too heavily applied for the specific situation. A more dilute solution, thinner applications, slower drying or a heat- or solvent-reactivated film format could reduce the shrinkage tension of an adhesive.

Reversibility

The reversibility of an adhesive is a standard goal in conservation. In practice, the degree of removal of an adhesive may be influenced by factors such as the location (object shape or location of damage) and degree of penetration of the adhesive (porosity of material or surface tension of solvent).

Cohesive strength

The tensile or shear stress on a join through gravity or construction technique must be matched by the internal strength of an adhesive.

Adhesive strength

Ideally, the cohesive and/or bond strength of the adhesive should be slightly weaker than the internal strength of the affected area of the object material so that, if stress is placed on a mend, the adhesive or the bond will fail before the object material. In practice, this information is not always available. Adhesive properties may be fairly well known, but the properties dictated by basketry construction techniques and material morphology may be impossible to quantify.

Flexibility

The flexibility of the adhesive should match the object's use in a collection. Will it be static, requiring no movement in handling or need to open, or must it be flexible, such as a rolled barkcloth that requires viewing or a container that needs to be opened?

Gloss

The final sheen of the adhesive must match that of the object, from matte to satiny to shiny. While the sheen is determined by the adhesive type and thickness, it will also be affected by object material character and may be shifted by the application techniques used (e.g. solvent choice, drying atmosphere, spraying versus brush technique, or mixing with other adhesives or matting agents).

Adhesives frequently used

Adhesives and consolidants typically used for basketry are given in **Table 1**.

Starch paste

Starch pastes are frequently used in joining breaks, adhering local or overall threads, backings or facings, and adhering loss compensation (Florian et al. 1990; Smith and Winkelbauer 2006; Anastassiades 2001; Norton 1984; Turchan 1986; Agrawal 1984; Elliot and Harding 1987; Kite 1995; Thomsen 1976). They are also occasionally used in consolidation and gap-filling (Kite 1995; Florian et al. 1990; Hatchfield and Marincola 1994). They provide strong bonds to organic material, particularly those of a fibrous or porous texture. At low concentrations, the adhesive dries matte and patches and linings made with it retain the flexibility of the object, yet thick starch pastes can bond to and

Table 1. Typical adhesive types used for basketry conservation

Adhesives frequently used	Type of treatment
starch paste	breaks local or overall threads, backings or facings loss compensation consolidation
cellulose ether	breaks local or overall backings or facings loss compensation consolidation gap-filling
cellulose ether film for reactivation	local or overall backings or facings
animal glue	consolidation
dispersion	breaks local or overall backings or facings loss compensation consolidation
dispersion film for heat or solvent reactivation	lap or scarf joins local or overall backings or facings
resin in solution	breaks local or overall backings or facings loss compensation gap-filling
adhesive mixtures	breaks local or overall backings or facings loss compensation

hold thicker patches and stiff materials. Key to the usefulness of starch pastes is their water base, which allows for plastic modeling of backing paper and thread, allowing reinforcing materials to be tamped in and molded to the fine shape and texture of the object. This is important for good adhesion and also for good visual integration, especially if reinforcing materials are dyed or toned to match the object (Norton 2003). Wheat and rice starch are the most frequently used.

Reinforcing materials, which may range from very small patches to full linings, are the bridging materials that carry the adhesion from one surface area to another. Materials include long-fibred Japanese paper (**Figure 1**), cotton thread (Florian et al. 1990) (**Figure 2**), twisted-paper thread (Odegaard and Harvey 1984; Dignard and Schlichting 1991; Peacock 1991) (**Figure 3**) and silk crepeline.

CELLULOSE ETHER

Based on stability research (Feller and Wilt 1990), methylcellulose (MC) is, in general, the more stable of the cellulose ethers (CEs) and would be the preferred choice for use with basketry materials. For joining breaks, adhering local or overall backings or facings, and adhering loss compensation, CEs are useful primarily where somewhat weak adhesion is sought and tensile and shear stress is low. For example, when joining overlapping layers of degraded material, such as textile, papyrus or barkcloth, where contact but little stress is maintained, MC applied as a small clump of fairly dry paste (2–4% w/w in water) offers sufficient strength and can be reversed mechanically with a metal or Teflon micro-spatula. The structure of a join mend is critical to adhesive success. Lap or scarf joins are appropriate for the use of CEs. The high stress placed on the object and adhesive at unreinforced butt joins often results in failure of the join and further damage to the object element ends. For examples of MC use in mending

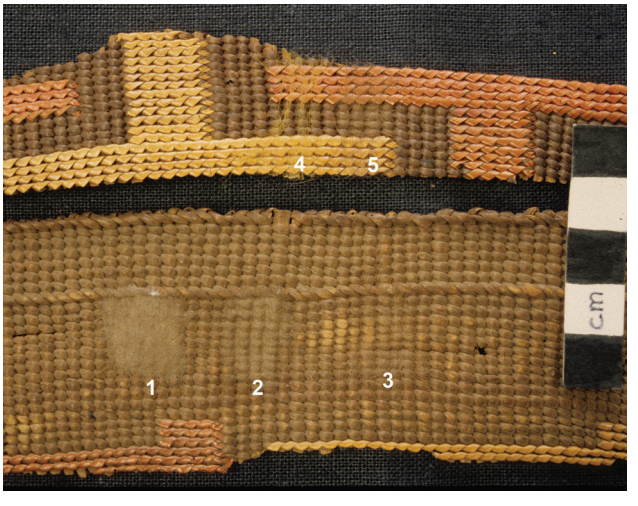

Figure 1. Examples of patch mends using Japanese paper and wheat starch paste adhered to fragments of a Native American, Northwest Coast basket (study collection, Buffalo State College).The thicker paper is Papier Nao RK17 kozo fibre paper. The thinner paper is Papier Nao RK1 kozo fibre paper. Papers are dyed using Procion Fiber Reactive dyes. Bottom fragment: (1) RK17 dyed brown laid on surface and dried. (2) RK17 dyed brown laid on surface and tamped in with the tip of a flat oil painting brush. This process illustrates the importance of molding the paper to the basket surface to attain best strength and appearance. (3) RK1 dyed brown covers the right half of an area of abrasion. This process illustrates the transparency of a patch using thin paper. Top fragment: Patches covering red, brown and yellow areas of basket fragment. When patch covers an area of more than one colour (as in patches 4 and 5 covering the yellow, brown and red bands of basketry), paper dyed to match the medium value colour is less obtrusive. (4) RK1 dyed yellow. (5) RK1 dyed red-brown.

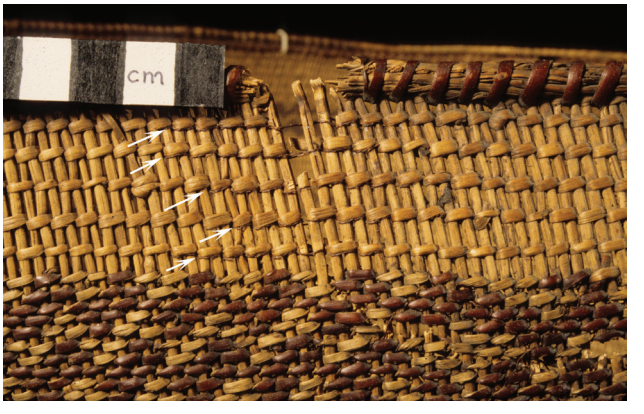

Figure 2. Example of mending a vertical break using cotton thread on a basket from California (Buffalo and Erie County History Society, 64.1046r). Mends made with cotton embroidery floss, soaked in wheat starch paste, tamped into groove below horizontal elements.

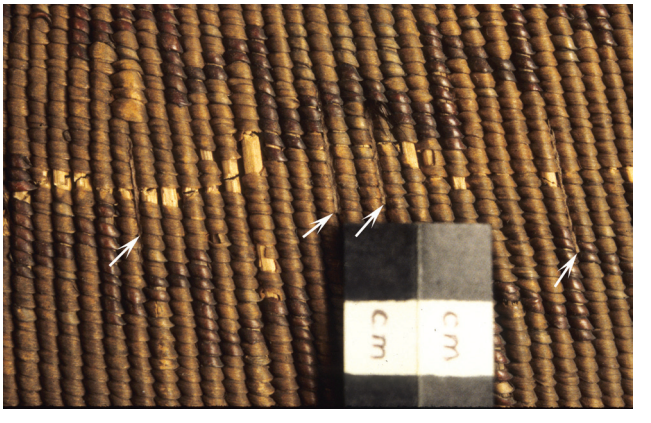

Figure 3. Example of mending a horizontal break using a thread of twisted dyed paper on a California Pomo basket (Buffalo and Erie County History Society). Mends made with strips of dyed Japanese paper soaked in wheat starch paste, rolled and pressed into groove between vertical elements.

Figure 4. Examples of conditions where consolidation with a cellulose ether would be appropriate on an Australian Aboriginal wiranua (National Museum of Australia, AIA ADC108). Image of a container made of eucalyptus paper bark. The outer bark is lashed together and resin covers the seams. Separation of layers in outer bark results in flaking off of thin layers. Outer bark consolidated by introducing methylcellulose (MC) paste between layers and gently pressing together with fingers. Crumbling resin consolidated by introducing very thin MC into cracks with brush tip and allowing capillary action to draw in the consolidant.

treatments, see Takagi et al. (2006), Liow et al. (2005) and Suryawanshi (2000).

Because CEs dry matte at the low concentrations needed, they are very useful for the consolidation of degraded plant materials and matte paints (**Figure 4**). Very low concentrations (0.5–1.0% w/w in water) of MC (Methocel A4C) are used for consolidation of fine particle material. Higher concentrations (2–10% w/w in water) are necessary for consolidation of more coarse particle or fibre materials (Florian et al. 1990; Tworek-Matuszkiewicz 1987). Dilution with ethanol or pretreatment with ethanol/water are usually required to counter the high surface tension of the original water solution. Hydroxypropylcellulose has been used for consolidation (Allington 1986; Firnhaber and Erhardt 1986).

The matte texture and water-base nature of CEs make them useful in gap-filling as the binder for paper, paper pulp, fibre, sawdust and fumed silica (Hatchfield and Marincola 1994; Florian et al. 1990; Sah 2002). The very high viscosity of the putty and the high surface tension of the water help confine the fill to the gap, avoiding the staining common with other fills. Very strong under compression, MC bound fills have held up well under significant force. Shrinkage during drying makes it necessary to fill incrementally.

Cellulose ether film for reactivation

CE films, usually on very thin Japanese paper (7–10 g/m^2 weight), are useful for lining thin, fragile objects like deteriorated fine barkcloth, papyrus, woven banana and *Phormium* fibre cloth. Hydroxypropylcellulose (e.g. Klucel E or Klucel G) film can be reactivated with ethanol applied by vapour chamber, spray or brush. For examples of other CEs used in film form, see Cruickshank and Morgan (1993). The thin films allow flexibility and transparency to patches and linings, and the non-tacky film allows alignment of object fragments before and during reactivation.

Animal glue

Animal glue, such as gelatin, is sometimes used in the fairly rare occasions when high adhesive strength is needed and the solvent used for application and removal must be water (e.g. when adhesives and consolidants are applied to resin or wax). The increase in object moisture content of plant materials limits the use of water for aged adhesive removal. Isinglass in very dilute form (0.5–1.0% w/w in water) is useful for consolidation of thin films, such as matte paint and friable plant material, where other consolidants leave tide lines or where non-aqueous solvents cause staining through migration of extractives. Because of its high surface tension, MC in water must often be diluted with ethanol to achieve penetration, while the much lower surface tension of isinglass solutions can penetrate by capillary action without dilution or pre-treatment with alcohol-water solutions.

Dispersion

Acrylic dispersions are used in basketry conservation for joining breaks, adhering local or overall backings or facings, and adhering loss compensation (Fenn 1980; Gilberg and Grant 1983–4; Barton 1986). For rigid joins where hardness and high cohesive strength (no stretching under tension) is needed, adhesive dispersions made with polymers of higher glass transition temperature (T_g) (e.g. Rhoplex B-60A, Rhoplex AC-33) are employed. Where flexibility is needed and lower cohesive strength can be tolerated, adhesive dispersions made with polymers of lower T_g (e.g. Rhoplex AC-234, Plextol B500) are used. Dispersion use is somewhat restricted by high gloss on setting, although this can be reduced by matting agents like fumed silica. Given the highly diverse

materials found in basketry, the use of dispersions is also restricted by the need for object tolerance to aqueous solvents on application and non-aqueous solvents for removal.

Dispersions are also used for consolidation of matte and satin-gloss paint, and degraded plant materials. As these applications have low reversibility, it is critical that the dispersions have high long-term stability (Horton-James et al. 1991).

DISPERSION FILM FOR HEAT OR SOLVENT REACTIVATION

Films, particularly reinforced films, are useful in adhering lap or scarf joins. They are also useful as local patches or overall backings or facings where positioning of components and clamping or weighting is difficult, where exposure to wet systems is problematic, and where little penetration of the adhesive is sought.

Dispersions made with low T_g polymers (e.g. Rhoplex AC-234, Plextol B500, Rhoplex ML-200) are needed to allow timely reactivation by solvents and reactivation by heat at low temperatures. The object tolerances to liquid or vapour solvent, or mild heat are selection factors. As one goal of the technique is limited exposure to solvents, dispersions that will reactivate quickly in fast evaporating solvents are used. Films that comprise the adhesive alone are useful in very small areas with lap or scarf joins, but are difficult to manipulate in larger areas or spots with complex positioning. Reinforced films offer greater ease in positioning and also offer strength across breaks and areas of weakness. Dry film is cut to size, positioned and reactivated. For solvent reactivation, solvent may be applied in liquid or vapour form. In liquid form, it is wicked into the area by capillary action from brush or capillary pipette. In vapour form, the object is exposed in a full vapour chamber or local vapour tent. Heat reactivation is achieved by a tacking iron or a hot air tool (e.g. Leister Labor Tool). For other examples, see Robson (1986) and Barton (1986).

Reinforcing materials include silk crepeline, polyester gauze (e.g. Tetex), Japanese paper (for flexibility and translucency, a fine paper is preferred) and nylon gossamer.

RESIN IN SOLUTION

Resins in non-aqueous solution are used for joining breaks, adhering local or overall backings or facings, adhering loss compensation and gap-filling, and, to a lesser extent, consolidating and gap-filling (Kronkright 1981; Gilberg and Grant 1983–4; Liow et al. 2005). Advantages are the range of solubility and rates of solvent evaporation available, and the non-swelling of hydrophilic materials like paper, inner bark, split leaves and wood splints, commonly found in basketry. Disadvantages include the plasticization and migration of materials such as extractives (e.g. resin, wax, cutin, oil) and the absorption of solvents by many types of plastic, causing dissolution and stress cracking. Because of its outstanding aging properties, Paraloid B-72 is a resin of choice. Its solubility range in alcohols, ketones and aromatic hydrocarbons is an advantage when treating objects of diverse composition. Recently, resins like Aquazol have come into use.

Although effective consolidants, resin solution use on relatively porous basketry and related materials is somewhat limited. Thin, powdery surfaces can be consolidated without becoming darkened and glossy if a very slow evaporating solvent is used (Welsh 1980). Thicker, more coarsely friable surfaces become dark before consolidation can be achieved. Consolidation of solid materials, such as wood or bamboo attacked by powder post beetle or microbes, is effective, but the low surface tension of most solutions makes it difficult to control penetration and often results in disfiguring darkening of adjacent areas.

Acrylic and poly(vinyl acetate) resins have been used in a film for heat and solvent reactivation (Howatt-Krahn 1986; Himmelstein and Appelbaum 1977). However, they require

Figure 5. Example of use of a reinforced heat-reactivated resin on a mask from Papua New Guinea (National Museum of Australia, AIA MRM26). Image shows a layer of coconut leaf sheath over a rattan framework. Leaf sheath is extremely brittle with losses that threaten structural stability. Area outlined in white is lined with brown Stabilitex coated with BEVA 371 resin. Lining is positioned and then adhered by reactivation with a tacking iron.

a higher reactivation temperature than the dispersion films now in use. BEVA 371 Film is also used for heat (**Figure 5**) and solvent reactivation (Drummond 1994). Applying resins to a fine textile or paper to create a reinforced film increases the film strength and makes it easier to position on the basketry item.

ADHESIVE MIXTURES

Starch and CEs are sometimes mixed with acrylic dispersions to gain the adhesive strength and flexibility of the acrylic and the quick water reversibility of starch and CE. These properties are especially important when joining breaks or applying patches or linings to extremely smooth surfaces, or where the object has been oiled. Combinations include wheat starch paste and Rhoplex ML-200, or Methocel A4M and Rhoplex AC-234 (50:50 common, 25:75 or 75:25). For examples, see Firnhaber and Erhardt (1986).

Paintings

by Wendy Baker, Canadian Conservation Institute

Virtually all treatments that address damage to paintings require one or more interventions with adhesives. These materials re-establish bonds by stabilizing cracking, flaking and cleavage in and between paint and ground layers, and by reattaching these layers to underlying supports. Consolidants can also prevent erosion of a powdery paint surface by reinforcing or re-establishing the pigment-medium bond. Adhesives hold the edges of tears together in canvas paintings, attach linings to original canvases and reinforce degraded supports, as required. Adhesives bond facing tissues to damaged painted surfaces before and during treatments, and serve as binding media for fills and inpainting. The choice of which adhesive to use is based on many parameters. To begin, the conservator will choose a category of adhesives that addresses a particular condition. Next, the conservator will ascertain sensitivities of the object to be treated and its compatibility with the adhesive. The final choice of adhesive(s) will be informed by a number of variables, not the least of which will include potential for removal or reactivation of the adhesive, preference with regard to working properties, ease of preparation and application, length of work time, toxicity, versatility and availability.

This section offers some practical suggestions for the choice and application of a number of adhesives and consolidants normally used to treat paintings on canvas and that address delaminations between and among layers, facings, structural repair (lining) and tear repair.

First considerations

Extent of treatment

As a first step, it is vital to establish the nature and cause of damages and then to ascertain the conditions to which the painting will return. If conditions are ideal [e.g. stable relative humidity (RH)], then treatment can focus on specific damages, such as tear repair or adhesion of localized flaking. Non-adhesive blind or stretcher bar linings can provide additional support against vibration or buffer against moderate changes to ambient RH. The underlying goal in any treatment is to keep intervention and the addition of new materials to a minimum. If the damages are extensive or the painting is to return to an uncontrolled environment, then a more extensive and interventive treatment may be required.

Reversibility

Every layer in a painting structure, once cracked, allows for the passage of liquids. When dispersed into the painting system, the adhesive will be bound within the paint structure and will be difficult, if not impossible, to remove. This lack of reversibility underlines the need for careful consideration of materials and treatment procedures; for instance, adhesives must have superior aging properties, such as retention of neutral pH, and all procedures should aim at minimal intervention (e.g. nap-bond versus impregnating lining adhesive). Where an adhesive cannot be removed, it should, at least, retain the potential for reactivation.

Change in saturation

The use of some adhesives, in particular situations, will result in a change in saturation to paint, ground and/or support layers. This is especially the case for powdery paints or exposed canvas where the consolidant penetrates into design or support layers and significantly alters the saturation of the material.

Water, solvents, heat and pressure

Adhesives normally used for the conservation of paintings are dissolved or suspended in either water or a solvent, and often, but not always, require heat to complete activation. Water, solvents and heat can cause irreversible damage if applied inappropriately, and damage can be exacerbated when pressure is added during the drying or setting phase of an adhesive treatment. The painting's response and potential sensitivities must be known prior to selecting the treatment processes and adhesive(s). For instance, solvent-based adhesives cannot be used for the treatment of acrylic paintings, nor can these same adhesives be considered for many newer oil paints where full polymerization of the oil is not complete, or for oil paintings containing blends of resins and other solvent-sensitive additives. Very lean oil paints can also be sensitive to water, as are glue-based (distemper) paints. Glue grounds, as opposed to oil grounds, will be sensitive to

water-based adhesives. Knowledge of the painting materials is critical as this can prevent inappropriate treatment and adhesive use.

Conditions and choice of materials

Delamination between layers

There are a number of adhesives used to re-adhere delamination between or among layers in a painting structure. The following represent the most common, but by no means all, of the available adhesives.

Adhesives for re-adhering paint and ground layers

Collagen or protein-based adhesives classed as gelatins, such as rabbit skin glue, parchment size, fish glue and isinglass, are commonly used for the securing of detached paint and ground layers because of their superior flow and tack, and compatibility with similar materials in traditional easel paintings. *Junfunori*, an extract of the red algae *Gloiopeltis furcata*, has specialized uses as an adhesive for securing painting layers, as do the cellulose derivatives Klucel E and Klucel G (hydroxypropylcelluloses). Both *junfunori* and the Klucels can be used where paint layers are matte and sensitive to discolouration from adhesive infusion. Other synthetic adhesives are also deployed to treat detached or delaminating paint and ground layers on various supports. These include, but are not exclusive to:

- vinyl acetate/di-butyl maleate copolymer dispersions (Mowilith DM427);
- poly(vinyl acetate) (PVAC) resins in solution (Mowilith 30, Mowilith 50, Mowilith 60);
- vinyl acetate/ethylene copolymer (VAE) dispersions (Jade 403, Jade R, BEVA D8);
- ethylene/vinyl acetate copolymer-based BEVA 371;
- acrylic dispersions (Rhoplex AC-33, Rhoplex AC-73, Rhoplex AC-234, Plextol B500);
- acrylic resins in solution (Paraloid B-72, Plexisol P550); and
- poly(2-ethyl-2-oxazoline) (Aquazol).

An effective adhesive should have adjustable or low viscosity, good wettability or fluid properties, tack, long-term flexibility, long-term neutral or near-neutral pH, and sufficient bond strength to address potential stresses. Where an adhesive must secure cracked and cupped paint and/or paint and ground layers, bond strength of the adhesive needs to be high to counteract the stress placed on it by potential movements in these and adjacent layers. Counteracting ongoing stress in a painting structure is beyond the strength of many adhesives, especially when the painting support is a hygroscopic material such as canvas. Often, adhesive failure will occur unless the painting remains in ideal conditions (Michalski 2008, p. 37).

Required properties of adhesives used to secure paint and ground layers

Bond strength and flow

PVAC dispersions and some acrylic dispersions generally have large particle size and this, combined with the presence of stabilizers and emulsifiers (acrylic dispersions generally have less stabilizers and emulsifiers than PVAC dispersions), can make for very poor penetration into and through cracked paint structures (Koob 1981, p. 86). These adhesives can offer strong bonding of detached paint or ground if the area to be secured is exposed, for instance, in the case of a fully detached chip of paint (**Figure 1**).

Many adhesives, when used at sufficient concentrations to provide adequate bond strength [e.g. Paraloid B-72 (20% in either toluene or ethanol)], are too viscous to flow well through small gaps and cracks in paint and ground layers. Gelatins, traditionally the adhesive of choice to re-adhere delaminating paint to ground layers and ground layers to wood supports, can provide very high tack and bond strength at concentrations of 5–7%,

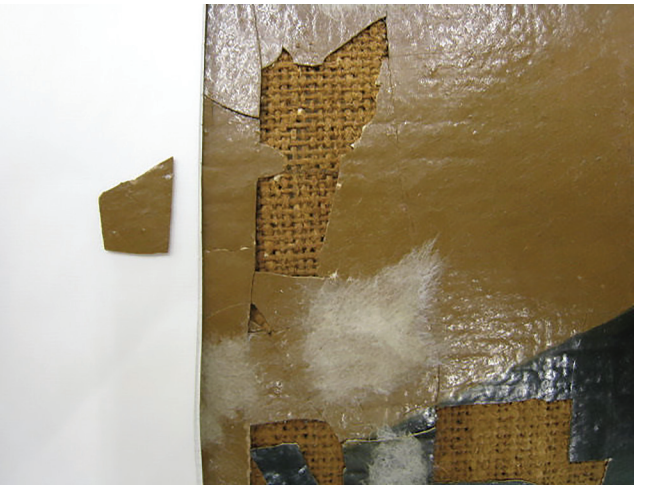

Figure 1. Completely detached paint chip. Placement requires a strong adhesive such as a vinyl acetate/ethylene copolymer dispersion (e.g. Jade 403, BEVA D8) over the entire surface. This is a reasonable choice for an adhesive provided that the placement of the chip is accurate and that the canvas has not shrunk, which would compromise the fit. Paraloid B-72 or BEVA 371 could be substituted; however, in these cases, the solvent content could damage the brown overpaint layer. The use of a low viscosity adhesive with high wettability (e.g. gelatin) would result in rapid absorption into the canvas, with insufficient adhesive remaining to guarantee a good bond.

and in the case of isinglass (sturgeon glue), at even lower concentrations. This high tack combined with good flow through paint structures, especially if the gelatin is kept warm during application, makes these adhesives good choices for reattaching flaking paint and ground layers. BEVA D8 dispersion has good fluid properties due, in part, to the wetting capacity of surfactants used to stabilize the polymer.

BEVA D8 can penetrate, unlike many PVAC dispersions, through larger cracks in paint and ground layers, resulting in strong bonds at full concentration or at mild dilution (e.g. BEVA D8/water 4:1) (**Figures 2a** and **b**). Rhoplex AC-234 and Plextol B500 are often employed to secure flaking paint to absorbent wood substrates with resulting strong bonds (**Figures 3a** and **b**).

BEVA 371 solution, designed as a multi-purpose adhesive, can be used diluted to re-adhere delaminations in paint layers. Its flow characteristics are improved by warming the solution (**Figure 4**).

Adhesive flexibility

Many of the synthetic adhesives, such as BEVA 371, BEVA D8, Jade R, Mowilith DM427, Plextol B500, Klucel E, etc., form flexible films that allow for a degree of movement in the paint and ground layers

Figure 2a. BEVA D8 is injected, at full strength, through facing tissue to re-adhere badly flaking paint to a plaster ceiling.

Figure 2b. Flaking and curled paint has relaxed through a combination of humidification and light pressure after consolidation with BEVA D8. Facings applied with water only are easily removed from the treated surface.

Figure 3a. Flaking oil paint and oil-based ground layers on a hardboard panel are consolidated with Rhoplex AC-234.

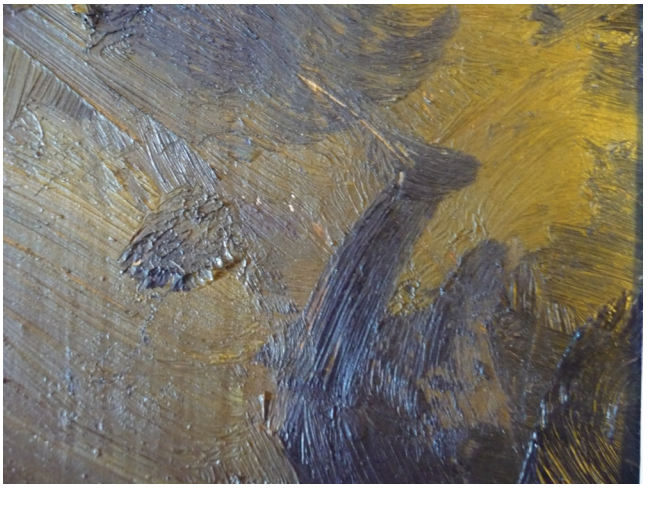

Figure 3b. Area of flaking after treatment with Rhoplex AC-234 and mild heating with tacking iron through facing tissue.

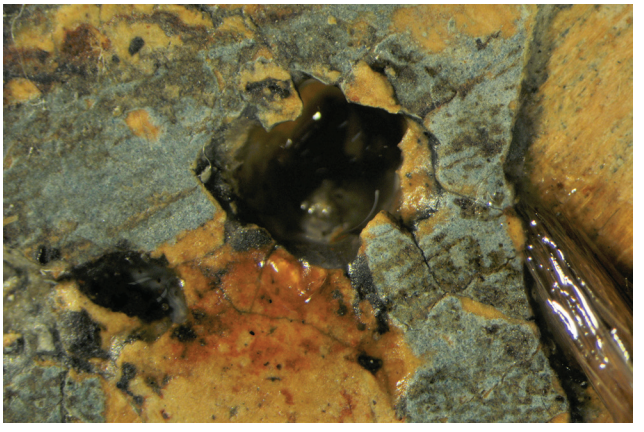

Figure 4. BEVA 371 (1:1 with toluene) is applied by brush to reattach paint and ground layers that have detached from a wood support. BEVA 371 was chosen for several reasons, mainly because the thick ground layers are water sensitive and cannot sustain wetting with a water-based adhesive, and BEVA 371 is compatible with the wax infused into the wood from a previous treatment.

without compromise to the adhesive bond. Low molecular weight polymers in adhesives provide for a lower viscosity with better penetration, but have a tendency over time to form brittle films, which are counterproductive to long-term bond stability. Aquazol 50, despite its low molecular weight, remains flexible upon drying because it can readily absorb moisture. Although it has low tack, Aquazol 50 can be used to plasticize other more brittle adhesives, both aqueous and solvent-based, such as gelatins, Paraloid B-72, etc. (Wolbers 2008, p. 115).

Impediments to good adhesion

When applied to porous and complex layers in a painting, some adhesive solids can separate out of solution, be adsorbed, flow away from the bond site, or be displaced by reverse migration during drying (Michalski 2008). Some of these effects are described below.

Adsorption

If an adhesive is adsorbed before reaching the delamination zone, the bond will be starved. This is especially the case for paints with low binder-to-pigment volume where the exposed pigment particles can attract adhesive from the solvent. To decrease the risk of this premature adsorption of the adhesive, especially if the adhesive solvent is not strongly eluting for that adhesive (meaning that the solvent is capable of stripping adsorbed adhesive from the pigment) (Michalski and Dignard 1997, p. 123), either the solvent can be changed for one with a stronger attraction for that adhesive, or a blend of polar solvents (e.g. ethanol/water 1:1) can be introduced into the crack or at the edge of the flake prior to adhesive application. These polar solvents will be preferentially adsorbed, allowing the adhesive, when introduced, to pass by and be deposited where it is needed in the delamination zone.

Capillary competition

Smaller pores exert a stronger capillary pull than larger pores and cracks or gaps, and will pull adhesive away from the bond. A lack of adhesive or joint starvation at a bond site can be overcome by introducing adhesive in successive applications or in increasing concentrations. The initial applications will be readily taken up by the smaller capillaries and pores, allowing for subsequent and/or more concentrated applications to remain where these are required. The problem of joint starvation is mitigated through the use of gelatins. These adhesives are not prone to migration by capillary action as, for the most part, they gel upon reaching room temperature (see *Advantages and disadvantages of glue*).

Reverse migration

Reverse migration of an adhesive, especially solvent-based adhesives, can occur as the solvent phase evaporates. Adhesive will migrate within the painting depending on the location of the evaporation front. In situations where consolidation of non-superficial flaking is necessary, such as between the ground, multiple paint layers (including impasto layers) and the support, reverse migration may prevent the adhesive from remaining where it is required. One method of slowing migration is to reduce evaporation rates by draping the painting, usually the front, with an impermeable membrane after adhesive application (Hansen et al. 1993).

Advantages and disadvantages of glues

The ongoing use of traditional collagen adhesives, or glues, for the treatment of paint/ground layer cleavage in paintings can be explained by a number of factors. Collagen adhesives like gelatin are readily available, easy to prepare, non-toxic, have good fluid properties, provide very good bond strength, and can be reactivated by warming. More importantly, many collagen glues gel as they cool and are not displaced from areas of delamination due to capillary loss or reverse migration (Michalski 2008, p. 36). There is an advantage to choosing adhesive materials similar to those used in the production of the painting, such as glue used for sizing canvases. If introduced in the correct concentration, the behaviour of the "repair" gelatins will neither interfere locally with the response of similar, original materials to environmental stimuli nor will they impose a new set of behaviours. Animal-based glues can be exploited for their distinct

characteristics; for instance, rabbit skin glue's strong contractive strength can draw flaking paint back onto the substrate. Isinglass, on the other hand, is valued for its greater tack and lower viscosity than rabbit skin glue, and is frequently the choice of adhesive for the restoration of traditional icon paintings (Petukhova and Bonadies 1993). Glues can also be reactivated at much lower temperatures than many of the synthetic thermoplastic adhesives. The major drawback to glues is their brittleness and the contractive force that they exert at low humidity.

ADVANTAGES AND DISADVANTAGES OF HEAT-SENSITIVE GELS AND OTHER THERMOPLASTIC ADHESIVES

Adhesives that can be reactivated by heat or solvents have a distinct advantage over those that cannot. For instance, a safe, cohesive deconstruction of these joins can occur if the need arises for more intervention or if the bond fails (Horie 2010, p. 111). The successful treatment of flaking, cracked and cupped paint often involves exposure to low levels of heat, pressure, humidity and sometimes solvents to relax deformed layers. Adhesives that are responsive to these elements will allow for some additional movement in the delaminated areas, resulting in better adhesive bonds. Heat activation temperatures vary. Synthetic resins soften at a much higher temperatures than glues (65–68°C for BEVA 371, as opposed to 40–45°C for most animal glues). Thermoplastic adhesives based on solutions of resin in organic solvents can only be used on fully polymerized oil paint or tempera surfaces where a combination of solvent exposure followed by heating will not disturb the paint medium.

CONSOLIDATION OF LEAN PAINT

Flaking and interlayer cleavage in lean paints can be very difficult to remedy as the adhesive will double as a medium and can darken or impart gloss to the paint. Adhesives traditionally used in low concentration for lean paint include Klucel E and Klucel G (**Figure 5**) and carboxymethylcellulose (CMC). *Junfunori* has been very successful in consolidating matte paint, giving better results than either of the Klucels or CMC (Geiger and Michel 2005). Unfortunately, cost and availability can often restrict the use of this adhesive.

CONSOLIDATION OF POWDERY PAINT

Powdery paint absorbs any substance applied to its surface with the potential for darkening of the surface if too much or too high a concentration of consolidant is used. As well as delivering too much product, mechanical means of applying adhesives (e.g. brushing) will result in loss of friable pigments.

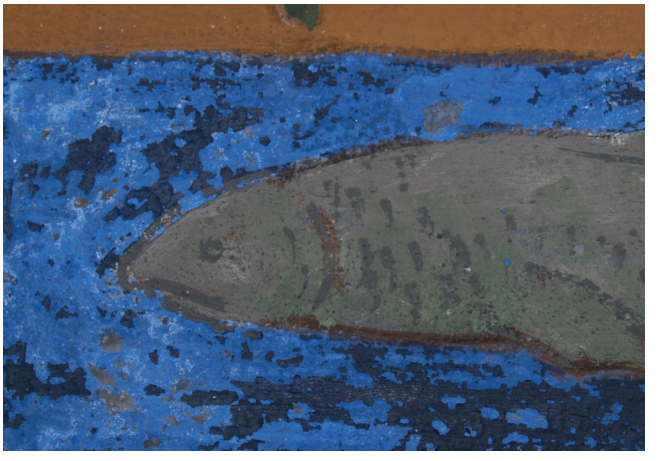

Figure 5. Consolidation of lean paint. Dark blue oil overpaint and underlying fresco secco (matte, water-sensitive tempera) are flaking from an underlying, original fresco layer. Klucel E (2% w/v in isopropanol) was used to secure the tempera layer to the frescoed plaster without darkening the tempera layer.

Minimal amounts of consolidant should be employed, yet, in the case of powdery paint, a sufficient amount must be used to effect a mechanical connection between particles (Horie 2010, p. 107). Ultrasonic misting of adhesives and glues provides an effective delivery system (Michalski et al. 1998). Weak solutions of gelatin (0.5–1% in water), CMC (0.5–1% in water) or Paraloid B-72 (0.5–1% in ethanol), have been successfully deployed by ultrasonic mister. More consolidant can be deposited on the surface of the paint by repeated misting, although the weak solutions are capable of providing sufficient bond strength. Aquazol 5 or 50 can be successfully used as a consolidant for powdery paint via the ultrasonic mister because the small size of the molecules allows for nebulizing at greater concentrations. Although not a high tack adhesive, Aquazol has more than enough bond strength to stabilize powdery paint (**Figure 6**).

CONSOLIDATION OF PAINT LAYERS ON NON-POROUS SURFACES

Non-porous, normally hard support surfaces present specific challenges for correction of problems of adhesion. These problems include difficulty in introducing the adhesive, potentially reduced capillary flow, and necessary drying of solvent or moisture exclusively through the paint/ground layers. Often, delamination of paint and ground layers from these surfaces is common as there is usually no "keying in" (mechanical bond) possible. Flaking paint from hardboard often occurs when it is painted on the smooth side, with large-scale delamination as a result (**Figures 3a** and **b**). Flaking paint is a common

Figure 6. Consolidation of powdery paint. Misting Aquazol 50 (5% w/v in water) onto a friable and powdery paint surface.

problem on glass supports and, as the introduced adhesive is located between paint and glass, it is visible. The adhesive must be water clear, wet the glass and flow over the whole area of delamination, leaving neither gaps nor air bubbles (**Figures 7a** and **b**). Normally, Paraloid B-72 (7% in toluene) is the adhesive of choice. The treatment of flaking paint on corrosion-prone supports, such as copper, is also problematic. Water-based adhesives do not flow well on metal and can provoke corrosion. Reactivation of thermoplastic resins, such as BEVA 371, increases the risk of further delamination of the paint layer. Testing shows that best results are obtained using Paraloid B-72 and Paraloid B-48N (both in toluene), Mowilith 20, Plexisol P550 and Bedacryl 122X (Horovitz 1996).

CONSOLIDATION THROUGH THE REVERSE OF THE CANVAS

An adhesive or consolidant can be applied through the reverse of a painting (on the canvas side). This will result in an intensive consolidation, or impregnation, and should only be done in situations where delamination is difficult to access from the recto, where there is overall cleavage or crumbling of layers, or where canvas consolidation is required. Treatments involve brush applications of dilute adhesives such as Plexisol P550 or BEVA 371 onto the back of the canvas. Wax and wax resin mixtures can effectively

Figure 7a. Reverse painting on glass before cleaning and consolidation. Delamination of paint from the glass can be seen in the figure's face, collar and in parts of the bonnet.

Figure 7b. After cleaning and consolidation. Paraloid B-72 (7% w/v in toluene) was used to re-adhere paint to the glass surface.

Image of Ingeborg Nissen, born in Engelstoft, Denmark, 1768–1862. Great-great grandmother of Birgitte Rump (owner). Reprinted with permission from Birgitte Rump.

bond (or embed) crumbling paint. However, their use as impregnating adhesives remains controversial due, in large part, to a history of paintings visually and physically altered by these treatments.

APPLICATION METHODS FOR RE-ADHESION OF PAINT AND GROUND LAYERS

Adhesives can be applied by brush, by syringe or by misting with a nebulizing device. Penetration of adhesives can be enhanced through the use of a localized low-pressure suction device. This can be placed behind the painting without removing it from an auxiliary support. Spot suction draws the adhesive down and into cracks in the paint and ground layers. Heat-sensitive glues and thermoplastic resins or waxes can be reactivated by localized treatment with a heated spatula (**Figure 8**), on a vacuum hot table using a built-in or auxiliary heat source (**Figure 9**), or on a multi-purpose low pressure table that can provide simultaneous clamping, heat and moisture. Where painting components are solvent-sensitive, if the adhesive is water-based, localized weights can be used to hold paint and ground layers in place during a consolidation treatment. In this case, blotters must be employed between weights and the paint layer to prevent the build-up of moisture, which can result in damage to the paint. Where the adhesive has a solvent phase, restricting the release of the solvent by localized weighting can result in severe damage to solvent-sensitive paints. Consolidation, in these cases, should be carried out only through heat reactivation of the adhesive once all of the solvent phase has evaporated from the adhesive.

Figure 8. Application of heat and pressure. Reactivation of heat-sensitive glue, as is the case here, or of thermoplastic resin adhesives using a heated spatula is always carried out using a barrier, such as this paper facing, in order to avoid damaging the paint layer from direct contact with heat and a polishing surface, such as a metal spatula.

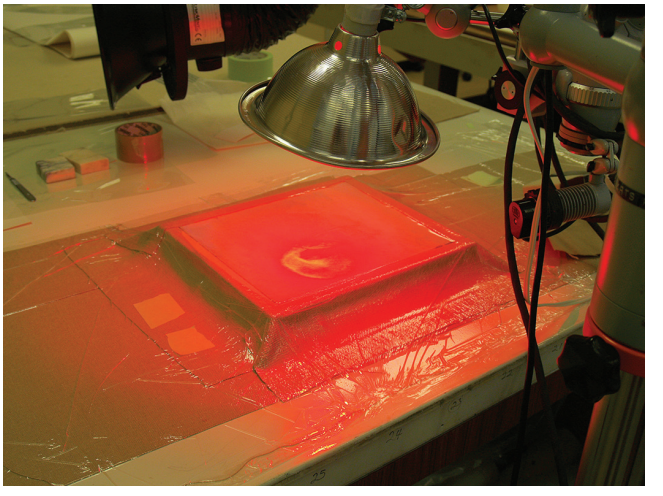

Figure 9. Application of heat and pressure. A heat-set lining taking place under vacuum pressure with heat supplied from above using an infrared lamp.

FACINGS

Facings for stabilizing cracking and flaking

Paper facings can be applied locally or overall, depending on their function. The facing adhesive can simply hold protective facings in place or supply the adhesive, either partly or in full, for consolidation of a cracked paint layer. Overall consolidation using rabbit skin glue, isinglass or parchment size can be facilitated by using facings attached with these products (**Figure 8**). If only used to attach a facing, the adhesive must not impede the passage of other materials that may be used to treat delamination. If a varnish layer is to be preserved, then the facing adhesive must not compromise varnish integrity. This normally implies the use of a gelatin-facing adhesive. Two or more layers of facings are applied if the treatment involves significant manipulation of the canvas (removal of old lining and lining adhesive). In this case, the two layers of facings are applied using different adhesives: one water-based, the second solvent-based. Facing materials will differ; normally paper facings will be adhered directly against the paint layer using a solvent-based adhesive, and a second or third layer of facings, usually fabric, will be applied above the paper facing with a collagen glue.

Treatments on the vacuum hot table, where the painting will be exposed to heat and pressure, require the presence of overall facings—applied loosely with or without an adhesive—to prevent polishing of the paint surface. For paint that is sensitive to adhesives and requires localized and very temporary facings, cyclododecane can be used

either with or without paper (Cagna and Riggiardi 2008). Cyclododecane has found a variety of uses in conservation because of its ability to sublime at room temperature.

Facings as Treatment

The contractive force of collagen can be used, in conjunction with facings, to gently flatten superficial cupping or flaking. This can be done either locally or overall. Treatments for the removal of wall or fresco paintings exploit the contractive forces of collagen glues. When fully faced with glue-impregnated fabric where no plasticizing agent, such as the traditionally used honey, has been incorporated into the glue, the surface of a wall painting or fresco can be peeled away from the underlying plaster (this technique is referred to as *strappo*). If a plasticizing agent is added to the facing adhesive, the paint remains secured to the underlying plaster, and a layer of plaster is removed along with the paint (this technique is referred to as *stacco*).

STRUCTURAL REPAIR

When canvas degrades, various levels of structural intervention can be undertaken. Initially, the tacking margins fail at the turn over. These areas can be repaired or reinforced with a backing fabric attached with an adhesive (often BEVA 371 Film or original formula). This process is referred to as strip lining. When the entire canvas weakens to the point where it no longer supports the paint layer or large tears require significant intervention, full linings are often the treatment of choice.

Glue and glue-starch linings were once commonly used for structural reinforcement, as were wax and wax-resin linings. Starting in the mid-19th century, wax and wax-resin linings were carried out, becoming increasingly popular from the 1950s onward with the introduction of the vacuum hot table (Phenix 1995, p. 26). There are some advantages to the use of wax as a lining adhesive. It is a hydrophobic material that fully impregnates the canvas during lining and can, as a result, decelerate moisture uptake or loss in the canvas (Young and Ackroyd 2001, p. 99). Canadian Conservation Institute research shows that wax resin formulations used to impregnate a canvas (lining or original support) will increase the tension of the fabric (at least in the short term, making this a more effective support for paint and ground layers), can assist in the prevention of cracks, and can impede the increase in cupping (Michalski and Daly-Hartin 1996). Wax formulations have, however, severe disadvantages, such as potentially high activation temperatures (54–80°C) that provoke weave transfer, increased transparency of paint and ground layers, and darkening, hardening and potential acid-degradation of canvases.

The conference on Comparative Lining Techniques, held in association with the National Maritime Museum, Greenwich, in 1974 (Villers 2003), addressed issues and solutions for lining and continues to influence the selection of materials and techniques. The current philosophy promotes minimal intervention (Phenix 1995; Ackroyd 2004). Minimal intervention implies either non-adhesive linings or strip linings, and low heat or cold linings where a nap-bond (non-penetration of adhesive into the canvas fibres) rather than a full impregnation of the original canvas is the goal. Lining is no longer expected to provide a single-step solution for the resolution of damage.

When linings are necessary, there are a number of alternatives from which to choose. Heat-set or solvent-reactivated BEVA 371 or BEVA precast films remain popular in North America. The adhesive, if applied in liquid form, can be flocked (sprayed) or brush-applied to the lining fabric, and a reliable nap-bond can be achieved on the vacuum hot table at temperatures between 65–68°C (Berger and Russell 2000). Cold linings, developed in the 1970s, were intended to have low impact. Mehra (1975) developed a cold-lining technique using a thickened acrylic dispersion adhesive Plextol B-500 applied through a screen to the lining canvas only. The painting is placed above the prepared lining canvas and drying occurs on a low pressure airflow table. Other cold or low heat-seal linings have been carried out using acrylic copolymers Plextol D360 and Plextol D498 on a multi-purpose low pressure table (Hacke 1978). BEVA Gel produces a lining with a very strong bond (Cser 2002). Cold linings, while avoiding many of the dangers and potential side effects of heat-based linings, present the complication of managing exposure of the original canvas to moisture when the lining adhesive is a dispersion. Protection against moisture transfer can be provided by coating a moisture barrier of solvent or heat-activated adhesive film on the back of the painting canvas (Cser 2002, p. 4) or through solvent reactivation of the cold lining adhesive, referred to as "dry film cold lining" (Mehra 1981, p. 13).

Structural treatment using adhesives can also involve the re-adhesion of a painted canvas to its original wall or other original solid support. Large murals, which are painted canvases glued to plaster walls, are prone to partial delamination over time. The original adhesives used to attach canvas to plaster-finished walls were normally animal glue or starch paste, and it is best to either reactivate the original adhesive

using moisture or to inject an easily reversible adhesive to reattach sections of canvas that are lifting away from the wall. In the case of a painting on canvas epoxied to a plywood auxiliary support by the artist, the solution chosen for reattachment of the lifting canvas was to inject an adhesive, BEVA D8, by means of pre-drilled holes through the wood support into areas of delamination (**Figure 10**). The old epoxy sealed the wood, preventing it from responding to the moisture in the adhesive, while this same moisture assisted the canvas, ground and paint layers to relax back down onto the auxiliary support (Cyr and Baker 2010).

Tear repair

Invisible tear repair eliminates the need for patching tears and for linings or marouflage, treatments that were once undertaken to stabilize these damages (Heiber 2005). The tear repair technique reintegrates broken threads into the painting structure and permits the damaged area to respond, like the rest of the canvas, to changes in surrounding conditions. This technique eliminates the use of localized patching of tears and the inevitable problem of "patch transfer" (bulging of the canvas above the patch). The mending adhesive is a mixture of sturgeon glue (20% w/v in water) and wheat starch paste (WSP) (10% w/v in water) combined in equal parts per volume (Heiber 2005, p. 87). The sturgeon glue provides high tack and strength, and the WSP acts as a filler or bulking agent (**Figure 11**). Once in place, the adhesive is spot-welded at very low temperatures into the broken thread ends. The bond is strong, readily water soluble and can be reworked or undone if necessary. Jade R, a water-reversible PVAC dispersion, has also been used successfully for invisible tear repair. When repairing clean cuts with no overlapping threads, a quick-set epoxy is employed. This material can reliably hold cut thread ends together and is sufficiently strong to accommodate stresses placed on it by movement in the surrounding canvas. Other adhesives, such as the thermoplastic resin Polyamide Welding Powder, can be substituted, but this has a much higher melt temperature than sturgeon glue and WSP, and is not water reversible.

Figure 10. Reattachment of delaminating support layers. BEVA D8 is injected through a plywood auxiliary support into an area of delamination between the wood and the primary canvas support. The canvas, originally adhered to the plywood by the artist using epoxy resin, is now lifting and bulging away from the plywood in multiple locations.

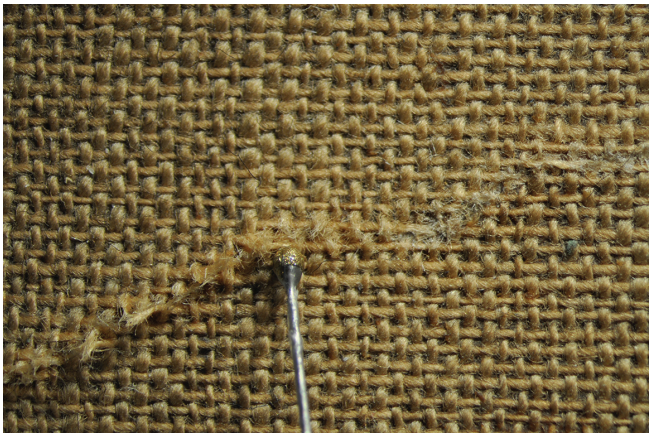

Figure 11. Invisible tear repair. A tiny dot of sturgeon glue combined with wheat starch paste is placed on the overlap of torn threads. The adhesive is then heat welded into the threads to effect a strong adhesive repair.

Archaeological Bone and Ivory

by Tara Grant, Canadian Conservation Institute

There are two major instances when adhesives or consolidants are used on bone and ivory: to adhere a loose fragment back to a larger portion of itself or, if a decorative element, to another substrate such as wood; and to consolidate a weakened, deteriorated structure with a powdery or porous surface. This section focuses on archaeological bone and ivory. Archaeological bone and burnt bone often need to be consolidated to maintain structural cohesion.

Polymers used as adhesives in bone and ivory conservation are sometimes used as consolidants when diluted. Since consolidants are more invasive than adhesives, selecting them is more complex. Therefore, adhesives will be discussed within the context of consolidants. Only significant differences for adhesives will be noted.

Properties of bone and ivory that influence choice of consolidant

Bone and ivory are composed of an organic network of collagen fibres with the interstices filled with an inorganic component of crystalline calcium phosphate called hydroxyapatite. The mineral component provides strength while the collagen provides flexibility. A consolidant must provide strength as well as flexibility to accommodate the dual nature of this material.

The structure of bone consists of an interior of an open network of lace-like bone, called cancellous bone, and an outer shell of compact, dense, solid lamellar bone. Lamellar bone is hard, rigid and relatively non-porous. It is difficult to penetrate with a consolidant. Cancellous bone, however, is lightweight, open, extremely porous and more likely to be in poor condition. Penetration of the cancellous areas with consolidant is much easier. Adhesives and consolidants must be chosen with low viscosity and concentration, and small particle size to penetrate the lamellar bone while having sufficient strength to provide adequate support for the cancellous areas. Solvent choice can also affect the viscosity and penetration.

Ivory consists of dentine—a dense, white, hard material. Unlike the cancellous part of bone, the centre of ivory is solid. Ivory is laid down in layers of concentric growth rings and will delaminate along these rings and split longitudinally as the individual layers contract and expand at different rates. Ivory is much more problematic to consolidate than bone because of its density and impenetrability.

When bone is burnt, the organic material is destroyed, leaving only the mineral structure. The bone is lighter in weight, porous and fragile. The colour ranges from chalky white through grey to black.

Archaeological bone deteriorates during burial and is often extremely fragile and in need of consolidation. Bone and ivory both deteriorate in a similar way when buried, but ivory deteriorates at a slower rate due to its denser, less porous structure, which slows the absorption of water, minerals, acids and bases. Depending on burial environment, selective deterioration of the organic or inorganic component can leave the bone or ivory severely damaged, weak and in need of consolidation. In an acidic burial (pH <6), such as a peat bog, the mineral component is destroyed, leaving a bone that, in the extreme, is flexible, flaky and very sensitive to humidity changes. Ivory delaminates and loses the surface gloss. In an alkaline burial (pH >8), such as a shell midden or limestone area, the hydroxyapatite is preserved and the organic component is destroyed, leaving bone that looks robust but is more brittle and less responsive to changes in relative humidity (RH). Bone is more likely to survive in an alkaline environment. Organic components may be replaced with mineral compounds, such as silicates and carbonates, that permineralize or "fossilize" the bone, increasing its brittleness and loss of cohesion. This permineralization increases the likelihood of needing consolidation while also increasing the density of the bone and making it less porous.

Both bone and ivory materials are sensitive to water, including changes in RH. The collagen component expands when exposed to high RH, while the mineral component remains static. Both materials are anisotropic, expanding and contracting to changes in RH in three directions: longitudinally, horizontally and diagonally. The higher density and alternating rings of collagen and hydroxyapatite in ivory make it particularly sensitive to changes in RH. The sensitivity

of these materials to water, particularly ivory, makes selecting an aqueous-based adhesive or consolidant a matter for serious consideration. Only bone in good condition should be exposed to aqueous adhesives or consolidants. Organic solvent-based adhesives or consolidants are recommended for ivory. Thin panels of ivory that are delaminating, splitting or flaking should not be exposed to water-based adhesives. All wet archaeological materials, however, can be treated with water-based materials.

Large quantities of degraded bone can be recovered from archaeological sites. For example, at the Windover site in Florida, 168 articulated and disarticulated individuals were recovered over a three-year period (Stone et al. 1990). A moderate-sized excavation can recover 5 to 20 kilograms of bone per day (Koob 1984). These large quantities necessitate choosing an adhesive that is easy to use, non-toxic, safe, inexpensive and has quick penetration and drying times. Organic solvent-based consolidants may not be desirable.

Non-aqueous consolidants have many disadvantages. They cannot be used on wet material because they form a filmy, white precipitate that obscures the surface. Water in the artifact needs to be replaced with a solvent prior to consolidation. Most solvents are flammable and toxic. They are expensive, must be disposed of properly, and cannot always be transported to remote sites. Suitable fume extraction and personal safety equipment are necessary for safe handling. Long-term exposure to organic solvents has health risks. Solvents have a lower surface tension than water and solvent-based consolidants generally penetrate better and faster. If the solvent evaporates too quickly (e.g. in a hot climate or the solvent is quite volatile), then there is little penetration and a skin is formed on the surface. This film obscures the surface detail and can trap moisture below, leading to stresses that crack the bone. A problem associated with aqueous consolidants is mould growth in the solution or during the drying period.

As with most materials, bone and ivory are best adhered or consolidated with a polymer with a neutral pH. The dual nature of bone and ivory, with organic and inorganic components, means that the materials will be affected by both high and low pH. As the mineral component of bone is the largest and most resilient segment, bone tends to survive better in alkaline burial conditions. Although slightly alkaline consolidants are often considered acceptable for bone, no studies have been done to determine this, and short immersion times are suggested (Stone et al. 1990).

Glass transition temperature (T_g) is important when choosing consolidants for bone and ivory. They need a high enough T_g to provide stiffness and strength without losing flexibility. Consolidants with a T_g in the range of 40°C are commonly used. Another consideration is the location and storage environment of the bone. Bone in hot climates where the display and storage environment is not controlled will need an adhesive with T_g above the ambient temperature to prevent a tacky surface, slumping or failure of the adhesive or consolidant (Koob 1981).

Glossiness is aesthetically displeasing and is generally unwanted. If applied too thickly, almost all consolidants will appear shiny. Often, this can be controlled by using minimal amounts of consolidant and removing excess. Due to its smoothness and greater impenetrability, cortical bone will often appear glossy, while the cancellous bone is matte.

Most adhesives and consolidants used in conservation are theoretically re-soluble, as they stay soluble in a solvent over a long period of time. In practice, most consolidants, when applied to bone and ivory, are not totally reversible. Artifacts that require consolidation are generally too fragile to withstand removal afterward. One study on earthenware that may illustrate the type of problem shared with bone and ivory showed that 50% of the polymer (Paraloid B-72) remained in the earthenware after 8 hours extraction with a Soxhlet apparatus (Horie 1982). Consolidants with poor long-term aging properties should never be used for consolidation.

There is an important shift in archaeology away from excavation and towards the study and scientific analysis of archaeological bone. This includes morphological examination, scanning electron microscopy, isotope analysis, radio-carbon dating and DNA analysis. Bone destined for analysis must not be consolidated, as it could interfere with the results. Samples for dating or other analysis should be selected and removed before treatment (Johnson 1994; Cassman et al. 2006; Unruh 1994; Nicholson et al. 2002).

Choosing an adhesive or consolidant

A multitude of adhesives have been used to mend bone and ivory artifacts. Provided that they meet general conservation conditions of stability and reversibility, most conservation-accepted adhesives can be used. The major properties to consider are sufficient strength to support the mend, but not stronger than the bone or ivory; some flexibility to match movement of the artifact; neutral or near

neutral pH; clear appearance; suitable T_g for the storage environment; and the choice of an aqueous or non-aqueous solvent.

Consolidants also need to possess all the good conservation qualities listed above. Of particular importance is good penetration. As larger quantities are used, toxicity and ease of use need greater consideration.

Most conservators tend to select from a small group of adhesives and consolidants that have been tested and are known to have good long-term aging properties.

ADHESIVES AND CONSOLIDANTS

While many consolidants have been used on bone and ivory, some of which are described below, conservators have predominantly used three: Rhoplex AC-33, Acrysol WS-24 and Paraloid B-72.

POLY(VINYL ACETATE) DISPERSIONS

Poly(vinyl acetate) (PVAC) dispersions have been used to adhere and consolidate archaeological bone and ivory since the 1950s, when they were introduced to archaeologists by paleontologists (Johnson 1994). Ordinary hardware-store-variety white glues, typically PVAC dispersions, were and still are extremely popular with archaeologists, as they are easy to use and acquire. These commercial PVAC dispersion adhesives should never be used as a consolidant. They are designed to be used as an adhesive and are too viscous to be used as a consolidant for bone and ivory. They have many disadvantages, such as low T_g, high emulsifier and plasticizer content, large particle size (0.5–1.0 µm average diameter), high viscosity, acidity (3.0–6.0) and poor water resistance (Koob 1984). Reversibility is also a problem. Additives and emulsifiers that improve working properties may aggravate crosslinking, causing irreversibility.

POLY(VINYL ACETATE) RESINS

PVAC resins have been used to consolidate and adhere bone and ivory. Solubility, hardness, strength and T_g can all be manipulated by the choice of molecular weight, and they are easily dissolved in less toxic solvents (e.g. acetone, ethanol). They are stable and reversible. The consolidant does not have good water impermeability and bone continues to absorb water, expanding and contracting with inevitable cracking. The light-aged resins can be acidic (pH ~4.5) (Down et al. 1996), which may affect the bone.

ACRYLIC DISPERSIONS

Acrylic dispersions have been in use in archaeology since the late 1960s (Johnson 1994) and are considered more suitable for consolidation than PVAC dispersions. They tend to have smaller particle size and therefore penetrate better than the PVAC dispersions, contain little or no emulsifiers or stabilizers, have higher T_g and greater water resistance (Koob 1984). Other advantages are that they are water-based, non-toxic and easy to apply. A major disadvantage of some of these dispersions is their high pH (up to 9.5) (Koob 1984). For alkaline dispersions such as these, immersion times should be kept to a minimum (Stone 1990).

The most common acrylic dispersion used in the past was Rhoplex AC-33. Although Rhoplex-AC-33-consolidated bone appears to have aged well (Stone 1990), testing has shown that the pH after aging becomes acidic (3.7–4.4) (Down et al. 1996), which may be unacceptable for bone. Also, although it has not been investigated scientifically, it was noted that when wet archaeological bone was immersed in Rhoplex AC-33, a sour milk smell developed after several hours or days; therefore, reuse of the solution was not recommended. Rhoplex AC-33 is no longer manufactured (CAMEO 2007).

In the 1980s, other acrylic dispersions started to appear that had a neutral pH (7.0–7.5) and smaller particle size (0.03 µm), which afforded better penetration. The most common ones used today for bone consolidation are the Acrysols, particularly Acrysol WS-24.

ACRYLIC RESINS

Paraloid B-72 is one of the most common adhesives used in conservation because of its known stability, lack of colour, moderate strength, good adhesion, good viscosity, reversibility and moderately high T_g (40°C). It is impermeable to moisture. Paraloid B-72 is the most common solvent-based bone/ivory consolidant.

POLY(VINYL BUTYRAL) RESINS

Poly(vinyl butyral) resins, particularly Butvar B-98 and Butvar B-76, are widely used in paleontology, but are only occasionally used to adhere or consolidate archaeological bone. They have good penetration, strength, stability and reversibility, and quick drying times (Kres and Lovell 1995). The T_g for these resins is high (range from 48–65°C), which makes them brittle.

CELLULOSE NITRATE

Cellulose nitrate (CN) has been in use in archaeology and conservation since 1899 (Horie 2010). Despite knowing within a decade of their introduction to conservation that CN was unstable, it continued to be used, as it was easy to acquire and had good working properties. CN becomes extremely brittle, shrinks so dramatically that it can detach pieces of the bone, yellows severely and is extremely acidic. Duco Cement and UHU Hart are still commonly used today as adhesives and consolidants (thinned with acetone), despite their drawbacks. A notable exception may be the HMG Heat and Waterproof Adhesive, which was found to be stable (for about 100 years) under museum conditions where it was protected from extremes of temperature and RH (Shashoua et al. 1992), although other studies found that it yellowed, became more acidic and more flammable (Koob 1982).

CYANOACRYLATES

Although widely used in paleontology for fossil preparation (Down 2001), cyanoacrylates are not commonly used in archaeology or conservation for bone and ivory treatments. Generally, the adhesive is brittle, has poor impact resistance and poor longevity. Most cyanoacrylates used in paleontology have superior penetration and wetting, are easy to use and dry extremely quickly, making them popular for field and lab settings. Cyanoacrylates can stain artifacts. All cyanoacrylates are difficult to remove.

POLY(ETHYLENE GLYCOL)

Poly(ethylene glycol) (PEG) of various molecular weights is commonly used to bulk the cellular structure of wood and leather to prevent shrinkage and collapse. Despite the differences in physical properties between the materials, PEG has been used as a consolidant for wet archaeological bone and ivory. It is a poor consolidant as it has minimal adhesive properties, little structural strength and is hygroscopic (Stone 1990). The PEGs with low molecular weight "sweat" out in high humidity, accumulating on the surface, masking morphological features and capturing dust and dirt. Darkening of bone ends due to PEG in the cancellous areas has been noted.

SILICONE POLYMERS

Silicone polymers, originally designed for preservation of anatomical specimens, have been used to consolidate wet archaeological wood, textiles, rope and glass, as well as bone and ivory recovered from shipwrecks. The material has good penetration, causes little dimensional change, treatment times are relatively short and the finished artifacts are aesthetically pleasing. On the negative side, there are no long-term stability studies, the process is irreversible and the consolidant is expensive. Wet artifacts must be dehydrated in acetone prior to silicone treatment, which is expensive and poses safety issues. The treatment increases weight (by about 25%) and slightly darkens and softens the bone (De la Cruz Baltazar et al. 1996).

TREATMENT METHODS

ADHESIVES

Adhesives are generally applied directly to the surface as a wet solution. They can be applied with a brush, applicator stick or syringe. The adhesive must be thick enough to prevent extensive penetration into the bone or ivory. Thickeners (e.g. fumed silica such as Cab-o-sil) can be added to increase viscosity, although they may lower strength and adhesion. The strength of the adhesive must be suitable for the size and weight of the pieces being assembled. If there is joint failure, it should occur at the interface between the bone and adhesive. Large, heavier pieces will need adhesives with greater structural strength. Dry pigments can be added to the adhesive to match the colour of the ivory.

CONSOLIDANTS

Surface application

For this application technique, a dilute solution of the consolidant is applied with a brush, pipette or syringe to the surface of the bone. Surface application provides good control. However, the brushing motion may lift powdery surfaces or break off small fragments of bone. Also, the cancellous areas of the bone may be difficult to penetrate and result in a consolidated surface layer and a weak unconsolidated interior. This may result in stresses from different contraction and expansion rates, leading to cracking and spalling of the surface. Brushing aqueous consolidants on wet bone will give better penetration than on dry bone.

Total immersion

For excavations that have wet, damp, large or huge quantities of bone, total immersion in the consolidant is often used. Immersion gives a more even application and better penetration than brushing. Bones can be placed on mesh screens or suspended in mesh bags to facilitate handling and to keep the bones suspended above any dirt settling to the bottom. Small bones can be immersed for a few minutes, while larger and lamellar bones may require immersion for

several hours. Excess consolidant should be removed from the surface with a sponge. The bones can be dried on a rack to minimize surface contact and to allow an even air flow around the bones. If drying on a flat surface, the bones should be flipped to allow even drying. The artifacts should be dried slowly to prevent cracking and warping (24–48 h for small bones, longer for large bones). To increase drying time, the RH can be raised with humidifiers, damp towels or by covering the artifact with plastic. Larger, more porous bones may need to be re-immersed to ensure adequate consolidation.

Wicking

Wicking is a less extreme form of immersion. It combines good penetration with better control. Dry bone or ivory when immersed in a liquid may develop air bubbles in cracks and pores, which could push loose fragments off the surface as they are released or which could block the entrance of the consolidant. In one method for wicking, the bone is placed in a shallow dish of the dilute consolidant, which is slowly wicked up the object by capillary action. An alternative method is to wet the object with pure solvent followed by slowly adding more solvent until the object is immersed, at which point the consolidant is slowly added (Snow and Weisser 1984).

Immersion under vacuum

Immersion in a consolidant while a low vacuum is applied is not commonly done to bone. While it works well for less porous substrates, such as metal and metal corrosion products, bone is generally porous enough to absorb the consolidant without additional aid. Placing bone under a vacuum may cause the piece to crack if internal pressures cannot equalize fast enough. Consolidation is improved if the vacuum is applied slowly before the liquid consolidant is introduced or the artifact is immersed for a long period in the consolidant before applying the vacuum. This allows for the removal of air from the artifact (Horie 2010, p. 111).

Doyles method

A method of treating sub-fossil bone was developed by Doyle (1987) that uses a gravity drip apparatus to apply dilute consolidant to large, partially fossilized bones. Originally used with PVACs, the method has also been used to apply acrylic colloidal dispersions. Disadvantages are that bones must be humidified (80–85%) prior to consolidation (which stresses the structure and encourages mould growth), that a fungicide must be used, and that impregnation can take several weeks for large bones.

Furniture and Wooden Objects

by Donald C. Williams, Smithsonian Museum Conservation Institute

The selection and use of adhesives is but one part of the decision-making process for preserving and restoring historic wooden artifacts. There are a number of broader discussions outlined by Williams (1998) and Rivers and Umney (2003, pp. 158–193) on the selection and application of adhesives within the framework of wood object conservation. Since their formulation more than three decades ago, the principles of utility, (non-)intrusiveness, stability and re-treatability for adhesives in conservation have remained essentially unchanged (Down and Lafontaine 1980).

Wood adhesives and their properties

There are two broad categories of adhesives commonly used for conserving wooden objects: natural materials, usually proteinaceous, and synthetic resins. Natural adhesives, such as shellac and rubber-based cements, are rarely used. While the number of synthetic resins used for wood glues is large, conservators usually consider only a minute portion of those [e.g. poly(vinyl acetate) dispersions, two-part reactive glues, synthetic resins—both in solution and dispersions—and hot-melt adhesives of various formulations and forms].

The following survey of adhesives for conservation serves only to familiarize the caretakers of wooden artifacts with the variety of adhesive materials employed.

Animal glue

Historically, the most common adhesive for wood, at least until the age of modern synthetic polymers, was protein glue (animal glue). For the overwhelming majority of wooden artifacts encountered, the protein glue used was animal hide glue. For more information on these glues, see the section on *Proteins* in Chapter 6 and Hubbard's indispensible review of the nature and use of animal glues (1977).

The descriptor gram-weight strength (gel or Bloom strength), assigned to hide glues by testing, reflects molecular weight and strength under stress (Rosser 1939; DeBeaukelaer et al. 1930; Fernbach 1907). In general, the gram-weight strength of glues normally used for woodworking is in the range of 200–300, although the range available is much broader (<100 to >500).

When preparing and using hide glues, the initial step is to soak the dry glue in cold water in order to completely swell it. This swelling facilitates the creation of a solution when subsequently heated for use. Longer protein chains, which have higher gram-weight strength, absorb far more water per given mass than do shorter chains. So, for a given viscosity, a low gram-weight strength solution will have a higher solids content than a high gram-weight strength mixture. Conversely, a high gram-weight strength glue will be more viscous than a low gram-weight strength mixture of the identical solid to liquid ratio. The exact proportions of dry glue to water depend on the glue grade and the circumstances for using the glue. Once the glue has reached the proper temperature (60–70°C) and viscosity, it can be applied to the gluing surfaces.

Hot hide glue dries and hardens in a two-step process. The first step is gel formation, when the glue has cooled to its gel-point. Depending on the glue grade, concentration and ambient temperature, this can take from a few seconds in a cool room to several minutes on heated surfaces. Following the gelling, the semi-solid mass of glue hardens through loss of water to the surrounding substrate or to the atmosphere. As with all solvent-release processes, the volume loss, or shrinkage, in the drying glue will equal the volume of solvent lost.

The preparation of cold liquid hide glue is similar to that of hot glue, with the exception that it is further modified with gel suppressants (e.g. salt, urea) that prevent gelling at room temperatures. Cold hide glue hardens only by loss of moisture with its inherent shrinkage. The properties of dried cold glue are similar to those of the dried hot glue. Commercially prepared cold hide glues are available, but there is no way of knowing their composition or, most importantly, their shelf life.

Modifiers for animal glue include protein crosslinkers to yield a water-resistant thermoset adhesive, and water-soluble plasticizers.

Probably the most important reason that hide glues are so widely used in wooden furniture artifact conservation

is that they are almost completely reversible while maintaining extremely high performance under reasonable environmental conditions.

The sometimes brief working time, due to the gelling properties of hot hide glues, is not necessarily a drawback, as joins can be manipulated by heating the gluing surfaces, by selecting glue grade and solids content, or by adding gel suppressants. Higher gram-weight strength glues may have more inherent tensile strength, but also greater shrinkage and resultant stress upon drying that increases the risk of glue-line fracture. They also have higher viscosity (less penetration and wetting) for a given concentration (w/w) than lower gram-weight strengths. In short, while a higher gram-weight strength glue may be stronger, it might yield a weaker joint due to shrinkage (stress) and incomplete wetting. Should the glue gel before work is complete, it can be re-liquified by the application of heat.

Cold hide glues have a working time of up to an hour, allowing for very complex gluing procedures. However, if the shelf life of the cold hide glue has been exceeded, the adhesive may not harden properly. Experience indicates that this is a problem only with those liquid glues with a urea gel-suppressant. Salt-infused glues can be used for several years after formulation.

The structure of animal glues suggests a true chemical affinity for wood (Von Endt 1986). However, they can be extremely brittle and easily fractured when hard. They do not function as a particularly cohesive material, especially in thick applications. Water-soluble plasticizers can help, but the best practice is simply to create the thinnest glue-line possible.

One final note is that the stability and properties of hide glue are greatly altered with a change in environmental moisture. When desiccated, the glue becomes brittle and can be fractured with little applied stress. If the humidity rises, the glue softens and is susceptible to plastic deformation.

NATURAL RESINS

Natural resins (e.g. shellac, colophony, dammar, asphaltum) are almost never used as adhesives for conservation, yet they are encountered in historic objects (Koob 1984b; Mantell 1942). The most extensively used resin was probably shellac, the properties of which depended on the source and refining process (Bose et al. 1963, also see *Shellac* section in Chapter 6). The primary use of shellac was to glue non-wood veneers to a wooden substrate.

CONTACT ADHESIVES

Contact adhesives, whether formed from natural or synthetic rubbers, in solution or dispersion, are rarely used in wooden artifact conservation (Feller and Encke 1982). For artifacts employing them as a laminar adhesive, contact adhesives, which are frequently rubber-based materials, are at least initially thermoplastic and can often be softened with heat and/or organic solvents.

SYNTHETIC-RESIN ADHESIVES

For wood conservation, the easiest way to group synthetic-resin adhesives is not by means of the chemical nature of the materials, but rather through the application method or the formulation for use. Within this grouping there are four major types: dispersions, solutions, hot-melts and reactives.

Dispersions

Dispersion adhesives, usually referred to as *emulsion adhesives* in the craft literature, are the most common adhesive in contemporary woodworking (e.g. Elmer's White Glue, Titebond yellow glues). They are also used with moderate frequency in conservation, usually to repair a break or fracture in a wooden element (e.g. Jade 403, Rhoplex AC-234).

Specialized dispersions can be obtained with a wide variety of specific properties, including molecular weight ranges and solubility characteristics for a hardened film.

Dispersions have several distinct advantages. They are easily obtained and used, have moderate working time, are easy to clean up with water and have a good shelf life. They require essentially no preparation and commercially available products are ready-to-use straight out of the container. Depending on the formulation and the environmental influences, they can be durable adhesives that remain stable for lengthy periods of time. They also remain, to some degree, solvent-sensitive and reversible. As with other glues, the best bond-line is very thin.

Solutions

Synthetic-resin solution adhesives are not widely used in the woodworking craft, but have many applications for wooden artifact conservation.

Individual resins, or blends, can possess very specific characteristics such as solvency and long-term stability. For example, resin solution adhesives may be designed that will function well but not affect the surrounding areas of the object. As thermoplastic solutions, they dry through solvent evaporation

and, depending on the formulation, may remain soluble for a long period of time. The stability of certain resin solutions has been well-documented in the conservation literature (see relevant sections in Chapter 6).

Hot-melts

Most thermoplastic materials could be classified as *hot-melt adhesives*, using a broad definition of the term. Hot-melt adhesives, as defined in this section, are a group of materials specifically *designed* to be used in a molten state that harden solely by cooling. With respect to the properties of the adhesive, the formulation of these adhesives can be very specific, not only when solid but also when liquid (Gutcho 1983).

Hot-melt adhesives come in two major configurations: sticks that must be used with a heated glue gun and sheets or films that can be used anywhere the gluing surfaces can be heated (e.g. BEVA 371 Film). Glue guns are normally used when there is a need to inject or apply molten adhesive to a surface, and sheet adhesive is used between laminar surfaces that will be heated and/or pressed.

Hot-melts are increasingly important in the conservation of wooden objects where flexible gap-filling properties are valued.

Reactive adhesives

Thermosetting multiple-component adhesives are used in conservation as consolidants when there is serious damage to the wood fibre matrix. Specific characteristic differences between these adhesives are of little importance for the purpose of this discussion.

Reactive adhesives include epoxy resins, urea formaldehydes, urethanes, phenolics and the like. They are problematic as wood conservation adhesives inasmuch as they are essentially intractable and irreversible once cured, and are usually much harder than the surrounding wood.

Cyanoacrylate, a popular one-part reactive adhesive, is likely to be encountered mainly as used in the fabrication or previous repair of musical instruments. It is rarely used in the conservation of wooden objects.

Reactive or thermosetting adhesives can be resistant to in-use deterioration resulting from thermal, physical or chemical phenomena, but not always. For these adhesives, there is generally negligible dimensional change upon curing. As such, they may be good gap-fillers in either their raw state or when modified with bulking agents. However, they tend to be much less flexible than wood.

The use of reactive adhesives in wood conservation is generally discouraged for routine gluing. By their very nature as crosslinking polymers, they are not easily reversible. Some can be swollen with solvents, but usually they must be removed mechanically, with the attendant risk to the substrate or adjacent surfaces. In addition, they may break down in a short period of time (a few years), especially in the presence of ultraviolet radiation.

SELECTION AND USE OF ADHESIVES IN FURNITURE CONSERVATION

The ideal adhesive for furniture conservation is one that would be perfectly stable over time, would be easily applied and manipulated, would be readily removable if further treatment is required later, and would form an adhesive bond strong enough to allow the object to fulfill its function, yet weak enough to be the sacrificial boundary in case of applied stress. Obviously, no single material fulfills all of these requirements and thus there is no single "furniture conservation adhesive". Instead, the conservator uses a palette of adhesives with known characteristics. The specific needs of the object are considered, and the adhesive most closely matching those requirements is used. Unless the circumstances are extraordinary, the adhesive chosen is a thermoplastic material. The most widely used adhesives are hot and cold hide glues, and hot-melt synthetics, with only a minor use of dispersions or solvent-borne resins. Crosslinking reactive and thermosetting adhesives are almost never used as replacement adhesives when treating joint failure.

The criterion of object condition and use, and the structural stresses placed on the object during that use is particularly important. The first step in assessing the condition of the object is to conduct a brief visual and tactile examination. If the object is no longer a whole unit, examine the gluing surfaces to determine their soundness. Proceeding from this point depends on the answers to the following questions. What came apart (what are all these pieces and how do they go together)? How did they come apart? And, why did they come apart?

If the object is still intact, or at least all the parts are in some way still connected, the focus of the initial examination questions is slightly different. Are portions of the object in the wrong orientation to the remainder of the object? Do any of the parts feel loose? Does the object move under stress and, if so, how and how much?

In addition, it is useful to draw distinctions between the types of damage being repaired. Is it a failure of an aged glue-line, a split in which there is separation but still some degree of attachment, or a break with complete dissociation?

In preparing to implement any conservation treatment, it is vital to assess the stability of the adherends. Not only must they be structurally intact but, as Pine (2006) has pointed out in his cautionary tale of finish adhesion or lack thereof, if the surfaces and wood matrix are contaminated, even purposefully, the success of any adhesion is in doubt.

TREATMENT REQUIRED AS A RESULT OF WOOD FRACTURE

Simple fractures

A simple longitudinal fracture where there is minimal distortion to the object and little damage to the gluing surface requires only the introduction of an appropriate adhesive, alignment and modest compression to complete the re-assembly. The adhesive selected depends on the stresses the object must withstand, the sensitivity of any decorative surfaces of the object, the complexity of the damage or the object, skill of the conservator, etc.

A partial fracture, where the object is still in one piece, sometimes tenuously, does not always give easy access to the gluing area, and glue must be applied by either flowing it into the void or forcing it in under some pressure. The first can be accomplished through gravity or capillary action, the second usually requires some hydraulic device, such as a syringe or fingertips pushing the glue into the joint using an impermeable plastic transparent sheet as barrier. For the latter method, hot glue is forced through the cracks at the surface using fingertips. In order to assure that adequate glue is available to be worked into the void, the application of hot hide glue is generous. Immediately following the application of the hide glue, a transparent impermeable plastic sheet is placed over the glue. The sheet, a very thin polyethylene film or a similar material, has several functions in the procedure. Once the transparent sheet has been placed over the glue, the surface is rubbed through it, working the glue into the cracks with a rolling and pushing motion of the fingertips with the sheet acting as a malleable plunger. The end of the procedure is reached when no more glue can be forced into the void. The transparent sheet also allows the working time to be extended indefinitely through retarding water loss and the application of heat from a lamp.

The complete fracture (more pieces than originally existed) presents the advantage of easy and immediate access to the gluing surfaces. Cross-grain fractures may require dispersion or reactive glues to impart necessary strength.

The final stage in the process is to align parts and clamp until the glue dries. Frequently, clamping squeezes out excess glue, which must be cleaned immediately.

Complex fractures

The most vexing circumstance appears when the gluing surfaces are no longer adequate for the task of re-assembling the artifact, either through distortion (leaving a void in the alignment), or damage to the gluing surface through displacement ("splintering" of the wood fibres). Alignment and substrate voids are not adhesive problems, they are structural. As with any damaged gluing surface, decisions must be made as to how intrusive the conservation work should be.

The existence of substrate voids, either in the fracture region as a whole or at the glue-line in particular, may contribute to the object's overall current or future structural instability, which usually determines whether the voids are to be filled.

When filling the voids, at issue is the degree to which the artifact is altered to facilitate the fill. In cases of a shredded or degraded gluing surface, either consolidation or trimming may be considered. If alteration of the artifact is not possible or desirable, the fill must be made to fit the void exactly.

Methods of accomplishing a fill often include fitting a wood piece to the void precisely or casting thermoset material into the void. Unless the fill is a tight wood-to-wood system, the gluing surface should be isolated from the fill with an easily reversible barrier coating [e.g. animal glue, synthetic-resin solution, hydroxypropylmethylcellulose, low molecular weight poly(vinyl alcohol)].

Wood-to-wood repairs generally work well only where there is a clean gluing surface, which is not shredded or undulating. If a surface is shredded or undulating, wood fills can be employed, but often with a bulked-out adhesive or fill material—such as liquid adhesive formulations that have been thickened with fumed silica, glass microballoons, phenolic microspheres, etc.—used between the gluing surface and the solid fill to eliminate the presence of voids in the repair system (Anderson and Podmaniczky 1990). Whatever procedure and materials employed,

it is critical for the fill to be weaker than the adjoining artifact. If stresses cause damage in the future, it should be to the repair, not to the artifact.

TREATMENT REQUIRED AS A RESULT OF DEGRADED ADHESIVES

Treatment options for failure resulting from degraded adhesives include one or more of the following three broad approaches: 1) the re-activation of the adhesive by solvents or other means; 2) the introduction of a new adhesive that is compatible with the old adhesive; and 3) removing the degraded adhesive and re-adhering the substrates with new glue.

Treatment strategy is limited by the properties of the old adhesive, the probable end use of the object (design for worst-case scenario) and the properties of the treatment.

While each adhesive system failure is highly specific, the responses revolve around the same set of concerns: how severe is the failure, what is causing it, how stable is the remainder of the adhesive system, what is the adhesive that failed, and what treatment can compensate for the failure? These questions, listed in rough hierarchy, are by no means limited or static. The exact identity of the failed adhesive, as opposed to the *characteristics* of the adhesive, is frequently of only modest importance.

Re-activating adhesives

Re-activating existing adhesive is the most restrained intervention. By definition, this technique can be applied only to materials that are thermoplastic. Even when successful, this approach rarely yields a bond of structural strength, and the object may be incapable of fulfilling a normal use requirement. Because of these limitations, this method is usually reserved for objects that need to only support their own weight.

Identifying the existing adhesive's *properties* is especially important. Evaluating the possible re-formation of a failed adhesive is possible by careful trial-and-error solvent testing combined with visual identification and reasonable assumptions.

One common procedure is the re-activation of aged hide glue. First, clean the surrounding surfaces of dirt and other contaminants. Warm water is then introduced to the gluing area by syringes into the aged glue line, allowed to swell, and the elements are lightly clamped until the glue re-hardens. The fissures of the aged glue may become re-consolidated, but there will probably be no thorough re-formation of the bonding material, which usually requires the introduction of excessive water for several hours, an undesirable circumstance for wooden objects. Due to these limitations, the strength of the resultant bond may be quite low. The most widespread incidence of this practice is the transparent sheet technique, mentioned above under *Simple fractures*, applied to delaminated marquetry.

Solvents used to reactivate synthetic solvent-based adhesives could damage the object's decorative surface. Certain of these adhesives can be re-activated with heat instead, which may re-introduce increased plasticity to allow re-attachment. There are, of course, associated risks involved whenever a wooden object, or portion, is heated.

Adding new adhesive

Any newly introduced adhesive must be compatible with, and bond to, the existing adhesive. If the appropriate adhesive is carefully introduced, the chance of success for this approach is greater than that of the previous method.

The introduction of new adhesive can range from re-gluing elements that are already apart to re-gluing elements that are restrained or cannot safely come apart even with the most careful effort. The usual methods of introducing new adhesive are syringe injection, allowing the adhesive to flow from a saturated glue brush into the crevice, or brushing the adhesive on the gluing surface if the object is apart.

There is growing interest in using synthetic hot-melt adhesives for this type of treatment. Hot-melts flow well, are versatile, and are available in formulations that are easily reversible with heat or solvents.

As with the technique of re-forming existing glues, when adding new adhesive, it is advantageous to introduce small amounts of solvent into the area prior to the new adhesive, which is then introduced by brushing, wicking or injecting. If there is no opening adequate for the introduction of glue, a channel must be created to allow the glue to be injected with a syringe. This option is undertaken only after extensive deliberation since it involves the removal of existing material from the artifact.

Elements that are already apart with stable gluing surfaces are re-adhered in a straightforward gluing process. For treatments involving the introduction of crosslinking thermoset adhesive, the importance of including a barrier coating for the adherends' surfaces cannot be overstated (Ellis and Heginbotham 2003).

Replacing existing adhesives

The option of completely removing the aged adhesive is available only when there is access to all gluing surfaces and when the continued presence of the old adhesive might further deteriorate the artifact.

Mechanical removal and application of solvent are used together, sometimes repeatedly, to remove degraded adhesive. If the adhesive is completely intractable, the choices by the conservator become more difficult. An example would be a crosslinked adhesive that is impervious to softening. The conservator must then weigh the relative benefits of a clean gluing surface, which can be obtained only by aggressive mechanical removal, against the possible risks of such a cleaning.

Following cleaning of the gluing surface and necessary repairs to the structure for re-gluing, the object is prepared for re-assembly.

Complex gluing problems, such as those encountered in delaminated wood veneers or marquetry, require a combination of the previously stated models: re-activating, introducing and replacing previous extant adhesives (Von Revenlow 1988; Edwards 1997).

Adhesives as gap-fillers

Adhesives can also function as gap-fillers. The subject has been discussed in conservation literature and work progresses in identifying and using new materials (Storch 1994; Grattan and Barclay 1988; Barclay and Grattan 1987). Provided the gluing margins are sized with an appropriate isolating barrier coating and the filling adhesive is rheologically compatible with the adjacent wood, great latitude is available to the conservator. In this case, reactive adhesives, particularly epoxies bulked with fumed silica or microballoons, or silicone rubbers can be and are employed in conservation treatments. For a more complete discussion, especially on the use of molten ethylene/vinyl acetate copolymer as a preferred flexible gap filling adhesive, see Williams (2011).

Waterlogged Archaeological Wood

by David W. Grattan, Canadian Conservation Institute

Archaeological wood, or wood that has been excavated from an archaeological site, is often wet or waterlogged and usually requires considerable treatment to repair or stabilize before it can enter a museum collection for preservation or display. This often means impregnation to control collapse or shrinkage for wet or waterlogged wood, and consolidation to deal with all types of fragile wood. It can also mean adhesive repair of loose or detached parts or fragments; however, for this sort of material this often implies gap-filling because clean glue surfaces are a rarity. Rice (1990) has written a very useful overview of this issue. Dry archaeological wood can be adhered just as you would any other ethnographic or historical wood object.

Properties and condition of the object that influence choice of adhesive or consolidant

The dominant concern in waterlogged wood is the desire to retain the item in a form that closely resembles the original artifact. While long-term aqueous immersion often seems to preserve wood, frequently it is only the outward form that is preserved. It is often found that considerable deterioration has occurred as the cellulose component of the wood is consumed by anaerobic bacterial action. The older the wood is, the greater the degree of deterioration. Paradoxically, older, very deteriorated wood is sometimes easier to treat than less deteriorated wood because the cell structure is more accessible to the impregnant.

Because of the propensity to shrink or even disintegrate upon drying, waterlogged wood has received considerable study and an exceedingly wide variety of polymeric and, sometimes, non-polymeric organic materials has been used successfully as impregnants. The most commonly used impregnant is poly(ethylene glycol) (PEG) because of its solubility in and compatibility with water, its low cost and simplicity of use, and because it usually gives good results.

Selecting adhesives for waterlogged wood is problematic because PEG-treated wood, for example, is difficult to adhere to anything. The presence of waxy water-soluble PEG can inhibit good bond formation with an adhesive. Thus, a good adhesive for treated waterlogged wood is one that is not softened or plasticized by PEG.

Selection of consolidants and adhesives for waterlogged wood

Consolidants

Impregnants for waterlogged wood are usually, but not exclusively, macromolecular organic materials that dissolve in water. Water-soluble impregnants can offer a degree of reversibility and there are examples of this in the conservation literature (Cooke et al. 1994). The water-soluble impregnants most often used include the PEGs of grades ranging from liquid 200 to solid 3350, various types of saccharides, including sucrose, mannitol, lacitol and sorbitol, and some of the water-soluble celluloses, including hydroxyethylcellulose and methylcellulose. Silicone condensation resins have been strongly advocated in recent years. Choice is influenced by the cost and ease of treatment, the ability of the impregnant to diffuse into the wood, its control over the shrinkage and collapse, and the appearance, physical strength and stability of the wood after treatment. These issues are discussed at great length in the extensive literature on the subject, but it should be recognized that no ideal impregnant has been found (Hoffmann 2005, 2002, 1991; Bonnot-Diconne and Hoffmann 1999; Hoffmann et al. 1997, 1994; Rice 1990). Issues such as penetration versus hygroscopicity versus duration of treatment and several other factors have influenced choice. PEG, however, has these advantages:

- it can be imparted in aqueous solution or dissolved in alcohols such as isopropanol or t-butanol, although the trend now is to avoid solvent use because of safety and health concerns; and
- it can be used in an air-drying or freeze-drying process.

PEG has been criticized on the basis of excessive cost, lack of oxidative stability and poor appearance of treated wood.

Sucrose is attractive in terms of cost, but does not control shrinkage as effectively as PEG and there are lingering worries about its biostability.

Mannitol, lacitol and sorbitol are saccharides that have been studied and occasionally used with varying degrees of success, but have not been adopted as widely as PEG because the results are less predictable.

The cellulose derivatives penetrate poorly by comparison with PEG but are occasionally useful as superficial consolidants.

Silicones have also been studied for use as impregnants for waterlogged wood and may have some potential yet little-explored advantages, such as long-term stability (Kavvouras et al. 2009).

Thus, PEG of various molecular weights (grades) offers the most reliable compromise (the greatest likelihood of a successful result) and is still employed most often by conservators. The Canadian Conservation Institute has developed software to aid in the use of PEG combined with freeze-drying. This software is called "PEGcon" and it allows selection and control of the PEG (CCI n.d.).

Adhesives

A thermosetting adhesive such as an epoxy resin or a polyurethane is often considered most efficacious for gluing waterlogged PEG-treated wood, but ordinary white poly(vinyl acetate) (PVAC) dispersion adhesives have also been employed (Rice 1990). The PVAC adhesives have mostly been adopted by trial and error on an ad hoc basis and there is almost no research on the topic of using them for waterlogged wood. They function by penetration into the first few millimetres of the wood and are employed only once the impregnant (e.g. PEG) has been removed from the vicinity of the joint with an alcohol-based solvent. As PEG will probably migrate into the region of the bond after adhesion, it is desirable that the adhesive not be adversely affected by phenomena such as plasticization or softening. The adhesive properties should be retained in the presence of PEG. There are many references in the wood technology and popular woodworkers' literature to the use of resorcinol and epoxy adhesives for waterlogged wood treated with PEG; the article by Leslie (1973) is typical.

Plastics and Rubbers

by Julia Fenn, Royal Ontario Museum

Theoretically, selecting an adhesive to repair a plastic or a rubber artifact is similar to choosing adhesives for other materials: the adhesive should adhere adequately but not excessively, and its chemical and physical properties should be, and should remain, compatible with both substrates. However, many elastomers and thermoplastics have characteristics that severely limit the choice of usable adhesives from both a practical and a conservation perspective. They do not bond well and are often not compatible with the materials used for adhesion, consolidation and gap-filling. Consequently, acceptable adhesive repairs for historical plastics and rubbers in museums are still at an early, experimental stage.

Plastics and rubbers are very versatile and come in many forms, from flexible foams to rigid laminates. They are combined with other materials to make boats, cars, art, clothing, household appliances, etc., so virtually any type or strength of bond might be required when repairing such varied materials.

Butt joins frequently need additional support because many plastics are thin-walled, or have fragile, distorted edges. With translucent or transparent plastics, both adhesive and lining need to be transparent and free of bubbles if shadows from the backing are to be minimized. Flexibility for drape is less important because clothing made from sheet polymers usually becomes quite stiff. Sewing and interleaving techniques are usually better than adhesives for repairing woven textiles made from synthetic fibres.

Consolidation is sometimes attempted to reinforce crumbling or environmentally stress-cracked artifacts. Consolidation and fills appear to be helpful in shielding cracked polyurethane foams from ongoing environmental exposure, especially if antioxidants are added to a compatible consolidant (van Oosten 2005). However, it is less successful on hard, translucent plastics such as polystyrene. The liquid consolidant can aggravate stress, be patchy and irreversibly unsightly.

Introducing the consolidant in gaseous form seems to be less stressful. Degraded plastic samples at the Royal Ontario Museum consolidated under vacuum during the 1980s as part of the Canadian Conservation Institute Parylene [poly(p-xylylene)] project have survived very well compared to controls. Plastics with acidic surfaces were the exception; cellulose acetate and nitrate samples disintegrated much faster than controls and the Parylene was blanched and weakened. A flexible poly(vinyl chloride) (PVC) doll's shoe, which was exuding plasticizer but was not acidic, ceased to ooze and can now be handled. The control shoe continued to ooze, shrank, yellowed and distorted.

Plastics adhesion during manufacture

Adhesive methods have been used in the manufacture of plastics and rubber items since the 19th century, particularly solvent and heat-welding systems that make the materials of the artifact itself act as the adhesive; for example, solvent-welded strips forming the attachment loop on early 20th-century buttons. Many of these attached loops are still firm and functional today.

Unfortunately, dissolving or melting an artifact is not an option for most conservation repairs, not only because of the ethical issues, but because solubility parameters and softening ranges of many synthetic resins and elastomers change as they age, greatly increasing the potential for damage and bond failure.

Manufacturers deal with new polymeric materials of known formulation that use tailored adhesives designed for specific purposes and that require specialized equipment for effective application. Industrial adhesives are not intended to be reversible and are often too strong for aged, museum plastics. It is unreasonable to expect them to be compatible with obsolete, degraded plastics that have unpredictable properties. Nevertheless, they make a good starting point to find what bonds a given plastic or elastomer. Information on industrial adhesives and adhesion methods can be found in Counsell (1993) and Shashoua (2008, pp. 213–220).

Properties of the plastic that can influence the choice of adhesive

Factors affecting adhesion

Many synthetic polymers, notably rubbers, nylons, fluoropolymers and polyolefins, have a low surface

tension so adhesives do not wet them sufficiently to permit good bonds. Manufacturers change the surface tension by selective degradation using ruthless methods such as flame, ultraviolet (UV) light, lasers, corona or plasma discharge, etc., which usually do not accord with conservation practices. If the operator is unskilled, the results can be uneven, misshapen and discoloured. Sanding, especially on polymers such as nylon and polyethylene, can interfere with bond fit without improving the wetting power of the adhesive.

In this context, natural aging can be useful. Weathered nylon combs and polyethylene toys have been repaired with acrylic adhesives, both dispersions and solutions, which would not have adhered before the plastics degraded. Consolidation was also attempted with dilute solutions of acrylic resins. The results allowed for careful handling, but uneven absorption of the consolidant created patchy translucency and gloss.

Oily additives also affect adhesion. Stabilizing soaps and plasticizers in PVC, residual release agents on moulded plastics and absorbed lubricants in industrial phenol-formaldehydes, which have an affinity for machine oil, can all migrate. They not only affect new joins, but interfere with pre-existing joins made before the artifact degraded. Wax resin adhesives have not been successful on oily plastics; however, modified rubber adhesives have had more success (Huys and van Oosten 2005).

Rubbers treated with silicone polish in the 1980s to improve appearance and increase resistance to oxidation are especially difficult to bond. Silicone is very mobile, so it contaminates other parts of the artifact and is difficult to remove. Unfortunately, some commercial polishes intended for use on hard plastics also contain silicone for disguising surface scratches. Such polishes are widely used by dealers and collectors, which might explain why plastics such as acrylic sculpture or cast phenol-formaldehyde jewelry that do not have an especially low surface tension sometimes fail to adhere. A useful test is to apply a tiny drop of water directly adjacent to the break; the degree of beading indicates wettability.

High viscosity in the adhesive can sometimes be used to overcome poor wetting *as long as the bond does not need much strength*. For example, thick, water-based pastes such as methylcellulose (MC) can be used to re-adhere original labels onto non-polar surfaces, if the plastic can safely withstand a short-term exposure to moisture. MC adheres surprisingly well to rubber artifacts. However, cellulose ethers are not an effective barrier against migrating components from underlying substrates, so porous paper labels re-adhered to items such as degrading PVC or vulcanized rubber eventually discolour. Similarly, viscous acrylic dispersions have been used to re-attach decals onto polyethylene Transformers toys. It is a weak bond, but adequate and mechanically reversible.

POTENTIALLY DESTRUCTIVE INTERACTION WITH COMPONENTS OF THE ADHESIVE

Adverse reactions to solvents

Plastics each have their own specific vulnerabilities. The problem is mainly with solvents, especially organic solvents, but other substances such as ammonia or organic acids used as emulsifiers in dispersions also affect many thermoplastics. Direct contact can cause immediate dissolution, disruption or blanching on the surface when all or part of the plastic artifact actually dissolves. The unsightly effect is permanent.

Aging degradation can increase a polymer's sensitivity to solvents. There is surface disruption and cracking when water is applied to light-degraded casein formaldehyde or Ebonite rubber. Both of these polymers are resistant to water before they age.

Solvents can cause stress cracking, especially in hard plastics, and is initiated by exposure to vapours. Tiny interconnecting cracks form within the body of the plastic. It is particularly noticeable in crystal polystyrene and poly(methyl methacrylate) because of their clarity and light-transmitting properties, but it can affect all hard plastics, and severely compromises their impact resistance. Stress cracking can be initiated by a single exposure to solvent vapours, but the crazing might not become visible for many weeks.

Each thermoplastic has its own vulnerabilities to different solvents, but susceptibility to stress cracking chiefly depends on their inherent stress levels. The more stressed they are, the greater the risk of crazing. Stress can be an inherent result of manufacturing processes or can be created by tensions between different components of composite artifacts. However, *stress can also be created by the repair process* if the plastic artifact is clamped too tightly or a strong adhesive shrinks.

Testing a small amount of glue or solvent in an inconspicuous area of the plastic can be dangerous and inconclusive. The test may actually initiate irreversible damage that may not otherwise appear for a long time. The issue of solvent damage to various plastics is discussed in more detail by Shashoua (2008, pp. 209–212) and Fenn (1993).

Acidic plastics

A few plastics are very acidic in the later stages of deterioration. PVC, cellulose nitrate and Ebonite can be so strongly acidic that they weaken, discolour and eventually break down any adhesive. Weaker organic acids are emitted by cellulose acetate, phenol-formaldehyde and urea-formaldehyde. They are much less aggressive, but can still contribute to adhesive discolouration and bond failure. It is advisable to test the pH of plastic artifacts before applying adhesives or consolidants.

OTHER CRITERIA IN SELECTING ADHESIVES

Adhesion and chemical compatibility between plastics and adhesives are the most important requirements and will limit adhesive choice, but there are other criteria:

RESPONSE TO TEMPERATURE CHANGE

Most thermoplastics have a high linear coefficient of thermal expansion (CLE); they are more responsive to temperature changes than metals. Thermoset adhesives (e.g. epoxy resin adhesives) usually have lower CLEs because they are crosslinked. Changes of even a few degrees can cause major stress if glues do not have either similar CLEs or enough flex to accommodate movement.

The glass transition temperature is also important. At room temperature, adhesives should have some flex and impact resistance, but should not be soft enough to creep. Rubbers and some plastics are often stored at low temperatures to slow the rate of chemical deterioration. At these low temperatures, acrylics, cellulose nitrates and many rubber adhesives become quite brittle and can fail when materials from cold storage are handled.

REFRACTIVE INDEX

Although transparency is an important property of artifacts, such as acrylic sculpture, matching the adhesive to the refractive index of transparent plastics is likely to be ineffective over the long term since plastics' colour and translucency tend to alter with age.

REVERSIBILITY

The concept of reversibility is a mantra for conservators, but it is not yet a practical option for *most adhesion* and *all consolidation* of rubbers and plastics.

IDENTIFICATION

Plastics, even those that look alike, can have very different properties. Consequently, it is very important to identify the plastic before choosing the adhesive. Rémillard (2007) has produced a useful booklet for micro-identity tests.

ADHESIVES

There has been great progress in the plastics adhesion industry in the last two decades. Adhesives are being made specifically for plastics, both hard and flexible types.

As a general rule, *thermoplastics* require adhesives with minimal solvent content, especially organic solvents. Water is also undesirable, particularly if its pH has been changed by acidic or alkaline emulsifiers. *Thermoset* plastics being crosslinked are less reactive, so they have a wider range of safe adhesives. However, if they have degraded or have poorly bound moisture-sensitive fillers, such as wood flour, water-based adhesives should be avoided. Except for the two situations mentioned earlier (degraded nylon and polyolefins), water-based adhesives are not recommended for heavily degraded plastics.

The following are adhesives in use for plastics:

ULTRAVIOLET-LIGHT-CURING ADHESIVES

UV-light-curing adhesives are usually acrylics or epoxy resin adhesives. Light of the desired intensity and wavelength must be able to reach the adhesive in the join to effect a cure. They are clear, able to bond a variety of surfaces, have no solvents and no volatile emissions. Unless specially modified, they have a low CLE and are inflexible. They are expensive and require specialized lamps with a focused beam of the correct wavelength.

ONE-PART POLYURETHANE ADHESIVES

One-part polyurethane adhesives (e.g. Excel and Gorilla Glue) cure by reaction with ambient moisture, so the open time is dependent on relative humidity. They expand and foam as they cure, forming strong joins with excellent filling properties and adhere to a variety of plastics and rubbers, including polyethylene and silicone. They are flexible, have excellent impact resistance and are manufactured in a range of CLEs. They have good resistance to acid surfaces and to cold temperatures. Three disadvantages are the initial colour, further discolouration with time and the need for very firm clamping to combat expansion during cure.

Cyanoacrylates

Cyanoacrylates have been greatly improved as adhesives for plastics and rubbers. Some are designed especially to bond with difficult surfaces such as rubber and polytetrafluoroethylene. They are available as liquids or gels with differing degrees of flexibility and gap-filling properties. However, many are pH sensitive, so not effective on acidic substrates, and some will blanch or etch thermoplastics. One irritating property is their tendency to harden in the bottle even when refrigerated and well-sealed.

Hot-melt adhesives

There are many resins, from nylon to polyurethanes, that are used as hot-melt glues for electric glue-guns. Conservators usually use ethylene/vinyl acetate copolymers because of the light colour, low melting point and their ability to adhere to polyethylene foams. They are tough and chemically inert but impossible to remove without damage to the substrate once they have bonded. Although the lower melting point varieties are safer for thermoplastics, they set up very fast, often before the adhesive has fully wetted the surface.

Epoxy resin adhesives

Two-part epoxy resin adhesives are solvent-free (not emission-free), but the uncured resin sometimes attacks thermoplastics. There is less risk with quick-setting epoxy resins, but even they usually etch very sensitive plastics, such as polystyrene, and sometimes become unacceptably hot while curing. Epoxy resin adhesives are tough and strong. Some industrial grades have been modified to give greater flex and CLE. During the 1970s, epoxy resin joins were reputed to fail when used to bond polyester glass-fibre-reinforced resins. Collector's lore also claimed poor bonds with Bakelite jewelry; however, new generation epoxy resins seem to be compatible with both. Emissions from degrading plastics seem to accelerate darkening in epoxy resin joins but this has not been properly studied. Where pale-coloured bonds are important, using optical epoxy resins might be a more promising route.

Pressure-sensitive silicone tapes

During the last decade, there have been promising developments in silicone tapes designed for surfaces with very low surface tension. Some are described by the manufacturers as reusable, which implies that they are easily removable. However, their aging properties are unknown and conservators are rightly wary about pressure-sensitive tapes. Silicones also have an unfortunate tendency to absorb organic acid vapours, so they might not be chemically inert. Each tape would have to be checked before use.

There are self-fusing silicone tapes (e.g. Rescue tape) developed for the military that have no adhesive. They bond to themselves but not to the artifact. These might be useful as conformable, clamping systems for plastics, or for temporary "holding" repairs to prevent loss or warping until a safe adhesive could be found.

Gecko glues

One potentially promising adhesive system is "gecko adhesion" (Fenn 2011). Geckos cling to ceilings by means of myriad, fine, keratinous hairs on their feet that allow intimate contact with a variety of surface textures. The adhesion is currently thought to be attributable to Van der Waals forces possibly in combination with capillary forces interacting with absorbed surface water. Gecko setae create a dry, directional, instantly reversible bond with surprising strength that requires no secretions and leaves no residue.

Gecko glues are still in the research stage, but tapes with directional adhesive nanofibres have already been produced experimentally (Jeong et al. 2009; Lee and Fearing 2008). Gecko setae have been simulated in a variety of synthetic materials including acrylics, silicone and polypropylene. A conformable, transparent, repositionable, solvent-free "adhesive" able to bond to difficult surfaces, made from chemically inert polymers, reversible with minimal stress and leaves no residue—a conservator's dream come true!

Repairing valued plastics, especially thermoplastics, with proprietary adhesives is extremely risky; there are so many unknowns. Given the difficulty in recognizing and avoiding the pitfalls and the irreversible consequences of error, it should not be undertaken lightly. It is often safer to prevent loss and differential warping by securing broken or cracked pieces to customized supports until better adhesive systems can be identified.

Ceramics and Glass

by Stephen P. Koob, The Corning Museum of Glass

Glass and ceramics can vary considerably in their composition, history, technology, deterioration and preservation, but the criteria for choosing a consolidant or an adhesive are similar. The general principles of minimal intervention and reversibility still remain as good theories of practice in selecting a consolidant or an adhesive, along with the modern approaches of *passive intervention* and *risk management*. Adequate, climate-controlled and monitored storage should also be an important factor for the long-term preservation of ceramics and glass.

Reasons for consolidation or adhesion

The primary reasons for active intervention include stabilization, prevention of additional deterioration, or strengthening of an object for additional conservation, including cleaning or adhesion (the assembly of fragments). Adhesion of fragments, or assembly, is one of the most common purposes, as separate fragments may not be presentable or intelligible. Consolidation may also be judged necessary simply to handle a fragment or object. The eventual disposition of an object, or groups of objects, should certainly be considered before any intervention is carried out, whether the object is to be housed in storage, studied, published or placed on display. The total time and cost of treatment should also be weighed, as it can be significant. Assembled objects also often require a larger storage space (shelving or cabinets) than individual fragments, which can be bagged or placed in drawers.

Consolidation

For successful consolidation of ceramics and glass, the voids and pores need to be accessible or permeable. A glazed brick may be porous, but cannot be consolidated as long as the glaze is intact (Grimshaw 1971) and may not require consolidation so long as the glaze is strong and intact. High-fired ceramics, such as china and porcelain, and solid glasses are not permeable, have minimal porosity (<1%) and do not require consolidation.

The most common consolidants are synthetic resins applied in dilute form (e.g. 5–10% w/v Paraloid B-72 in acetone). Solvent-based resins are very effective, but in some cases, a water-based colloidal dispersion may be a suitable choice.

Consolidation of earthenware and terracotta

Consolidation is primarily needed to strengthen very low-fired clay or ceramics, such as soft or friable earthenwares and terracottas, which can easily be damaged by any type of handling. The weakened nature of the material may be a result of poor manufacture (e.g. low-firing), or from deterioration subsequent to manufacture (e.g. from use, misuse, burial, chemical attack, or contamination from soluble salts). The surface of these ceramics may appear similar to unfired clay: dry, powdery, soft to the touch and with a friable or flaking surface that may become detached by handling or any movement, including air. Cracks and loose flakes or chips may accompany the weakened surface, and dirt, deposits and salt crystals may be embedded in recessed areas.

Consolidation is necessary when the above symptoms are visible and the object cannot be safely stored in a weakened condition. However, consolidation is a serious and permanent intervention as consolidant is not completely removable. Also, removal is likely to cause more loss of surface or more serious damage.

Consolidation will also likely change the appearance of a weakened ceramic as a result of saturating the surface. The surface may appear darker, with more colour. This is not necessarily a bad thing, as damaged surfaces such as blanched and deteriorated glazes of archaeological ceramics look better when consolidated. A successful consolidation will fill cracks and voids and can restore the refractive appearance of a glaze. Over-consolidation (excess resin on surface) produces a shiny, unaesthetic appearance.

Choice of a consolidant

Ideally, a consolidant should be dilute enough to penetrate and strengthen a weakened substrate (e.g. the clay body). For solution resin polymers, this translates into a solids/solvent ratio with a low viscosity. A resin concentration too low will penetrate well, but may impart little or no strength, while one too high may provide more strength, but

will not penetrate deeply. Poor penetration can result in a *surface* consolidation, which is no better than a coating. The solvent choice and application method have a significant impact on the consolidation effectiveness. Solvent resins, by their very nature, penetrate better than water-based emulsions or dispersions, but require more care and higher safety measures. Water-based consolidants do have applications, especially when large and very coarse ceramics or brickwork needs to be consolidated, and the use of solvents is almost impossible.

Paraloid B-72

Although numerous solvent resins are available, and many have been used for consolidation, Paraloid B-72 stands out as the best choice. It is stable, reversible, non-yellowing and has known solubility. It has a very low viscosity in solution, particularly at low concentrations (5–15%). It has a moderately high glass transition temperature (T_g) (40°C) and can be used safely in non-toxic solvents. It has very good strength and yet is permeable to water, if the consolidation is used as a pre-treatment to desalination.

Solvent choice is important, not only for the resin penetrability, but also as a health and safety concern. Early consolidation recommendations often suggested a very slowly evaporating solvent such as toluene or xylene, but these should not be used for two reasons. These solvents are highly toxic, and solutions have a significantly higher viscosity (Rohm and Haas 1983). Solutions in acetone or in acetone/ethanol are preferable, as they are less toxic and penetrate better. For example, Paraloid B-72 in acetone (5%) is half as viscous as Paraloid B-72 in toluene (5%), meaning the consolidant should penetrate twice as deeply. In reality, other factors may influence the penetration of the consolidant, including the application method, ambient temperature and humidity. The various application methods include brushing, spraying, immersion, and capillary draw (with and without vacuum) (see below).

Acrysol WS-24 acrylic colloidal dispersion

Another acrylic that has proven useful for the consolidation of ceramics and glass is the colloidal dispersion Acrysol WS-24 (a.k.a. Primal WS-24 or Rhoplex WS-24). It is supplied in water (36% w/v) and visually appears similar to other acrylic dispersions, but has several different properties (Koob 1981; Warson 1972). The very small particle size and charged particles keep the dispersion from settling out, and the viscosity is much lower than other acrylic dispersions. One additional benefit is the neutral pH (7.0), whereas other acrylic dispersion products are higher (8–10). The strength of the acrylic resin is similar to Paraloid B-72, and it has a similar T_g (40°C). The white dispersion dries clear, and is readily soluble in acetone.

Without question, the water-based Acrysol WS-24 does not penetrate as well as Paraloid B-72 in solution, but its advantages include the lack of solvents (no health hazard), particularly good for large applications, and lower associated costs. Because penetration is poorer, it is recommended for use on large, very porous ceramics, such as coarse earthenwares and brickwork.

Acrysol WS-24 should not be used as a pre-treatment for desalination, as the dispersion will partially re-swell in water, even after drying clear. If the ceramic is strong enough to withstand the application of a water-based acrylic, it should be strong enough to be desalinated, and the desalination should precede the consolidation.

APPLICATION OF CONSOLIDANT ON CERAMIC

Several different techniques can be used to apply either resin solutions (e.g. Paraloid B-72) or colloidal dispersions (e.g. Acrysol WS-24) on ceramics. The application method, amount of consolidant applied, and ambient environment affect the penetration and efficacy of the treatment. Each method has advantages and disadvantages, so the conservator must decide which method is most appropriate for each case.

Before any consolidation, the first assessments that need to be made are the strength of the ceramic and reason for consolidation. Weak ceramics may require consolidation for strength prior to assembly with an adhesive, bringing the fabric of the ceramic up to the strength of the adhesive. Even Paraloid B-72 is too strong for extremely weak and friable ceramics, and these ceramics benefit from a simple but complete consolidation.

The ceramic should be examined carefully, properly documented (e.g. written documentation and/or photography), and permission must be received to carry out the intervention. This may seem irrelevant, but considerable information has been lost by the omission of these steps. Furthermore, if the ceramic is an important artifact from an archaeological or anthropological site, information and samples may be required for dietary analysis, wear, use, dating or provenance. Consolidation may interfere with these analyses, and therefore should not proceed until these questions are resolved, or samples are taken for analysis.

Consolidant concentrations

The consolidant solution should be made to achieve the desired result. A very low concentration (<3% w/v) may impart little or no strength, while a high concentration (>15%) may not penetrate at all. Even so, there are very dense ceramics, such as very fine terracottas, where a 4–5% solution is ideal, and there are large coarse storage jars or bricks that have such an open porosity that a 15–20% solution, or a water-based colloidal dispersion such as Acrysol WS-24, will easily penetrate. This needs to be assessed for each class or group of ceramics being treated, and simple testing with a few different solutions often answers the question.

Several different concentrations (5%, 7.5%, 10% and 15% w/v) should be prepared, and if kept sealed, can be used and reused. Paraloid B-72 solutions can be quickly made by weighing the resin, placing it in cheesecloth tied with cotton string, and suspending it in a jar of acetone (correct amount according to desired concentration). No agitation is required; even 150 g will be dissolved in 1 L of acetone in 1 to 2 h.

For warm or hot climates, ethanol can be added (acetone/ethanol 90:10). High quantities of ethanol are not recommend because ethanol is extremely hygroscopic and attracts water to itself and the Paraloid B-72 solution.

Acrysol WS-24 concentrations are made simply by diluting the supplied Acrysol WS-24 with deionized water [e.g. water/WS-24 (9:1) gives 3.6% concentration]. The concentrations for this product should be lower than Paraloid B-72 solutions (3%, 6%, 9% w/v), to ensure maximum penetration.

Application methods

Numerous methods can be used for the application of a consolidant to a ceramic, including brushing, spraying, dripping by pipette, immersion and capillary draw. The condition of the ceramic usually indicates which method is preferable (e.g. a flaking surface is best consolidated by applying small amounts by brush or with a pipette).

Maximum consolidant penetration is almost guaranteed by using capillary action, or draw, also referred to as *wicking up*. In addition, consolidation can be done under vacuum to evacuate air trapped in the pores of the ceramic and to ensure deep penetration of the consolidant. The ceramic must be strong enough to withstand the air evacuation through the consolidant.

Drying and clean-up of excess consolidant

A consolidated ceramic may be very weak until the consolidant has fully set and all the solvent has evaporated. This can take anywhere from hours to days, and can easily be determined by weighing the ceramic. Once a constant weight is achieved, it is assumed that all, or most, of the solvent has evaporated.

For Paraloid B-72, excess consolidant can be cleaned-up easily using acetone and a soft brush, constantly rinsing the brush in fresh acetone. Cotton swabs or towelling are not recommended as they may disturb the surface.

Extreme temperature and humidity conditions

The application of any solvent or even water-based consolidant will be more difficult under extreme climate conditions. Ideally, consolidation or adhesion should be carried out in a well-ventilated, climate-controlled environment (18–24°C, 35–55% relative humidity). A high humidity (>55%) can affect the penetration and setting time of any consolidant or adhesive, possibly causing blanching and poor adhesion. Extreme high temperatures (>40°C) will result in very rapid evaporation of the solvent (or water), leading to poor consolidant penetration or improper film formation. Extreme low temperatures (≤0°C) will significantly slow down evaporation and the setting of a solvent resin, and will freeze emulsions. Modifying one's work conditions may be necessary for extreme environmental conditions.

Outdoor, on-site archaeological conservation should be avoided unless the working area is shaded (**Figure 1**). Indoor and shaded areas will be cooler and more comfortable than working in the sun, and more stable and better controlled for applying consolidant and adhesive.

CONSOLIDATION OF CHINA, PORCELAIN AND GLASS

High-fired ceramics such as china and porcelain can almost be classified as glass for the purpose of choosing an adhesive or consolidant, as their physical natures are closely related. China, porcelain and glass are virtually non-porous, but deteriorated or weathered surfaces may require consolidation. Archaeological china, porcelain and glass with fragile, friable or lifting weathering layers may require consolidation. However, the glass itself and the high-fired ceramic bodies of china and porcelain are non-porous, and an application of a consolidant would simply coat the surface.

Figure 1. Assembly of large ceramic vessel outdoors using Paraloid B-72 adhesive.

In some cases, weathered layers are so thin and fragile that touching the surface causes loss, and even a light wind or uncontrolled breath can cause the layers to lift and fly away. A dilute solution of Paraloid B-72 (10–15% w/v in acetone or acetone/ethanol 90:10) is recommended. The consolidant can be introduced by brush or pipette. Spraying and immersion are not recommended, as these techniques may cause the weathering to be lost. It is often sufficient to introduce a small amount of consolidant along the edges, and allow it to be drawn in by capillary action. In this manner, the consolidant gently finds its own way through the open and permeable layers, strengthening and binding the layers as it dries. Paraloid B-72 is also recommended as a surface consolidant for deteriorated glass paint on stained glass (Chapman and Mason 2003).

Entire consolidation is not recommended as this "seals" and coats the surface of the china, porcelain or glass, and may trap moisture underneath. This can potentially lead to other stabilization problems.

Excess can be cleaned-up using acetone and a soft brush, especially if the consolidant drips onto an area of the ceramic or glass that does not need treatment. Reversibility is not always practical, as the consolidated area might swell and dislodge. Even so, Paraloid B-72 is a much more logical choice than a less stable resin that may discolour over time.

The acrylic dispersion Acrysol WS-24 has been recommended for use as a pre-treatment consolidation for waterlogged glass (Roberts 1984).

Adhesives for ceramics and glass

Adhesives are primarily used to assemble broken fragments of ceramics or glass. Numerous adhesives have been recommended, but few provide consistent results. Some guidelines (Koob 2006) for selecting adhesives are that the adhesive should:

- not damage the ceramic or glass, either during application, curing or aging;
- bond well;
- be removable or reversible without damage;
- be "water" clear;
- remain stable (Class A resin—100 years); and
- be easy to use with consistent results.

Two completely different adhesives are recommended for the repair of ceramics and glass: Paraloid B-72 and epoxy resin adhesives. Although there are many others, these have become the most popular and familiar. Paraloid B-72 is the acrylic copolymer discussed above for consolidation, but for this application is made up as a thicker resin/solvent solution that sets by loss of solvent. It has a moderate strength, which is especially suited for earthenwares and terracottas, but can also be used on stoneware, porcelain, china and glass.

Epoxy resin adhesives are more complex thermosetting resins. They are two-part systems, consisting of a *resin* and a *hardener*, and when mixed, crosslink over time to a solid, insoluble polymer. There are several very good quality "conservation-grade" epoxy resin adhesives (**Table 1**) whose working properties, setting times and stabilities vary (Koob 2006).

Paraloid B-72 and epoxy resins have advantages and disadvantages. They behave very differently, and require significant experience to master. The advantages of Paraloid B-72 adhesive include the above-mentioned stability, reversibility, fast setting time and low cost. In addition, Paraloid B-72 is a thermoplastic resin, so joins can be easily moved after setting using a hair dryer. The only notable drawback, specifically on glass, is the fine air bubbles that develop within the join as a result of solvent loss. For this reason, the epoxy resins are preferred for clear glasses.

Epoxy resin adhesive disadvantages include the potential to yellow, particularly when exposed to high light levels (Down 1986), extremely high

TABLE 1. EPOXY RESIN ADHESIVES COMMONLY USED IN CONSERVATION

Epoxy	Refractive index	Viscosity (mPa.s)	Working time (hours)	Setting time (hours)	Stability (years)
Hxtal NYL-1	1.52	200–300	8–12	48–96	≤100
Epo-Tek 301	1.539	100–200	2–4	4–8	≤50
Epo-Tek 301-2	1.564	300–600	6–8	16–24	≤100
Fynebond	1.565	moderately low	8–10	24–35	≤50
Araldite 2020	1.553	130	6–10	16	≤50

strength, very long setting times, short shelf life and cost (Skeist 1962) (**Figures 2** and **3**). Epoxy resin adhesives are also virtually insoluble when cured and very difficult to remove.

PREPARING PARALOID B-72 FOR USE AS AN ADHESIVE

Several papers have been published detailing the preparation and use of Paraloid B-72 adhesive for both ceramics and glass, including detailed recipes and step-by-step instructions (Koob 1986, 1996, 1999, 2006, 2009). The commercially available Paraloid B-72 has been modified with cellulose nitrate (Nel and Lau 2009), so it is highly recommended that Paraloid B-72 solutions be made fresh by conservators, especially for ceramics or glass.

PREPARING EPOXY RESINS FOR USE AS AN ADHESIVE

In general, epoxy resin adhesives do not require any special preparation. They are supplied in two parts. One should always read the manufacturer's recommendations for their use, their materials safety data sheets and the specifications for their shelf life and storage. Epoxy resins are slightly toxic during handling, so gloves should be worn when mixing. Cured epoxy resins are not deleterious to health (Skeist 1962, p. 327).

APPLICATION OF PARALOID B-72 AND EPOXY RESIN ADHESIVES

Careful planning is required before any repair or assembly is undertaken (Errett 1972), no matter what the adhesive. All fragments should be clean, dry and arranged in a logical order for assembly. This is particularly important for epoxy resin adhesive joins, as they cannot be moved once the epoxy has cured. It is generally suggested that a "dry-run" be carried out to see in what order pieces should be joined. This can be done with tape, sticky wax (applied across joins) or very small metal staples temporarily tacked across joins using a cyanoacrylate adhesive.

Figure 2. Yellowing of epoxy resin adhesive and fill.

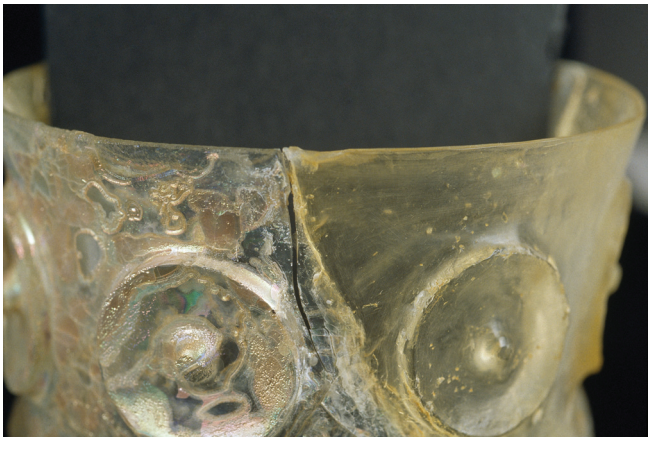

Figure 3. Damage to glass from epoxy resin fill.

TECHNIQUES OF ASSEMBLY

Application of Paraloid B-72

There are several methods and techniques of Paraloid B-72 adhesive application on both ceramics and glass (**Figure 4**). Once the order of assembly is

Figure 4. Roman hexagonal jug before (left) and after (right) treatment with Paraloid B-72.

determined, the fragments are arranged in the order that they will be joined. Porous ceramics should be primed or sealed before the Paraloid B-72 adhesive is applied, but non-porous ceramics and glass do not require this step. The adhesive is then applied along one edge of the fragment to be joined and a "piece-by-piece" assembly is carried out, checking all joins as the assembly progresses. Depending on the size and weight of the fragments being assembled, joins may be held, taped or positioned until the adhesive has set. The time required for the Paraloid B-72 to set varies considerably (e.g. minutes to hours) with the size and thickness of the fragments being assembled and ambient temperature. A binocular microscope is a valuable tool in examining the joins to ensure that they are correct.

A more advanced technique that works well with very thin china, porcelain and glass, but not with earthenwares or terracottas, is to completely assemble all the fragments with tape or small clamps across the joins and introduce the Paraloid B-72 along the cracks. This requires thinning the adhesive (25–30%), but can be very effective. It is commonly used for epoxy resin applications (see below).

Application of epoxy resin adhesives

Epoxy resin adhesives are generally not recommended for porous ceramics such as earthenwares, terracottas and soft-paste glazed wares, since the epoxy resin soaks into these porous fabrics and causes irreversible darkening and staining. They are best suited for high-fired ceramics and glass.

Epoxy resins are very different than solvent-resin adhesives, and new techniques for their application must be learned (Koob 2003). They can give beautiful repairs, and can be selected to match a specific refractive index of glass, but require absolute care and attention in their preparation and use (**Figure 5**).

Epoxy resins can be applied using similar methods as Paraloid B-72 (see above), either by piece-by-piece direct application or by capillary draw, which is generally preferred. Precise weighing out and mixing is important for dependable results. Epoxy resins have also been successfully used to stabilize cracks in high-fired ceramics and glass. The epoxy resin adhesive should be used within the first hour of mixing, when its viscosity is lowest.

Excess epoxy resin should be cleaned off the surface of a ceramic or glass as soon as possible after application, otherwise, very strong solvents or mechanical removal may be necessary, which poses a risk of damaging the surface. Uncured epoxy resin adhesive can be easily cleaned off using acetone or ethanol.

Work environment for adhesive use

The difficulty of working in hot climates has been mentioned, but it should also be noted that neither Paraloid B-72 nor any epoxy resin adhesives will provide consistent and dependable results if the humidity is unduly high. If the humidity exceeds 60%, it is difficult to work with any adhesive.

Figure 5. Glass lid previously repaired with cellulose nitrate (left) re-treated with Hxtal NYL-1 (right).

Fossil Vertebrate Specimens

by Amy Davidson, Lisa Kronthal Elkin and Samantha Alderson, American Museum of Natural History

Paleontological collections contain many types of fossils. The following section will focus on one particular category: vertebrate remains preserved in sediment (matrix). Fossil vertebrates are typically found encased in a matrix, with the fossil and matrix treated as a single specimen. Paleontological specimens are often treated in two phases: initially in the field to ensure stability for excavation and travel, and later in the lab where the matrix may be partially or completely removed to expose the fossil for research or exhibition. Adhesives are often an essential part of successful treatment in both the field and lab. They are most commonly used for consolidation, joining and gap-filling.

Adhesives are used in fossil preparation for many tasks, and the specimens to which they are applied can vary in their properties, including size, density and strength. Adhesive choice and application method will depend on the physical and chemical properties of the specimen, the type of treatment required (e.g. gap-filling, consolidation, joining), the properties of the available adhesives, and the demands of the researcher. In addition, preparation often requires reworking adhesives used on both older and newly excavated specimens, which must be taken into account when choosing an adhesive.

Although treatment is often necessary, in some cases adhesives are detrimental to scientific research. Adhesives can contaminate analyses or obscure surface features (Tuross et al. 1994; Wittmeyer and Schweitzer 2006, p. 140A; Fernández-Jalvo and Monfort 2008). Reconstructions and cosmetic repairs can mislead researchers. To avoid these problems, non-adhesive supports, computerized tomography, rapid prototyping, surface scanning and digital reconstructions are increasingly used as supplements or alternatives to traditional adhesive treatments.

Properties of the specimen that can influence the choice of adhesive or consolidant

Scale and shape

From massive sauropod limb bones to tiny shrew ear ossicles, vertebrate fossils range a great deal in size and shape, significantly impacting treatment decisions. Consolidating or joining extremely small specimens presents specific challenges and solutions (Madsen 1996). These treatments require precise application of minute quantities of adhesives, as excess can obscure surface detail and can be difficult to remove without damaging the specimen. Very large, heavy specimens often require exceptionally strong adhesives, particularly for joins and fills. Fragmentary specimens or individual skeletal elements such as ribs, tusks, flanges and frills may present awkward and unwieldy shapes. Inherent stresses in such shapes make them likely to break, and prone to re-breaking after repair. Treatment strategies may include consolidating parts to strengthen them before joining, or allowing the join to move or fail without creating fresh breaks by choosing weaker, more flexible adhesives. Alternatively, it may be preferable to build a support to hold the fragments in place rather than joining with adhesives.

Permeability and density of fossil and matrix

Fossil bones and matrices vary widely in hardness, strength and porosity. Some specimens are very fragile due to natural porosity or cracks and gaps resulting from erosion, while others are extremely dense and hard. Weak, porous specimens generally require softer, more flexible adhesives that are easily reworkable, allowing continued separation from the matrix without risk of damage. Fossils in very hard, dense matrices are frequently retrieved by quarrying, rejoining and air-scribe preparation. Joining quarried pieces requires an adhesive that will gap-fill where bone is missing, compress to a thin film, and set very strong, allowing the bone to resist the force and vibrations of subsequent preparation. Very dense specimens often have hairline cracks, calling for an adhesive with excellent wicking ability and high strength. Some dense matrices can only be separated from the bone by dissolution in acids or other chemicals. This type of preparation requires surface consolidants or coatings that can resist attack and remain adhered through multiple baths and rinsing (Lindsay 1987).

Sensitivity to moisture and relative humidity

Many types of fossil specimens are unstable and suffer damage due to changes in moisture content and relative humidity (RH). These include fossils with layered structures and/or an organic component, such

as tusks and subfossil bone, those collected when wet or damp, specimens in hygroscopic matrices such as clay or shale, and fossils containing salts and/or pyrite. Resulting damage may include spalling, delamination, crumbling and breakage. These problems are often addressed by applying adhesives, with varying degrees of success (Child 1994; Newman 1998; Rixon 1976). Before using adhesives on these types of specimens, it is essential to address the root cause of the damage and instability, e.g. by removing soluble salts or drying specimens at an appropriate rate and, above all, by providing a stable storage environment.

Previous treatment

Vertebrate fossils in collections have often undergone multiple treatment campaigns. Past treatments are frequently undocumented, complicating subsequent adhesive treatments. New treatments (e.g. coating for moulding) can disturb or destabilize past treatments; this is especially critical for tiny or thin joins that easily redissolve. Specimens that have been moulded frequently retain traces of old Plasticene fills, waxes or mould-release agents that may interfere with repairs and re-treatment. Also, contamination with old wax-based adhesives in joins and fills can inhibit adhesion. These difficulties are among the many reasons that all fossil treatments, including adhesive applications, must be fully documented.

Treatment conditions in the field

Fossils are often treated in field conditions outside a lab setting. The outdoor environment presents its own set of challenges, including extreme environments (e.g. high temperatures and RH, cold, rain, wind) and limited access to some materials (e.g. adhesives, solvents, tools). These factors may significantly impact the selection of adhesives and application methods. Preparation tasks may require different adhesives depending on whether they are performed in the lab or on-site.

Consolidants and adhesives for fossil vertebrate specimens

Consolidation

Important factors to consider for the consolidation of fossil specimens include degree of penetration, reworkability, strength and stability. Depth of penetration depends upon several properties, including viscosity, set-time and ability to wet the surface. Achieving deep, even consolidation of an appropriate strength may be difficult. Patchy, overly hard consolidation can be worse for a weak specimen than no consolidation at all. Fossil preparation often requires temporary consolidation before or during removal of the matrix and this can result in thick, glossy, superficial coatings. It is crucial that excess consolidated matrix and surface applications can be removed safely as treatment progresses, without damaging the bone. The long-term stability of consolidants must also be considered since, even if adhesives are used for temporary stabilization during preparation, they often persist as part of the completed specimen.

Figure 1. Consolidation with Butvar B-76.

Solutions of synthetic resins in solvent are regularly used for fossil consolidation, including poly(vinyl butyrals) (PVBs) such as Butvar B-76 or Butvar B-98 (**Figure 1**), acrylics (most commonly Paraloid B-72), and poly(vinyl acetates) (PVACs), such as McGean B-15 (formerly Vinac B-15). These resins are versatile. Their properties may be manipulated by varying solvents, concentrations and application techniques. They remain soluble after setting and are thus easily reworkable or removable. Most have been proven stable over time, guaranteeing reworkability in the long term. They do, however, have a tendency to migrate to the surface during setting, making deep penetration difficult. Surface consolidants with low glass transition temperature (T_g), such as some of the lower molecular weight PVACs, can become sticky and entrap dust, especially in hot storage conditions (Horie 1987, p. 92).

Wet or damp specimens require aqueous consolidants, usually acrylic dispersions (e.g. Acrysol WS-24, Primal WS-24, Rhoplex B-60A). These adhesives are not water soluble after setting, which can limit their reworkability. They also have short shelf lives and stock needs frequent replacement, which can be expensive and inconvenient. When an aqueous system is required, acrylic dispersions are preferable to PVAC dispersions, especially commercial products like Elmer's Glue-All, which are acidic, contain many additives and are likely to age poorly (Down 2009).

Cyanoacrylates are more prevalent as consolidants in fossil preparation than in any other area of conservation. As a very low viscosity material, these undiluted liquid monomers have a superior ability to penetrate into many porous fossil substrates and wick into hairline cracks. Lacking a solvent component, cyanoacrylates deposit more polymer than solution adhesives and, unlike solution adhesives, do not tend to reverse migrate. However, the deep penetration achieved with cyanoacrylates is not always necessary, and may be undesirable in some instances, as when contamination of interior bone must be avoided (Tuross et al. 1994). Cyanoacrylates also have many other potentially undesirable properties. Chief among them is tenacity; once set they can be very difficult, if not impossible to remove or rework safely, and may permanently bond matrix to bone. They are extremely hard and can shrink upon setting, and if applied to very weak, porous specimens, may cause damage (Vestergaard and Horie 1996). The low viscosity cyanoacrylates have poor gap-filling properties and, in some cases, may achieve deep penetration without effective consolidation (e.g. specimens with open cracks). Only a few investigations into the aging properties of cyanoacrylates applied to fossils have been published and these have shown that cyanoacrylates may deteriorate faster on pH-neutral or alkaline fossil substrates (Down et al. 2006). The use of cyanoacrylate accelerators, such as sprays, for an instant bond is not recommended because of their tendency to turn fossils bright green, yellow or blue, reportedly in reaction to iron (Howie 1984).

JOINING AND GAP-FILLING

When choosing an adhesive for joining or creating fills for fossil vertebrates, appropriate strength and long-term stability are priorities. Used for joins on a wide variety of objects including fossils, the acrylic resin Paraloid B-72 is a good all-purpose adhesive with moderate strength and excellent aging properties (Koob 1986). It can also be bulked with a variety of materials to create a range of useful fill materials (Larkin and Makridou 1999; Wolf 2009). PVACs can be used for joining, although some have a T_g close to room temperature and thus may fail if the join is under stress, or the specimen is stored in a hot environment (Horie 1987, p. 92). PVBs may be used to create bulked fills and supports that are easily re-soluble. The removability and stability of all of these solution resins are advantageous, because assembled fragments frequently need to be adjusted or disassembled to correct mistakes or to accommodate a new fragment.

Solution resins are not appropriate for all fossil joins or gap-filling. Joins on large, heavy specimens may require stronger materials, such as epoxy resin adhesives, and structural fills may necessitate the use of commercial epoxy putties. Unfortunately, epoxy resin adhesives can be difficult to remove or rework. Using a soluble barrier layer (e.g. Paraloid B-72) can alleviate this problem. This is a common technique for joining stone that has been proven strong and effective (Podany et al. 2001). Like dispersions, liquid epoxy resin adhesives have inconveniently short shelf lives. Epoxy resin adhesives also vary widely in their stability, and it is important to use a well-tested, stable epoxy. Low-end commercial products, such as five-minute epoxies that are likely to age poorly, should be avoided (Horie 1987, p. 173; Down 1986).

Especially for joins, working properties such as tack and setting time must also be considered when selecting an adhesive. Paraloid B-72 can be formulated in acetone to have good tack and fast set-time, making it useful for quick assembly of fragments that are difficult to prop or clamp. However, evaporation of solvent from joins with large bonding surfaces can be slow. Paraloid B-72 can take a very long time to set completely, which may result in slumping,

particularly with cantilevered joins of large, heavy parts. Very small joins present their own challenges. Tiny drops of solution adhesives have high surface tension and very fast set-time, making them difficult to control in micropreparation. A slower set-time is often preferable for very small joins. Tiny drops of epoxy resin adhesives have conveniently long set-times, allowing time for adjustment (Amaral 1994). In such cases, the advantage of slow set-time in epoxies must be weighed against their strength and insolubility, which can be disadvantageous.

APPLICATION METHODS

The most common application methods used for consolidation are squeeze-bottle, dropper or brush. Penetration of solution adhesives can be improved with several application methods, including pre-wetting with solvents, covering to retard evaporation, and use of vapour chambers. Many types of fossils and matrices weaken when wet, so immersion, and immersion under vacuum are uncommon (Rixon 1976). Squeeze-bottles, tubes and brushes are used to apply adhesives for joins. Many conservators and preparators use self-loading tubes to dispense viscous solution adhesives such as Paraloid B-72 (50% in acetone—"Koob Tube") (Koob 1986). Precise application of low viscosity cyanoacrylates may be accomplished by wicking up drops with fine tweezers or by scratching up wetted cardboard fibres with a needle (Amaral 1994).

Most gap-filling mixtures used on vertebrate fossils, whether commercial products such as epoxy putty or viscous mixtures of liquid adhesives and bulking agents, are simply poured or pressed into place with a spatula. Occasionally, fills are easier to apply by pouring a dry bulking agent into cracks, followed with a consolidant. Unless the fills are temporary (e.g. to stabilize the matrix during preparation), the surfaces are usually tooled to look neat and distinguishable from the real surface of the specimen. Fills are occasionally toned, but exact colour matching and elaborate cosmetic manipulations are generally not called for when treating fossils.

Stone

by George Wheeler[1], Historic Preservation Program, Columbia University

The repair of stone with adhesives has a long history. The use of natural materials such as shellac, animal glue, casein, beeswax and pine resins were regularly employed for stone sculpture repair by the late 17th century and continued well into the 20th century. The landscape changed with the development of synthetically modified natural materials or fully synthetic resins starting in the late 19th century. By the mid-20th century, every class of adhesive currently in use for stone conservation had been formulated.

The repair of stone with adhesives comprises a number of important activities. Of these, the most common is the rejoining of fractured or mated surfaces. However, this activity is poorly represented in the conservation literature (Jorjani et al. 2009). On the other hand, the less common activity of consolidation of fragile stone with materials typically thought of as adhesives is well-covered in the literature.

Practicing conservators are quick to point out that there is not a well-defined division between the use of adhesives to glue pieces of stone together, whose surfaces are well matched, and their use to bridge gaps across poorly mated surfaces and to bind these surfaces together. In fact, almost all of the adhesives discussed below are also used as fill materials, independent of their joining abilities.

A survey of the literature and a poll of these same practicing conservators indicate a relatively narrow range of adhesives in current use. These include acrylic resins in the Paraloid series, epoxy resin adhesives of varying viscosities, and limited use of polyester and poly(vinyl acetate) (PVAC) resins. For consolidation of stone, commercial formulations are based on alkoxysilanes and ad hoc formulations on acrylic resins or on mixtures of acrylic resins and silicone resins.

Factors influencing the choice of adhesive for joint repairs

Strength and reversibility

One of the first considerations for stone repair is adhesive strength. Material scientists use the term *toughness* to describe the resistance of newly formed joints to crack growth. Clearly, toughness or strength is paramount with dense materials like stone, which ranges in density from 1600 to 3000 kg/m^3. The introduction of polyester resins and epoxy resin in the 1940s and their nearly instantaneous adoption as conservation materials appeared to have put the strength issue to rest. The nagging issue for conservators is that these adhesives are difficult or impossible to reverse. Indeed, the rapid uptake of polyester and epoxy resins for stone conservation applications may have been largely founded on their successful and wide-spread use in industrial and construction applications where high strength was essential, and reversibility not just a lesser concern, but undesirable. Apparently you can have strength or reversibility but not both.

Setting mechanisms and setting times

The move to high strength, thermosetting adhesives such as epoxy resins and polyester resins for stone repair could be viewed as surprising given the hundreds of years of success with natural adhesives such as shellac, animal glue, casein, beeswax and pine resins. These adhesives can generally be reversed, some with more difficulty than others. While we often speak of superior strength of thermosetting resins, their short setting times were even more attractive, limiting the length of time stone elements needed to be supported while the adhesive cured.

Our current selection of an adhesive for joint repairs starts with the fundamental question: do I employ a thermosetting or thermoplastic adhesive? For conservation practice, this decision can be framed more concretely: do I employ catalyzed organic resins that set relatively quickly through crosslinking and consequently develop high strength, but cannot be dissolved in solvents after setting, or do I employ resins that are dissolved in solvents at the time of application, rely on the relatively slow process of solvent evaporation to set and have low strength, but can be redissolved in solvents many years hence?

[1] The following conservators are acknowledged for sharing their experiences and knowledge of stone conservation: Kent Severson, Mark Rabinowitz, Trevor Proudfoot, Irving Slavid, John Griswold and Tom Roby.

CONFIGURATION OF THE JOINT

In reattaching pieces of stone, the joints may lie parallel to the floor or ground, perpendicular or at an angle. The forces experienced by the joining surfaces are consequently in compression (such as a sculpture broken straight across the knee), tension (such as a sculpture with an unsupported arm hanging downward and broken straight across the elbow), flexure (such as a sculpture broken vertically at the elbow of an extended arm), or shear (such as a sculpture broken at a 45º angle at the knee), respectively. The differently configured joints may require adhesives of different strengths: shear, flexure and tension generally needing stronger adhesives than compression. Many such joints also require dowels or pins (see *Dowels and pins* below). Setting time may also come into play. It is more difficult to hold pieces of stone in position for long periods of time when they are in shear, flexure or tension, so shorter setting times are desired.

SOLVENTS FOR THERMOPLASTIC RESINS

When employing thermoplastic resins such as acrylics or PVACs, the properties of the solvent influence both their short- and long-term performance. Of course, the first consideration is that the solvent dissolve the resin. Fortunately, many acrylic and PVAC resins are soluble in a wide range of solvents from alcohols, ketones and cellosolves to chlorinated solvents and aromatics. In recent decades, there has been a shift away from aromatic solvents (e.g. xylenes and toluene) for such resins as Paraloid B-72, largely for reasons of human health. The aromatic solvents were selected for their lower vapour pressures or evaporation rates that allowed for extended working times, and their abilities to easily wet surfaces due to their lower surface tensions. Today, in most conservation applications, they have been replaced with mixtures of acetone and ethanol. Not only are these safer for humans, their higher vapour pressures and evaporation rates help to minimize residual solvent in the resin that can lead to creep in an adhesive.

VISCOSITY

One of the advantages of solvent/thermoplastic resin adhesive mixtures is that the viscosity of the solution can be adjusted to the application. For repair applications, the formulation should wet the surface of the stone. Solutions can be made dilute enough for less porous substrates, such as marble or granite, through the addition of more solvent. Higher porosity substrates, such as some sandstones, limestones and volcanic tuffs, may require less solvent or even bulking with fumed silica to achieve the desired viscosity. Consideration should also be given to the evaporation rate of the solvent. For high vapour pressure solvents, such as acetone, the initial viscosity may be just right, but the rapid loss of solvent through evaporation allows the viscosity to rise rapidly, which limits good wetting and generates a stringy adhesive with high surface tension.

For catalyzed thermosetting resins, such as epoxy resins and polyesters, dilution with solvents can compromise curing and is generally not recommended by manufacturers. For that reason, the lower limit of viscosity is set by the selection of the product itself. For epoxy resins, there are a few good low viscosity choices, but polyester adhesives generally run from syrup to peanut butter in viscosity. Both epoxy resins and polyester adhesives can be bulked to achieve higher viscosities.

PRESSURE, JOINT THICKNESS AND COVERAGE

Another important consideration in the use of organic resins for joining repairs is the joint thickness. Unfortunately, the conservation literature does not provide clear directives on this point. Bradley (1984) posited 30 μm as the lower limit of a bond thickness, indicating that smaller joints are starved of adhesive. No mention is made of bonds thicker than 30 μm. Podany et al. (2001) suggested that bond thicknesses of 100–300 μm are commonly accepted and that bonds thinner or thicker have too many voids, causing weaknesses in the adhesive layer. For objects with numerous joints, such as a sculpture leg broken in several places, such bond thicknesses could lead to displacements in excess of 1 mm over the length of the leg. In contrast, Jorjani et al. (2009) directly measured bond thickness ranging from 20–50 μm for stable joints in marble repaired with a range of typical thermoplastic and thermosetting adhesives.

The pressure applied to joining surfaces with adhesive will affect the bond thickness and therefore the amount of adhesive retained in the joint. In the case of the marble, pressures ranging from 0.03 to 1.4 MPa have been applied. At 1.4 MPa, no adhesive remained and the mating surfaces simply fell apart. At 0.03 MPa the mating surfaces could be easily pulled apart while at 0.7 MPa—the pressure ultimately chosen—the surfaces were difficult but not impossible to disjoin. The ideal pressure is related to the viscosity of the adhesive and to the porosity of the substrate, and must be adjusted for both in field application.

To achieve the maximum effect of any adhesive system, complete coverage of the joining surfaces is essential. Any gap is a flaw where cracks can initiate at lower energies and propagate through the adhesive layer. Alternatively, it may be desirable to create joints that do not maximize the strength or toughness capabilities of a given adhesive. For this circumstance, incomplete coverage of the joining surfaces will reduce the overall toughness of the joint.

Environmental considerations

For most indoor environments, fluctuations in temperature and relative humidity (RH) and exposure to ultraviolet (UV) light have little effect on the adhesive repair systems mentioned so far. In extreme indoor conditions or in outdoor environments, other factors come into play. The most important of these are hydrolytic stability and glass transition temperature (T_g). Because the bulk of an adhesive serving to join two surfaces is not exposed to light, stability with respect to UV or solar radiation is not a very important factor.

Hydrolytic stability refers to how well a material will hold up when exposed to water or high humidity. Of the adhesive systems mentioned so far, epoxy resin adhesives have the greatest hydrolytic stability followed by acrylic resins. PVACs and polyester resins exhibit poor stability.

As for T_g, it will be no surprise that thermosetting resins, such as epoxy resins and polyester resins, perform well, but some acrylic resins have T_gs as high as 105°C. Materials become plastic when the T_g is exceeded so that a joint may fail if exposed to high temperatures for long enough periods of time.

Dowels and pins

Adhesives can be used to join pieces of stone in conjunction with pins or dowels. Pins and dowels are commonly used to counteract shear, flexural and tensile stresses that may be considered too high for the adhesive alone. In fact, for thousands of years, large pieces of stone were mechanically joined using iron pins surrounded by lead that had been either poured in the molten state or hammered in place, a procedure today achieved with lead wool. In current practice, pins are set with thixotropic or putty-like polyester or epoxy resins, or with plaster or cement mortars. To provide for reversibility, stone is first coated with shellac or a thermoplastic resin as barriers to the thermosetting resin or inorganic mortar (Podany et al. 2001). The bulkier materials are employed to fill space and provide physical support rather than for specific adhesive properties.

Factors influencing the choice of consolidant

Background

As new adhesives became available in the 20th century, they were adopted as consolidants for fragile stone. Early 20th-century museum stone consolidation efforts relied primarily on drying oils, such as China wood oil, or other natural materials, such as beeswax, but these were quickly replaced with paraffin wax and later with a range of organic resins. In current practice, that range has narrowed to a few acrylic resins. From the late 19th century onwards, outdoor stone work was more commonly treated with alkoxysilane systems based on ethyl silicate or mixtures of acrylic and silicone resins—a practice that continues to this day.

Viscosity and depth of penetration

One of the most important considerations in the selection of consolidants for stone is the viscosity of the system. Stone comprises porous networks that must be penetrated by the liquid consolidants. Viscosities must be below 2–3 x 10^{-6} m^2/s to provide adequate penetration into the smaller pores. One centimetre is considered a minimum depth of penetration, although filling the entire deteriorated zone is considered ideal.

The advantage of thermoplastic resins is their ability to be diluted with solvents to create the desired viscosity. The dilution factor is much higher for consolidants (1–10% w/v) to provide good penetration. Thermosetting resins, such as epoxies, have also been used for consolidation, and even the low viscosity versions must be diluted to provide adequate penetration.

One of the attractive features of alkoxysilane consolidants is that their inherent viscosities are quite low so that no dilution is necessary. Monomers can be as low at 0.4 x 10^{-6} m^2/s, and oligomeric commercial systems range from 0.8–2 x 10^{-6} m^2/s.

Selection of solvent

A distinct advantage of alkoxysilane consolidants is that no solvent is necessary. For organic resins or polymers, either thermoplastic or thermosetting, a solvent must be selected with compatible solubility parameters, viscosity, vapour pressure and safety considerations. The solvent system must dissolve the active consolidant and provide low enough viscosity for adequate depth of penetration. Once achieving that depth and as the solvent evaporates, it must leave the consolidant in place. Solvents that hold on too

tightly to the resin (solubility parameter too similar to resin) and solvents with high vapour pressures will draw the consolidant back to the surface during evaporation (reverse migration).

INFLUENCE OF STONE TYPE AND MINERALOGY

An attractive feature of acrylic polymers is that they exhibit good adhesion to a wide range of substrates. Geologists often divide up the stone world between rocks made primarily of silicate minerals, such as quartz and feldspars, and rocks made of carbonate minerals, such as calcite. Thus, a polymer such as Paraloid B-72 performs equally well as both an adhesive and a consolidant on sandstone, limestone, granite or marble. Conversely, alkoxysilane consolidants generally perform better on silicate rocks than carbonate rocks because of the chemical compatibility between silicate polymers and silicate minerals. Nonetheless, they are frequently used on limestones and marbles because of their hydrolytic and UV stability (see *Consolidants* below).

ENVIRONMENTAL CONSIDERATIONS

Consolidation of stone is performed in both indoor and outdoor environments. As mentioned above, in controlled environments such as museums many materials have been used for treating fragile stone. In recent decades, treatment materials have been limited to catalyzed alkoxysilane treatments and acrylic resins dissolved in a range of solvents (same concerns as above). In outdoor environments with exposure to solar radiation, changes in humidity and temperature, and rainfall, consolidation treatments are limited to a narrow range of alkoxysilane formulations or mixtures of acrylic and silicone resins. The advantage of alkoxysilane-based consolidants is that the resulting gel, essentially silica gel, is stable with respect to UV light and against hydrolytic or other chemical breakdown in a wide pH range, and has a high T_g. Organic polymers have lower resistance in outdoor environments, although some acrylic resins exhibit good stability. The lower T_g is a concern due to the increased rate of soiling as the T_g is approached or exceeded. An often asked question about consolidants is whether they should also exhibit water repellency in addition to their strengthening capabilities. Most practitioners opt for a separate water repellent treatment rather than attempting to achieve both with one treatment formulation.

CONSOLIDATION AND REVERSIBILITY

As with all conservation treatments, the issue of reversibility must be addressed. Alkoxysilane consolidants create crosslinked silicate networks similar to silicate minerals found in nature. This silicate network is responsible for the stability of the gel and cannot be removed once it has formed. In contrast, thermoplastic resins can be redissolved in solvents after they have been deposited, even many years after application in outdoor environments. However, consolidation treatments are generally carried out on fragile stone so that attempts to remove the consolidants lead to further damage. The irreversibility of alkoxysilane consolidants and the risks associated with removal of thermoplastic resins have led to the notion of *re-treatability*, i.e. that a current treatment does not preclude future treatments with the same or other materials. To a large degree, both consolidant types have demonstrated their receptivity to later treatments.

FACTORS INFLUENCING THE CHOICE OF FILL

Griswold and Uricheck (1998) provide an excellent review of loss compensation methods and materials for stone that is still highly relevant. Many of the issues that apply to adhesive repair and consolidation are also relevant for plastic repair (fills).

For indoor applications, hydrolytic and UV stability take on less importance, but even in these environments, polymers will change colour over time, in part, because so much more surface area is exposed compared to applications as adhesives or consolidants. Both thermoplastic and thermosetting resins have been used indoors with a wide range of fillers to meet the aesthetic requirements. Due to the controlled environments in which conservation treatments take place, setting time is also less of a factor.

In outdoor environments, all of the above-mentioned factors come into play, namely hydrolytic stability, UV stability and setting time. Rapidly curing polyester resins can be a great advantage outdoors. However, their widespread use is surprising given their susceptibility to water and light damage. More recently, epoxy resins have taken over the primary position for plastic repair because of their greater stability. Similar to their use for pins and dowels, barrier coats of thermoplastic resins are often applied to stone surfaces before plastic repair to ensure reversibility—a practice not limited to outdoor treatments.

There is no fixed boundary between materials used strictly as adhesives and those used to fill gaps.

This is particularly true of archaeological objects or broken outdoor sculptures or monuments in which mating surfaces have experienced wear and loss. To bridge these gaps, adhesive materials are often bulked with inorganic particles. Alternatively, cement, hydraulic lime-based mortar or plastic repair materials can be modified with casein or water-compatible acrylic dispersions to provide better adhesion during initial application.

Summary of adhesives and consolidants currently used for stone conservation

Adhesives and fills

Acrylic resins

Paraloid B-44, Paraloid B-48N and Paraloid B-72, and mixtures of Paraloid B-48N and Paraloid B-72 are much stronger than normally expected from thermoplastic resins (Jorjani et al. 2009; Koob 1991). Paraloid B-72 also has good light stability, but a relatively low T_g (40°C).

Epoxy resins

Companies such as Abatron, Bonstone, AKEMI, Sika and Hilti all provide a wide range of thixotropic adhesives. For lower viscosity products, Epo-Tek, Hxtal NYL-1, Ablebond and Araldite resins can be used.

Polyester resins

AKEMI Marmorkitt 1000 has been used. Polyester resins are now used less and less for stone conservation.

Hybrid systems

Casein and hydraulic lime, acrylic dispersion and cement (Edison Coatings), beeswax and pine resin have been used.

Consolidants

Alkoxysilane consolidants, primarily ethyl silicate-based, are provided by several companies, including Wacker Chemie AG (Germany), Remmers Limited (Germany and UK), COLCOAT Co., Ltd. (Japan), Evonik (Germany and US) and PROSOCO, Inc. (US). These consolidants are generally offered in hydrophobic and non-hydrophobic forms, and Remmers offers elastomeric and calcite-specific varieties. Paraloid B-72 or generic varieties of poly(methyl methacrylate) are the primary organic resins used for consolidation. A specific formulation often referred to as the Bologna Cocktail is a mixture of Paraloid B-72 and a silicone resin (Dri-Film 104; General Electric).

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Abbreviations

AIC	American Institute for Conservation of Historic and Artistic Works
CCI	Canadian Conservation Institute
ICOM	International Council of Museums
ICOM-CC	International Council of Museums – Committee for Conservation
GCI	The Getty Conservation Institute
ICCROM	International Centre for the Study of the Preservation and Restoration of Cultural Property
IIC	International Institute for Conservation of Historic and Artistic Works
IPC	Institute of Paper Conservation
JAIC	Journal of the American Institute for Conservation
JCAC	Journal of the Canadian Association for Conservation
JIIC-CG	Journal of the International Institute for Conservation – Canadian Group
MAC	Master in Art Conservation
SPNHC	The Society for the Preservation of Natural History Collections
SSCR	Scottish Society for Conservation and Restoration
UKIC	United Kingdom Institute for Conservation

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