



CAN UNCLASSIFIED

DRDC | RDDC
technologysciencetechnologie



Evaluation of Liquid Repellent Fabrics

Prepared by:
Dr. Claudia M. Grozea
Calian Technologies
and Dept. of Chemistry and Chemical Engineering
Royal Military College of Canada
PO Box 17000, Station Forces
Kingston, Ontario K7K 7B4

Royal Military College of Canada Contract Report CPT-1803
PSPC Contract Number: W4938-17-31S
Technical Authority: Eva Dickson, Defence Scientist
Contractor's date of publication: March 2018

Defence Research and Development Canada

Contract Report

DRDC-RDDC-2018-C229

November 2018

CAN UNCLASSIFIED

IMPORTANT INFORMATIVE STATEMENTS

This document was reviewed for Controlled Goods by Defence Research and Development Canada using the Schedule to the *Defence Production Act*.

Disclaimer: This document is not published by the Editorial Office of Defence Research and Development Canada, an agency of the Department of National Defence of Canada but is to be catalogued in the Canadian Defence Information System (CANDIS), the national repository for Defence S&T documents. Her Majesty the Queen in Right of Canada (Department of National Defence) makes no representations or warranties, expressed or implied, of any kind whatsoever, and assumes no liability for the accuracy, reliability, completeness, currency or usefulness of any information, product, process or material included in this document. Nothing in this document should be interpreted as an endorsement for the specific use of any tool, technique or process examined in it. Any reliance on, or use of, any information, product, process or material included in this document is at the sole risk of the person so using it or relying on it. Canada does not assume any liability in respect of any damages or losses arising out of or in connection with the use of, or reliance on, any information, product, process or material included in this document.

Evaluation of Liquid Repellent Fabrics

Dr. Claudia M. Grozea

Calian Technologies and Dept. of Chemistry and Chemical Engineering,
Royal Military College of Canada, PO Box 17000, Station Forces,
Kingston, Ontario K7K 7B4 Canada



Deliverable for Defence Research and Development Canada
06CC PROTECT – WBE 1.3

**Royal Military College of Canada Contract Report CPT-1803
March 2018**

- © Her Majesty the Queen in Right of Canada, as represented by the Minister of National Defence, 2018
- © Sa Majesté la Reine (en droit du Canada), telle que représentée par le ministre de la Défense nationale, 2018

Abstract

Fabrics coated with liquid repellent finishes can help to protect personnel from both hazardous toxic substances and everyday contaminants. Three standard test methods (ISO 4920, AATCC 193 and AATCC 118) and three test methods developed by university research laboratories (contact angle, sliding angle and shedding angle tests) were set up and used to measure five textiles coated with liquid repellent finishes, in order to establish some baseline expectations for results using these methods at the Royal Military College. The fabrics showed some variations in liquid repellency towards water, various oils, and the chemical warfare agent simulant methyl salicylate. The tests can be further expanded to include more realistic attributes such as movement and pressure.

Resumé

Les textiles enduits d'une couche résistante aux liquides peuvent aider à protéger le personnel contre les substances toxiques dangereuses et les contaminants quotidiens. Trois méthodes d'essai standard (ISO 4920, AATCC 193 et AATCC 118), et trois méthodes d'essai développées par les laboratoires de recherche universitaires (angle de contact, angle de glissement et angle de chute) ont été mises en place et utilisées pour mesurer cinq textiles enduit d'une couche résistant aux liquides, afin d'établir certaines attentes de base pour les résultats en utilisant ces méthodes au Collège Militaire Royal du Canada. Les textiles présentaient certaines variations de répulsion des liquides vis-à-vis l'eau, diverses huiles et l'agent de guerre chimique simulant le salicylate de méthyle. Les tests peuvent être élargis pour inclure des attributs plus réalistes telles que le mouvement et la pression.

Table of contents

Abstract	iii
Resumé	iii
Table of contents	iv
List of figures	vi
List of tables	vii
Acknowledgments	viii
1 Introduction.....	1
1.1 Test methods.....	1
1.1.1 Standard test methods	1
1.1.2 Research test methods	3
1.2 Aim of experiments	4
2 Experimental procedures.....	6
2.1 Sample treated fabrics	6
2.2 Standard test methods.....	6
2.2.1 ISO 4920	6
2.2.2 AATCC 193	7
2.2.3 AATCC 118	8
2.3 Research test methods	9
2.3.1 Contact angle measurement	9
2.3.2 Sliding angle measurement	10
2.3.3 Shedding angle measurement.....	10
3 Results and discussion	12
3.1 Results summary	12
3.2 Standard test methods.....	16
3.2.1 ISO 4920	16
3.2.2 AATCC 193	16
3.2.3 AATCC 118	17
3.3 Research test methods	17
3.3.1 Contact angle measurement	17
3.3.2 Sliding angle measurement	18
3.3.3 Shedding angle measurement.....	19
4 Conclusions.....	21
References.....	22
Annex A Contact angle measurement procedure.....	24
A.3.1 Measurement Procedure:.....	24
A.3.2 Image Processing:	25
Annex B Sliding angle measurement procedure.....	29
Annex C Shedding angle measurement procedure	31

UNCLASSIFIED // NON-CONTROLLED GOODS

Annex D	Photographs of results	33
D.1.1	ISO 4920	33
D.1.2	AATCC 193	34
D.1.3	AATCC 118	38
D.2.1	Contact angle test	44
List of abbreviations and acronyms		49

List of figures

Figure 1: Molecular structure of a C8 fluorochemical end group. 1

Figure 2: Spray test rating chart from 100 (no wetting) to 0 (fail). 2

Figure 3: Schematic of repellency rating scale where A is a pass, B is a borderline pass, and C and D are fails. 2

Figure 4: Contact angle measurement. 3

Figure 5: Schematic representation of sliding angle measurement. 4

Figure 6: Schematic representation of shedding angle measurement. 4

Figure 7: Spray test measurement set-up. 6

Figure 8: AATCC 193 measurement set-up. 7

Figure 9: AATCC 118 measurement set-up. 8

Figure 10: Contact angle measurement set-up. 9

Figure 11: Sliding angle measurement set-up. 10

Figure 12: Shedding angle measurement set-up. 11

List of tables

Table 1: Liquid repellency terminology.	3
Table 2: AATCC 193 solutions.	7
Table 3: AATCC 118 test liquids.	8
Table 4: Contact angle solutions.	10
Table 5: Summary of results.	13
Table 6: ISO 4920 results.	33
Table 7: AATCC 193 aqueous solution 6 results.	34
Table 8: AATCC 193 aqueous solution 7 results.	35
Table 9: AATCC 193 aqueous solution 8 results.	36
Table 10: Isopropyl alcohol results.	37
Table 11: AATCC 118 oil 3 n-hexadecane results.	38
Table 12: AATCC 118 oil 4 n-tetradecane results.	39
Table 13: AATCC 118 oil 5 n-dodecane results.	40
Table 14: AATCC 118 oil 6 n-decane results.	41
Table 15: AATCC 118 oil 7 n-octane results.	41
Table 16: AATCC 118 oil 8 n-heptane results.	42
Table 17: Methyl salicylate results.	43
Table 18: Water contact angle results.	44
Table 19: Diiodomethane contact angle results.	45
Table 20: Cooking oil contact angle results.	46
Table 21: n-Hexadecane contact angle results.	47

Acknowledgments

I would like to thank Dr. Eva F. Gudgin Dickson for her input, guidance, and assistance in preparing this document.

1 Introduction

Textiles modified with liquid repellent coatings can help protect personnel from hazardous liquids which they might encounter in their daily work such as oils, lubricants, cleaning solvents, as well as chemical and biological warfare agents [1][2]. A number of liquid repellent coatings have been developed and are available on the market. The most successful coatings to date are based on C8 (eight carbon atoms) fluorochemicals whose molecular structure can be seen in Figure 1. These coatings have excellent liquid repellency. They can repel a broad range of liquids ranging from high surface tension water to low surface tension oils. However, recent studies have shown that these coatings have a negative impact on the environment, and subsequently, regulations have been put in place to restrict or ban their use in some countries. It will become more difficult to acquire these finishes in the future.

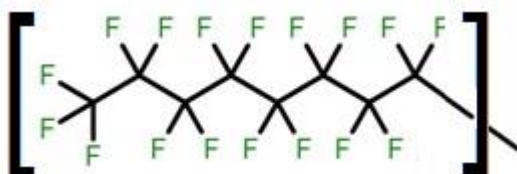


Figure 1: Molecular structure of a C8 fluorochemical end group.

Along with the development of liquid repellent finishes, methods have also been developed to evaluate the liquid protection performance levels of these finishes. Test methods to measure coated materials were developed by both university research laboratories and standard test councils. In particular, standard test methods include ISO (International Organization for Standardization) 4920 – Determination of resistance to surface wetting (spray test) [3], AATCC (American Association of Textile Chemists and Colorists) 193 - Aqueous liquid repellency: water/alcohol solution resistance test [4], and AATCC 118 - Oil repellency: hydrocarbon resistance test [5]. On the other hand, methods used by university research laboratories include the contact angle [6], sliding angle [2], and shedding angle tests [7].

1.1 Test methods

1.1.1 Standard test methods

1.1.1.1 ISO 4920

ISO 4920 is used to determine the resistance of a fabric to surface wetting by water [3]. Equivalent to this method are the AATCC 22 (Water repellency: spray test) [8] and CAN/CGSB (Canada/ Canadian General Standards Board) 4.2 No. 26.2 (Textile test methods: textile fabrics - determination of resistance of surface wetting (spray test)) [9] methods.¹ In the spray test method, the taut surface of a fabric is sprayed with water and a wetted pattern is produced. The pattern on the fabric is compared to a photographic standard chart and a rating is assigned to the coated fabric. The ratings are ISO 0 (fail) to 5 (no wetting from water) as shown in Figure 2. The

¹ Note the Canadian test is currently withdrawn but will likely be updated and reissued.

AATCC 22 scale is 0 (fail), 50, 70, 80, 90, and 100 (no wetting from water). Intermediate ISO ratings can be used in increments of 0.5.

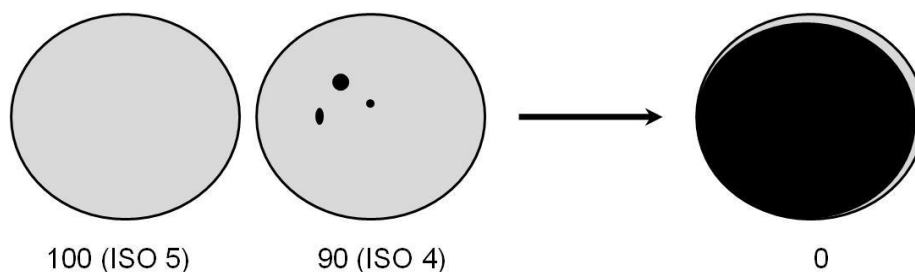


Figure 2: Spray test rating chart from 100 (no wetting) to 0 (fail).

1.1.1.2 AATCC 193

AATCC 193 is used to determine the resistance of fabrics to wetting by aqueous liquids with different surface tensions [4]. Droplets of water/isopropyl alcohol solutions with decreasing surface tensions ranging from 59.0 mN/m to 24.0 mN/m are placed on the coated fabric and visually observed for wetting for 10 seconds. The number assigned to the solution that does not wet the sample is taken as the repellency grade. The rating scale can be seen in Figure 3. There are two types of passing levels for this test, a level A pass where the droplet has a clear well rounded appearance with a high contact angle, and a level B borderline pass where the droplet is not as rounded and some partial darkening is observed. There are also two types of fail levels, a level C fail where wicking is apparent and/or complete wetting is observed, and a level D fail where there is complete liquid wetting.

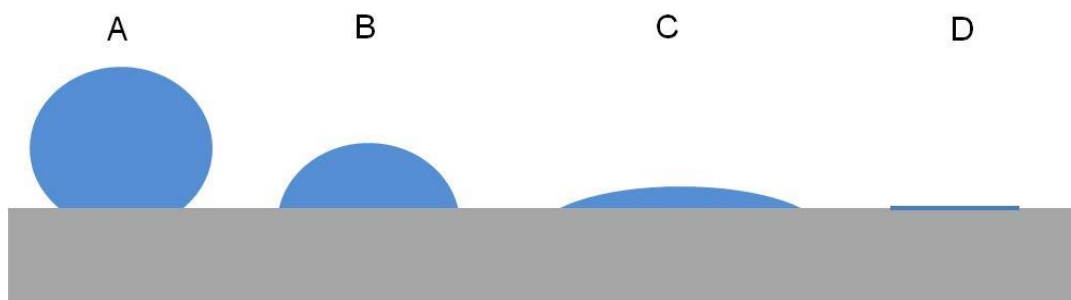


Figure 3: Schematic of repellency rating scale where A is a pass, B is a borderline pass, and C and D are fails.

1.1.1.3 AATCC 118

AATCC 118 is used to determine the resistance of fabrics to wetting by liquid hydrocarbons with different surface tensions [5]. The evaluation scale for the oils ranges from 0 (fail) using Kaydol, a mineral oil with a surface tension of 31.5 mN/m at 25 °C, to 8 (highest oil repellency) using n-heptane, which has a surface tension of 19.8 mN/m at 25 °C². Liquid droplets are placed on the

² Note: the AATCC 118 – 1997 edition contains an error: it lists the surface tension of n-heptane as 14.8 mN/m.

fabric and are visually observed for wetting for 30 seconds. The number assigned to the oil that does not wet the fabric is taken as the repellency oil grade. The rating scale is the same as the one for AATCC 193 and can be seen in Figure 3 above.

1.1.2 Research test methods

1.1.2.1 Contact angle measurement

The contact angle measures the interaction between a liquid droplet and a material's surface [6]. It is the angle between the solid-liquid and liquid-gas interfaces of a liquid droplet placed on a surface measured through the liquid droplet as shown in Figure 4. The higher the angle value, the higher the materials' ability to repel the liquid. The terminology associated with this measurement can be seen in Table 1. Ideally, a repellent surface should be able to repel both water and oil liquid droplets.

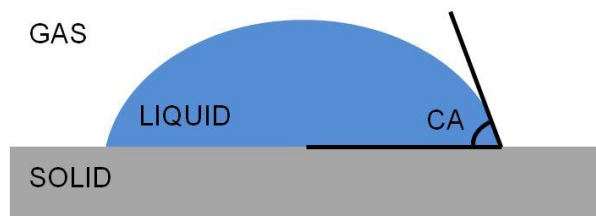


Figure 4: Contact angle measurement.

Table 1: Liquid repellency terminology.

Contact Angle (CA) range	Test Liquid Type		
	Water	Oil	Water and Oil
CA = 0°	Complete wetting		
CA < 90°	Hydrophilic	Oleophilic	Amphiphilic or Omniphilic
90° ≤ CA ≤ 150°	Hydrophobic	Oleophobic	Amphiphobic or Omniphobic
CA > 150°	Superhydrophobic	Superoleophobic	Superamphiphobic or Superomniphobic
CA = 180°	Complete non-wetting		

1.1.2.2 Sliding angle measurement

The sliding angle can help to differentiate the degree of wettability between surfaces that have the same contact angle values [2]. The sliding angle is the minimum angle of inclination at which a

liquid droplet starts to roll off the surface. A liquid droplet is placed on the surface of a material and the material is tilted from the horizontal position until the droplet starts to roll off the surface. This tilting angle is then taken as the droplet's sliding angle. A schematic of this measurement can be seen in Figure 5. Low sliding angles are associated with highly repellent surfaces.

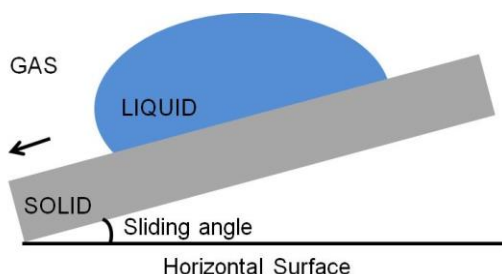


Figure 5: Schematic representation of sliding angle measurement.

1.1.2.3 Shedding angle measurement

The shedding angle can also help to differentiate the degree of wettability between surfaces that have the same contact angle values [7]. It determines the ability of a surface to repel liquid droplets upon impact. The shedding angle is the minimum angle at which a droplet dispensed from a distance onto a tilted surface rolls off the surface. A schematic of this measurement can be seen in Figure 6. The material is mounted at a tilt angle of 85° and the angle is subsequently reduced until a dispensed droplet no longer rolls off the surface. The shedding angle is then taken as the last angle the droplet rolled off the surface. The lower the value of the tilt angle of the surface, the higher is the ability of the coated surface to repel liquid droplets upon impact.

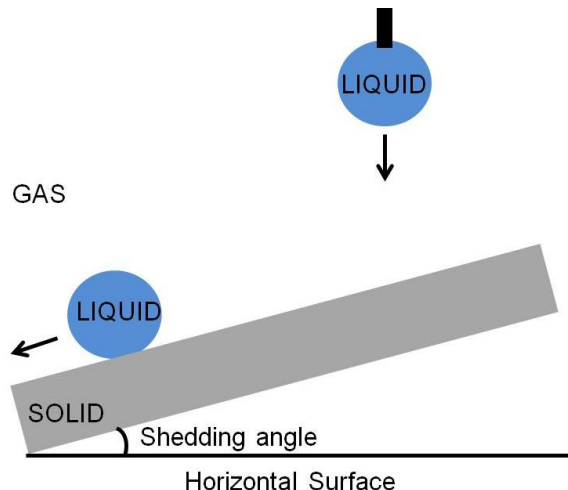


Figure 6: Schematic representation of shedding angle measurement.

1.2 Aim of experiments

The aim of these experiments is to set-up a number of liquid repellency evaluation methods and test a number of textiles in order to provide a baseline understanding of how these tests perform with a variety of repellent fabrics. In particular, this report covers the set-up of the three standard test methods and the three university laboratories methods discussed above. These methods were

then used to test a number of textiles available on hand. The exact details of the liquid repellent finishes used to treat these fabrics are not known. Nevertheless, many were likely treated with a type of C8 fluorochemical.

2 Experimental procedures

Three standard test methods and three university laboratories methods were set-up as described below.

2.1 Sample treated fabrics

The methods were used to test five coated fabrics, here labeled H, R, S, N, and F (where H, S and N were obtained from three different chemical-biological protective garments, R was a rain-protective fabric, and F was firefighter turnout gear outer shell material). These fabrics were chosen simply to provide various treated surfaces that could be investigated in order to exercise the methods.

2.2 Standard test methods

2.2.1 ISO 4920

The ISO 4920 test procedure was followed as described in the standard test document [3] with some modifications. The spray test measurement was set-up as shown in Figure 7. Briefly, 250 mL of water was poured into a funnel suspended above the taut surface of a fabric followed by tapping the sample on a solid object. The pattern produced by the water on the fabric was compared to a photographic standard chart and a rating was assigned to the fabric. Two samples were measured as opposed to three specimens. The funnel was smaller than in the specifications, 130 mm as opposed to 150 mm. However, the flow rate was similar to the specifications.



Figure 7: Spray test measurement set-up.

2.2.2 AATCC 193

The AATCC 193 test procedure was followed as described in the standard test document [4] with some modifications. The test measurement was set-up as shown in Figure 8. Briefly, three 50.0 μL droplets of water/isopropyl alcohol solutions were placed on the coated fabric and visually observed for wetting for 10 seconds. The number assigned to the solution that did not wet the sample was taken as the repellency grade. The sample size was smaller than the specifications since individual samples were used to test each liquid rather than place all the test liquids on the same large sample. Laboratory paper and a glass slide were used as opposed to textile blotting paper underneath the sample. The water/isopropyl alcohol solutions were prepared as describe in the text and a summary can be seen in Table 2. Additionally, isopropyl alcohol was tested which has a surface tension of 23.3 mN/m [10].

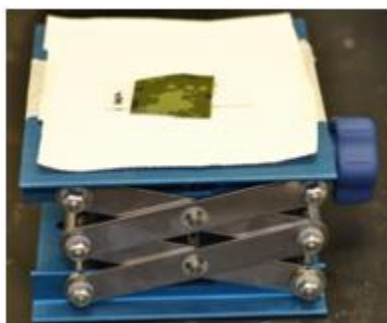


Figure 8: AATCC 193 measurement set-up.

Table 2: AATCC 193 solutions.

Repellency Grade Number	Test Liquid (vol/vol)		Surface Tension (mN/m)
	Water	Isopropyl Alcohol	
0 - Fail	98	2	59.0
1	98	2	59.0
2	95	5	50.0
3	90	10	42.0
4	80	20	33.0
5	70	30	27.5
6	60	40	25.4

UNCLASSIFIED // NON-CONTROLLED GOODS

Repellency Grade Number	Test Liquid (vol/vol)		Surface Tension (mN/m)
	Water	Isopropyl Alcohol	
7	50	50	24.5
8	40	60	24.0

2.2.3 AATCC 118

The AATCC 118 test procedure was followed as described in the standard test document [5] with some modifications. The test measurement was set up as shown in Figure 9. Briefly, five 50.0 μL droplets of oils were placed on the coated fabric and visually observed for wetting for 30 seconds. The number assigned to the oil that did not wet the sample was taken as the repellency grade (Table 3). The sample size was smaller than the specifications since individual samples were used to test each liquid rather than place all the test liquids on the same large sample. Laboratory paper was used as opposed to textile blotting paper underneath the sample. The properties of the oils are summarized in Table 3. Additionally, methyl salicylate was tested which has a surface tension of 39.2 mN/m [11].

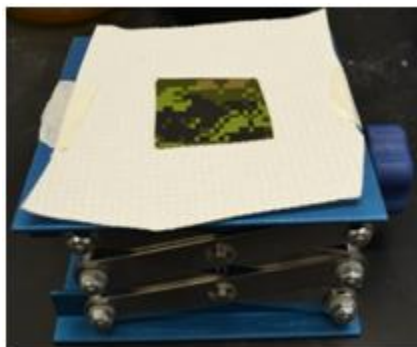


Figure 9: AATCC 118 measurement set-up.

Table 3: AATCC 118 test liquids.

Repellency Grade Number	Test Liquid	Surface Tension (mN/m)
0 - Fail	Kaydol (fails)	31.5
1	Kaydol	31.5
2	65:35 Kaydol:n-Hexadecane	29.6
3	n-Hexadecane	27.3

Repellency Grade Number	Test Liquid	Surface Tension (mN/m)
4	n-Tetradecane	26.4
5	n-Dodecane	24.7
6	n-Decane	23.5
7	n-Octane	21.4
8	n-Heptane	19.8 (Not 14.8)

2.3 Research test methods

2.3.1 Contact angle measurement

The contact angle was measured using the sessile drop method [6]. The test was set-up as shown in Figure 10. A 5.0 μL droplet of the test liquid was placed on the coated fabric and an image of the drop's profile was acquired using an optical system, in this case, a camera equipped with a macro lens. This process was repeated three times. The images were then processed using ImageJ to obtain the contact angle of the surface. The test liquids used were water, diiodomethane cooking oil, and n-hexadecane. Their properties can be seen in Table 4 [10],[12],[13].

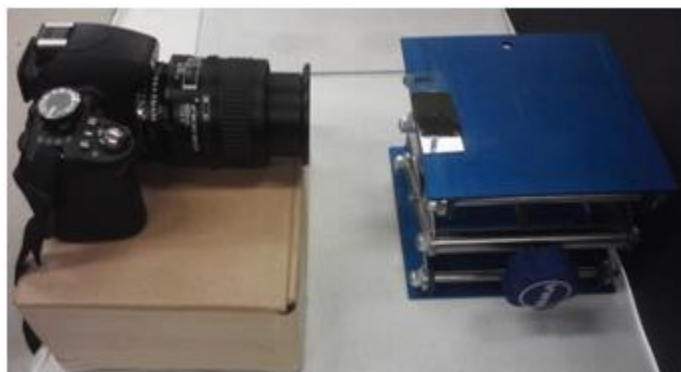


Figure 10: Contact angle measurement set-up.

Table 4: Contact angle solutions.

Test Liquid	Surface Tension (mN/m)
Water	72.8
Diiodomethane	50.9
Cooking oil	33.8
n-Hexadecane	27.3

2.3.2 Sliding angle measurement

The sliding angle was measured as shown in Figure 11. Three 10.0 or 5.0 μL droplets of the test liquid were placed on the coated fabric, which was on top of a Plexiglas plate positioned at an inclination angle of 0° . One end of the plate was raised using the lab jack until the droplets rolled off the fabric. The inclination angle of the plate where the droplets rolled off was measured with a protractor. The same test liquids as for the contact angle measurement were used, water, diiodomethane cooking oil, and n-hexadecane.

*Figure 11: Sliding angle measurement set-up.*

2.3.3 Shedding angle measurement

The shedding angle was measured with a similar set-up as the sliding angle set-up shown in Figure 12 [7]. One end of a Plexiglas plate with a coated fabric on top of it was raised using the lab jack until the coated fabric was at an inclination angle of 85° . A protractor was used to measure this angle. Three 10.0 μL droplets of the test liquid were released onto the coated fabric from a 1.0 cm height. If the droplets rolled off the fabric for at least 2.0 cm, the inclination angle of the plate was lowered. The experiment was repeated until the droplets no longer rolled off the fabric. The last angle at which all the droplets rolled off the fabric was taken as the shedding angle. The same test liquids as for the contact angle measurement were used, water, diiodomethane cooking oil, and n-hexadecane.



Figure 12: Shedding angle measurement set-up.

3 Results and discussion

3.1 Results summary

Three standard test methods, ISO 4920, AATCC 193 and AATCC 118, and three test methods developed by university research laboratories, contact angle, sliding angle and shedding angle tests, were set up and used to measure five textiles coated with liquid repellent finishes with results as outlined in Table 5, with photographs of the results found in Annex D. In general, for most of the tests performed, fabric R was the most hydrophobic and oleophobic and S the least. This is most likely due to a difference in the amount of repellent coating applied in each case, rather than a significant difference in its chemistry. However ranking of the five fabrics was certainly not the same in every case.

Some amount of the inconsistency in the ranking may have been due to errors in the methods. Note that this was considered a preliminary investigation to understand how the methods performed at a high level, and no attempt to rigorously estimate error or reproducibility of results has been made, which would have required a much more extensive set of experiments using more replicate samples drawn from different sources of original materials. Undoubtedly, aside from any irreproducibility in the treated fabrics themselves, the replicability of the result will be dependent on the flatness of the fabric surface and with which the sample can be mounted, as well as the quality of the mounting systems and of the tools used to record the results (photographic, angular).

Other factors that could change the ranking by test might have to do with more specific parameters such as weave and roughness of the fabric surface. Overall it is likely that a suite of tests would be needed in order to truly understand realistic repellency behaviour, including adding even more realistic test conditions.

A more detailed discussion of each of the individual test methods follows.

UNCLASSIFIED // NON-CONTROLLED GOODS

Table 5: Summary of results.

Sample → Test ↓	R	H	S	N	F	Comment
ISO 4920 water spray (AATCC 22)	ISO 5 (100)	ISO 5 (100)	ISO 4 (90)	ISO 5 (100)	ISO 4.5 (95)	S least water repellent
AATCC 193 water/alcohol	8A	8A	8A	8A	8A	No differentiation in water/alcohol repellency
Isopropyl Alcohol	A	C	D	C	C	Only R repellent to isopropyl alcohol and S least repellent
AATCC 118 oil	7B	6B	4A	6B	5A	R most repellent and S least repellent to oils
Methyl Salicylate	A	A	A	A	A	All repellent to methyl salicylate with no differentiation
Water Contact Angle (°)	154 (super- hydrophobic)	150 (hydro- phobic)	144 (hydro- phobic)	152 (super- hydrophobic)	151 (super- hydrophobic)	R most repellent and S least repellent to water

UNCLASSIFIED // NON-CONTROLLED GOODS

Sample → Test ↓	R	H	S	N	F	Comment
Diiodomethane Contact Angle (°)	119	139	126	132	131	R most repellent H least repellent to diiodomethane
Cooking Oil Contact Angle (°)	141	139	132	141	140	S least repellent to cooking oil
n-Hexadecane Contact Angle (°)	123	123	121	117	125	N least repellent to n-hexadecane
Water Sliding Angle (°, 10.0 / 5.0 µL)	46 / 90	45 / 90	54 / 90	38 / 67	40 / 70	N most repellent S least repellent to water
Diiodomethane Sliding Angle (°, 10.0 / 5.0 µL)	No / No	No / No	No / No	No / No	No / No	None repellent to diiodomethane
Cooking Oil Sliding Angle (°, 10.0 / 5.0 µL)	No / No	No / No	No / No	No / No	No / No	None repellent to cooking oil
n-Hexadecane Sliding Angle (°, 10.0 / 5.0 µL)	No / No	No / No	No / No	No / No	No / No	None repellent to n-hexadecane

UNCLASSIFIED // NON-CONTROLLED GOODS

Sample → Test ↓	R	H	S	N	F	Comment
Water Shedding Angle (°, 10.0 µL)	23	16	32	21	14	F most repellent S least repellent to water
Cooking Oil Shedding Angle (°, 10.0 µL)	No	No	No	No	No	None repellent to cooking oil
n-Hexadecane Shedding Angle (°, 10.0 µL)	No	No	No	No	No	None repellent to n-hexadecane

3.2 Standard test methods

3.2.1 ISO 4920

ISO 4920 is a spray test used to determine the resistance of a fabric to surface wetting by water [3]. It requires a specialized nozzle and funnel; however, it is straightforward to set-up and use. It was not possible to acquire the specified funnel when this test was conducted due to its unusual large dimension, 150 mm; thus, a smaller funnel had to be used, 130 mm. The size of the funnel should not have affected the results since the specified water flow duration and height from the sample was maintained. The challenge of this test is the size and number of samples required for this experiment, at least three samples of 180 mm square. It requires a lot of material which might not be easily available. Only two specimens were tested from each coated fabric type as opposed to three.

All the coatings showed high water repellency with no or minimal amount of water left on the surface as can be seen in Table 5 and Annex D.1.1 - Table 6. This was expected since the fabrics are supposed to be coated with a liquid repellent finish. It is typically not as difficult to achieve water repellency due to the high surface tension of water, 72.8 mN/m [10]. This test is done at a 45° inclination angle; future work could focus on modifying this test to include testing the fabrics at various inclination angles. This test can also be made more realistic by performing it on a stationary, as well as a moving, subsystem such as an arm or a leg or even a full body.

3.2.2 AATCC 193

AATCC 193 measures the resistance of fabrics to wetting by aqueous liquids with surface tensions ranging from 59.0 mN/m to 24.0 mN/m [4]. These solutions are prepared by mixing water with isopropyl alcohol. This test does not require any specialized equipment. Once again, the difficulty arises in acquiring enough material for testing, two specimens of at least 20 x 20 cm. Small individual samples were tested with each liquid rather than placing all liquids on the same large sample. This made observations much easier.

All the coatings showed high repellency towards aqueous solutions and had the maximum rating of 8 out of 8 and an A grade as can be seen in Table 5 and Annex D.1.2 - Tables 7 to 9, which include photographs of results for solutions 6 to 8. If aqueous droplets come into contact with these fabrics while in use, the droplets will not wet and penetrate into the fabric unless influenced by some external factor such as pressure. More information can be obtained from this test if the behaviour of the droplets is observed for a longer period of time and if the samples are tilted to see if the droplets roll off. Pressure can also be applied to simulate more realistic conditions such as a person brushing against a surface or kneeling.

Additionally, the fabrics were tested using isopropyl alcohol as can be seen in Table 5 and Annex D.1.2 - Table 10. In this case, only R resisted surface wetting. This could be due to a tighter fabric weave, a more repellent coating, or a higher amount of coating which typically tends to increase repellency properties.

3.2.3 AATCC 118

AATCC 118 measures the resistance of fabrics to wetting by liquid oils with surface tensions ranging from 31.5 mN/m to 19.8 mN/m [5]. This test does not require any specialized equipment, except it requires very specific chemicals. Once again, the difficulty arises in acquiring enough material for testing, two specimens of at least 20 x 20 cm. Small individual samples were tested with each liquid rather than placing all liquids on the same large sample. This made observations much easier.

Results from this test can be seen in Table 5 and Annex D.1.3 - Tables 11 to 16, which include illustrative photographs of results for solutions 3 to 8 with only three liquid droplets. Evaluation tests were carried out as specified with five liquid droplets. In this test, differences can be seen between the coatings. It is challenging to develop coatings that can repel very low surface tension liquids. In addition to the type of coating used on the fabric, differences in weave openness, bundle and fibre dimension, or coating amount, can have a high impact on the level of liquid repellency in the low surface tension region. Fabric R had a slightly better repellency performance than H. If droplets of oil 7 were left on H, some darkening of the fabric was observed; thus, an oil grade of 6 was assigned to this fabric. When oil 8 was used, H had a fail grade of D, while R had a fail grade of C. High ratings are expected of these coatings since both of these fabrics showed good repellency in other tests. The difference between them could be due to the openness of the fabric's weave, as H appears to have a more open weave than the others. Nonetheless, the coating type and amount could have played a role too. Both N and F had some repellency towards oils as expected from previous tests. Fabric S showed the worst oil liquid performance; however, this is not unusual since it also had a lower liquid repellency in tests with higher surface tension liquids.

If liquid droplets come into contact with these fabrics while in usage, the droplets will not wet and penetrate into the fabric if the surface tension of the liquids is the same or higher than the surface tension of the oil repellency grade or unless influenced by some external factor such as pressure. Once again, more information can be obtained from this test if the behaviour of the droplets is observed for a longer period of time and if the samples are tilted to see if the droplets roll off. Pressure can also be applied to simulate more realistic conditions such as a person brushing against a surface.

Moreover, the fabrics were tested using the chemical warfare agent simulant methyl salicylate as can be seen in Table 5 and Annex D.1.3 - Table 17. Methyl salicylate is an inert physical simulant for the chemical warfare agents sulfur mustard and soman [14]. All the samples performed well and resisted wetting by methyl salicylate. The surface tension of methyl salicylate is 39.2 mN/m [11], which is higher than the surface tension of the oils the fabrics can repel as tested per AATCC 118. Thus, it is not surprising all the fabrics showed good repellency.

3.3 Research test methods

3.3.1 Contact angle measurement

The contact angle is used to determine the degree of liquid wetting of a surface by measuring the angle between a liquid droplet and the surface [6]. Typical test liquids include water, diiodomethane cooking oil, and n-hexadecane, which have surface tension ranging from 72.8

mN/m to 27.3 mN/m [10]. Photographs of these probe liquids sitting on the various fabrics were taken using a camera equipped with a macro lens, followed by post-processing to obtain a contact angle value. This measurement is challenging to perform with a handheld camera as opposed to a contact angle measuring instrument due to the difficulty in acquiring a clear image of the water-surface interface. Camera focus, lighting, or contrast can introduce errors into the measurement. Additionally, textiles are challenging to measure since they are non-reflective, flexible and have macroscopic roughness. This affects the determination of the surface's baseline, which is the contact line between the liquid droplet and the surface. A way to try to mitigate this error is to flatten the textile using a weight before measurement. A detailed procedure is given in Annex A.

Results from this test can be seen in Table 5 and Annex D.2.1 - Tables 18 to 21, which include photographs of these results. All the samples tested showed liquid repellency in the omniphobic regime. They can resist wetting by both water and oils. The value of the contact angle typically decreases as the surface tension of the probe liquid decreases. This was observed for these samples except when diiodomethane was used. The diiodomethane contact angle is lower than expected. This could be due to the challenge in obtaining a clear photograph of the liquid-surface interface. Diiodomethane has a high density, 3.325 g/mL [15], resulting in a more oval droplet and a smaller gap between the droplet's edge and the surface. When this is combined with a rough surface, it is difficult to clearly see the contact point. Some differences could be seen in the samples such as lower water and cooking oil contact angle for S, but in general the samples were similar. This was expected since previous tests such as ISO 4920, AATCC 193, and AATCC 118, showed a similar behaviour in liquid repellency for the samples in the 72.8 mN/m to 27.3 mN/m surface tension ranges as was used in this test. The difference in values could be attributed to both errors introduced from the photographs as well as differences in fabric types and coatings. If liquid droplets come into contact with these fabrics while in usage, the droplets will not wet and penetrate into the fabric unless influenced by some external factor such as pressure. More information can be obtained from this test if it is combined with other tests such as the sliding and shedding angle tests in which samples are tilted to see if droplets roll off the fabric. Pressure can also be applied to simulate more realistic conditions such as a person brushing against a surface.

3.3.2 Sliding angle measurement

The sliding angle was measured by placing a liquid droplet on the surface of a material and tilting the material from the horizontal position until the droplet starts to roll off its surface [2]. The minimum angle of inclination at which the droplet starts to roll off the surface is taken as the sliding angle. This test can help to differentiate between different surfaces that have the same contact angle values. The most difficult part of this test is to accurately measure the sample's angle of inclination. Previous measuring attempts were to measure the length and height of the sample, followed by calculating the angle from these values. This approach was discontinued and a protractor was used instead to directly measure the angle of inclination. This simplified the measurement and resulted in greater accuracy. In the case of textiles, another challenge is protruding fiber ends and pores from the weave structure, which can sometimes cause the droplets to stick to the fabric and prevents them from rolling off the surface. A detailed procedure is given in Annex B.

Results from this test using 10.0 and 5.0 μL liquid droplets can be seen in Table 5. High contact angle values suggest the possibility of high liquid repellency with low sliding angles. However, the water sliding angle values are not very low, which indicates the existence of a liquid repellent coating, but not the presence of a highly repellent coating. There is some sticking of the droplet to

the fabric because the entire droplet is probably not suspended above the fabric on top of trapped air pockets, but instead part of the droplet is sitting inside small protrusions present on the fabric's surface. The contact angle test should be used in conjunction with other tests such as the sliding angle test to get a better understanding on the behaviour and level of a coating's liquid repellency. As similar to previous tests, samples showed similar sliding behaviour with S having a slightly worst performance than the other fabrics. The difference in values can be explained again by the difference in fabric structure, in the coating, as well as negative effects caused by individual protruding fibre ends. The sliding angle values were also higher for larger droplets due to a stronger gravitational force. If a liquid droplet is in contact with the fabrics while in usage, the droplet could come off the fabric if it were tilted to or above the sliding angle such as by bending an arm or leg. This test can be made more realistic by performing it on a stationary, as well as a moving, subsystem such as an arm or a leg or even a full body.

On the other hand, with the other probe liquids, diiodomethane, cooking oil, and n-hexadecane, the liquid droplets did not slide off the fabrics; however, the droplets sometimes moved down the fabric leaving traces with little droplets attached to the fabric. These liquids have a lower surface tension than water and it is more difficult for them to slide off the fabric. If a liquid droplet is in contact with the fabrics while in usage, the droplet would not roll off the fabric if it were tilted.

3.3.3 Shedding angle measurement

Finally, the shedding angle was measured by tilting the fabric and dispensing a liquid droplet from 1.0 cm above the sample [7]. The shedding angle is the minimum inclination angle at which the droplet rolls off the sample's surface. This angle determines the ability of the sample to repel liquid droplets upon impact; thus, it also helps to differentiate the degree of wettability between surfaces that have the same contact angle values. To ensure a more accurate measurement of the inclination angle, a protractor was used in this case too. It was not possible to release 5.0 μL liquid droplets using either the microsyringe or the micropipette; hence, only 10 μL droplets were used in this experiment. In this case too, the structure of the fabric such as the weave will influence the behavior of the droplet and can cause the droplet to stick to the surface. A detailed procedure is given in Annex C.

Results from this test using 10.0 μL liquid droplets can be seen in Table 5. The contact angle measurement suggests the possibility of very low shedding angles; however, if the contact angle test is combined with the sliding angle test, only a low value for the shedding angles is predicted. The sliding angle shows the existence of a partial droplet pinning effect, which increases the angle at which a droplet can roll off the surface. The water shedding angle values are lower than the sliding angle ones due to the kinetic energy of the droplets. As similar to previous tests, samples showed similar behaviour with S having a slightly worst performance than the other fabrics. The difference in values can be explained by the difference in fabric structure, in the coating, and in the number of individual protruding fibre ends. If a liquid droplet falls onto the fabrics while in usage, the droplet could roll off the fabric if it were tilted to or above the shedding angle. This test can be made more realistic by performing it on a stationary, as well as a moving, subsystem such as an arm or a leg or even a full body.

On the other hand, diiodomethane could not be used for this test due to its high density. Diiodomethane droplets could not be released as a single droplet, but instead multiple little droplets were dispensed by the microsyringe. When cooking oil and n-hexadecane were used as the probe liquids, the liquid droplets did not roll off the fabrics. These liquids have a lower

surface tension than water and it is more difficult for them to roll off the fabric. If a liquid droplet is in contact with the fabrics while in usage, the droplet would not roll off and would be stuck to the fabric, which could enhance the likelihood of penetration or permeation.

4 Conclusions

In this study six liquid repellency evaluation methods were set-up and tested on five different fabrics coated with liquid repellent finishes. In particular, three standard test methods were set up, ISO 4920, AATCC 193, and AATCC 118, and three university laboratory research methods, contact angle, sliding angle, and shedding angle. Subsequently, the five sample fabrics were tested with these methods. The chemical composition of the liquid repellent coatings and the level of liquid repellency are not known.

All the tested fabrics showed liquid repellency towards both water and oils indicating a fluorochemical type liquid repellent finish. To date, only fluorochemicals can achieve this level of performance, and given the age of the textiles, it is likely that some or all of these fabrics were treated with a type of C8 fluorochemical. R and H showed the broadest repellency performance; however, R was slightly better possibly due to the weave type. N and F showed similar properties, while S performed the worst in most of the tests. Moreover, they all had good repellency towards methyl salicylate, a chemical warfare agent simulant. The differences in the repellency performance could be explained by differences in fabric properties such as weave openness, bundle and fibre dimensions, as well as the amount of coating on the fabric. The type of coating could also have played a role, but it is not certain if the fabrics are coated with the same / similar finish or a different finish.

Overall, there were no major problems encountered with setting up and using the liquid repellency evaluation test methods. Some minor modifications could be made to improve the methods such as flattening the samples for improved accuracy, a larger funnel for the ISO 4920 method, or some lamps for better light for the contact angle method.

More importantly, as these standardized methods do not necessarily do a clear job of differentiating amongst the fabrics and their treatments, with the ranking of materials sometimes varying by method, the methods should be expanded to better reflect real world situations. For example, testing could be performed on both stationary and moving subsystems such as an arm or a leg or even a full body as opposed to measuring only fabric swatches. Pressure could also be applied to the liquid droplets to better simulate more realistic conditions such as a person brushing against a surface, or bending an arm, or kneeling.

The tests can also be expanded upon by preconditioning the fabrics to realistic conditions. Fabrics are typically worn multiple times and will likely be washed at some point during their lifetime. Fabrics should be laundered by using methods such as the CAN/CGSB 4.2 No. 58 (Textile test methods: dimensional change in domestic laundering of textiles, which is currently withdrawn) [16], and then have their repellency properties retested. Additionally, during everyday usage fabrics would undergo abrasion such as rubbing against each other or against an outside surface; thus, they should also be tested after abrasion preconditioning. Other preconditioning could include the effect of encountering substances such as food, cleaners, decontamination, mud, or sweat, as well as temperature, humidity, or wind effects.

References

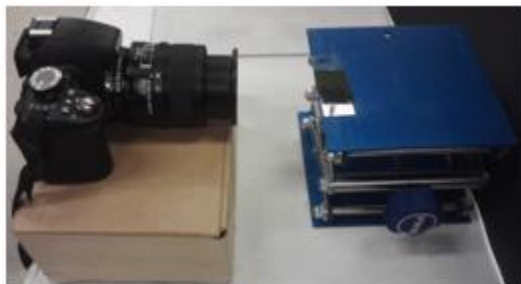
- [1] Gudgin Dickson, E.F. (2013). Personal protective equipment for chemical, biological, and radiological hazards. John Wiley & Sons: Hoboken, NJ.
- [2] Williams, J. (Ed., 2018). Waterproof and water repellent textiles and clothing. Woodhead Publishing: Cambridge, MA.
- [3] International Organization for Standardization (2012). “ISO 4920 - Textile fabrics - Determination of resistance to surface wetting (spray test)”.
- [4] American Association of Textile Chemists and Colorists (2007). “AATCC 193 - Aqueous liquid repellency: water/alcohol solution resistance test”.
- [5] American Association of Textile Chemists and Colorists (1997). “AATCC 118 - Oil repellency: hydrocarbon resistance test”.
- [6] Dataphysics (2017). “Sessile drop method - optical determination of the contact angle”. <http://www.dataphysics.de/2/start/understanding-interfaces/drop-shape-analysis/sessile-drop-method/>. (Accessed December 2017).
- [7] Zimmerman, J.; Seeger, S. (2009). “Water shedding angle: a new technique to evaluate the water repellent properties of superhydrophobic surfaces”. *Text. Res. J.*, 79 (17), 1565–1570.
- [8] American Association of Textile Chemists and Colorists (2014). “AATCC 22 - Water repellency: spray test”.
- [9] Canadian General Standards Board (1994). “CAN/CGSB - 4.2 No. 26.2 - Textile test methods textile fabrics - determination of resistance of surface wetting (spray test)”.
- [10] Jasper, J. J. (1972). “The surface tension of pure liquid compounds”. *J. Phys. Chem. Ref. Data*, 1, 841–1009.
- [11] Wohlfarth, C. (2016). “Surface tension of methyl salicylate”. In: Lechner M.D. (eds) *Surface Tension of Pure Liquids and Binary Liquid Mixtures*, 28. Springer: Berlin, Heidelberg.
- [12] Korosi, G.; Kovats, E. S. (1981). “Density and surface tension of 83 organic liquids”. *J. Phys. Chem. Ref. Data*, 26, 323–332.
- [13] Esteban, B.; Riba, J.-R.; Baquero, B.; Puig, R.; Rius, A. (2012). “Characterization of the surface tension of vegetable oils to be used as fuel in diesel engines”. *Fuel*, 102, 231–238.
- [14] Salter, W. B.; Owens, J. R.; Wander, J. D. (2011). “Methyl salicylate: a reactive chemical warfare agent surrogate to detect reaction with hypochlorite”. *ACS Appl. Mater. Interf.*, 3, 4262–4267.

- [15] Sigma-Aldrich (2018). “Diiodomethane”.
<https://www.sigmaaldrich.com/catalog/product/sial/158429?lang=en®ion=CA>.
(Accessed March 2018).
- [16] Canadian General Standards Board (2004). “CAN/CGSB - 4.2 No. 58 - Textile test methods - dimensional change in domestic laundering of textiles”.

Contact angle measurement procedure

The contact angle measures the interaction between a liquid droplet and a material's surface.

A.1 Equipment Set-Up:



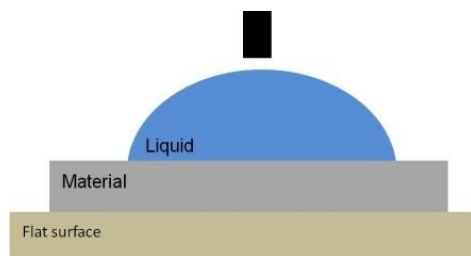
A.2 Chemicals:

- Water
- Diiodomethane
- Cooking oil
- n-Hexadecane

A.3 Procedure:

A.3.1 Measurement Procedure:

1. Put on gloves to avoid contaminating the textile swatches with oils/liquid from hands.
2. Cut out a textile swatch (minimum size 2.5 x 2.5 cm) and attach it to a glass slide using double-sided adhesive tape. If necessary, place tape around the edges to make the swatch as flat as possible or place a weight on the swatch to help flatten it.
3. Place the slide on top of a flat surface.
4. Fill a syringe/pipette with the test liquid chemical such as 5.0 or 10.0 μL .
5. Dispense a 5.0 μL droplet of the test liquid and place it on the textile swatch. 10.0 μL could also be used.



6. Use a camera equipped with a macro lens in the “Macro” setting to take a photograph of the droplet’s profile. The lens should be set to “M” (Manual) and to “Full”. The lens should be fully extended to get as close as possible to the droplet. Focusing is done by moving the whole camera back and forth until the image is clear. It should look similar to this:

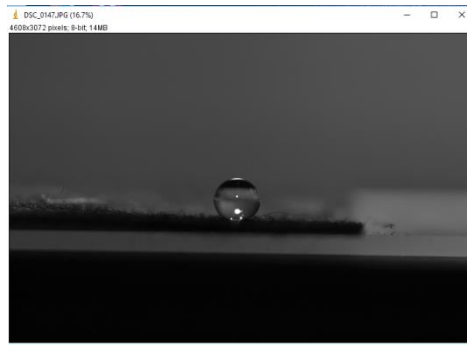


7. Repeat until three (or five) different positions on the swatch have been measured in total.

A.3.2 Image Processing:

1. Download and Install the “ImageJ” software from <https://imagej.nih.gov/ij/download.html>
2. Download and Install the “DropSnake and LB_ADSA (contact angle measurement)” plugin from <http://bigwww.epfl.ch/demo/dropanalysis/>
3. Open ImageJ.
4. Select File – Open. Select your picture by clicking on it. Select Open.

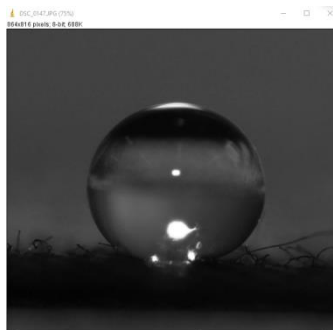
5. Select Image – Type – 8-bit.



6. Select the rectangular selection button (1st button). Draw a box around the droplet.

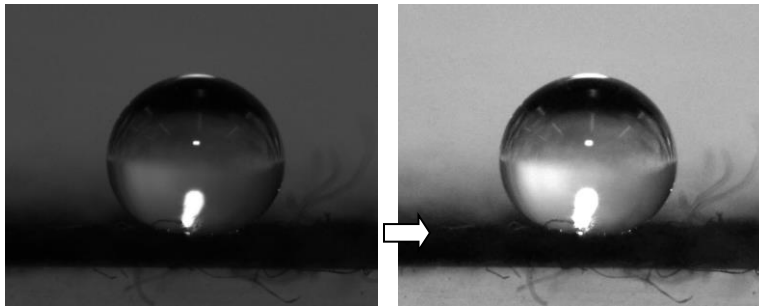


7. Select Go to Image – Crop.

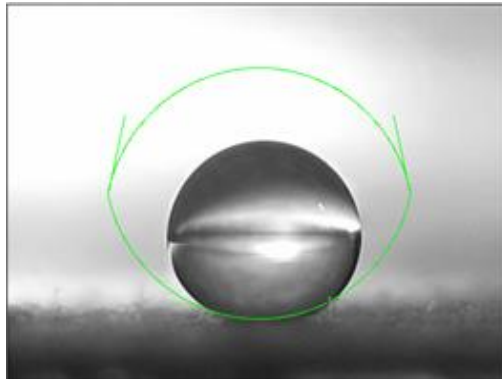


8. Select File – Save as – Jpeg. Give it a new name. Select Save.

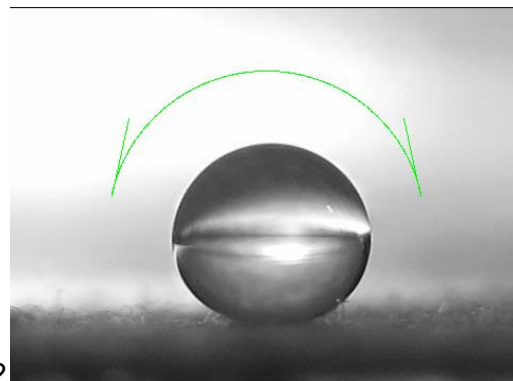
9. If the image is too dark. Select Image – Adjust – Brightness/Contrast. Move the Maximum level. Select Apply.



10. Select Plugins – Drop analysis – LB_ADSA.

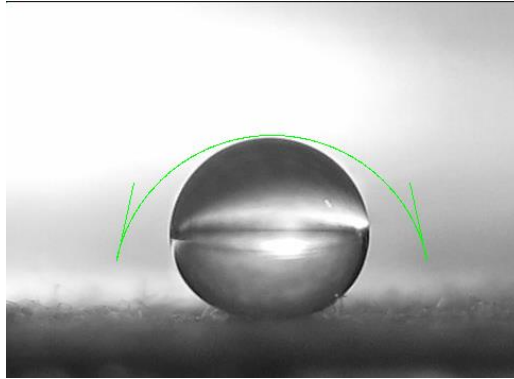


11. Adjust d from 265 to 0 (removes the reflection)

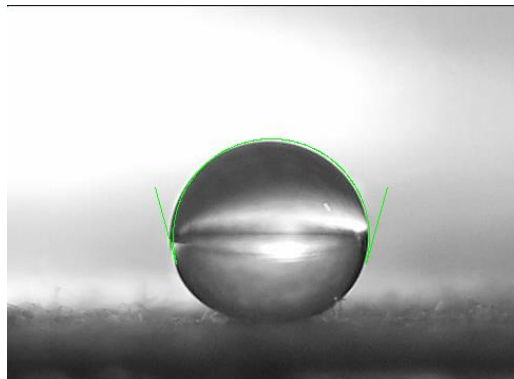


2

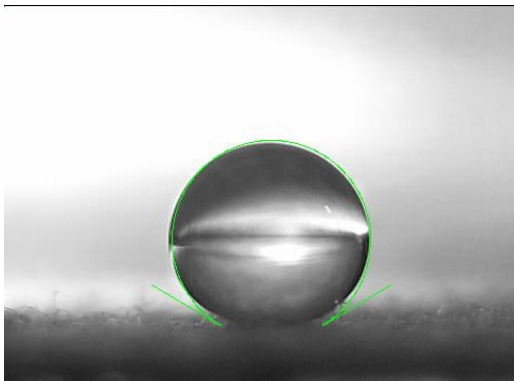
12. Adjust y – Adjust x (moves the green line in the x and y directions)



13. Adjust b (expands and contracts the green line)



14. Adjust h (increases and decreases the length of the green line)

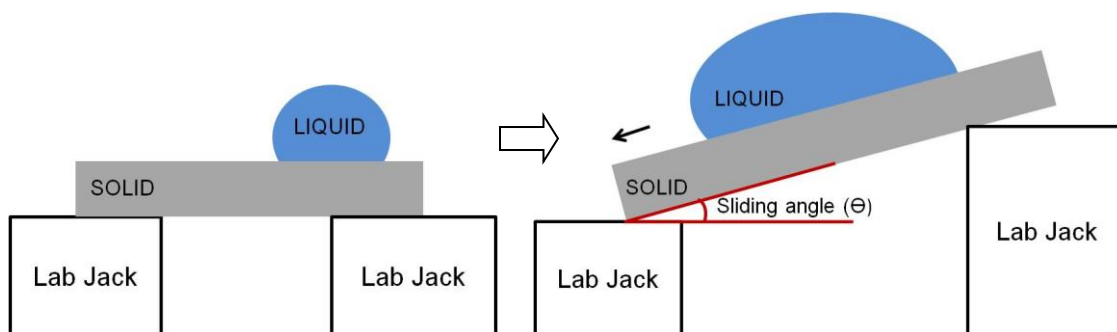


15. Adjust all necessary parameters to get a good fit of the circle at the bottom of the droplet. Write down the contact angle number from the Contact angle (Canvas) line.

Sliding angle measurement procedure

The sliding angle is measured as the lowest inclination angle at which all the drops completely rolled off the surface through sliding.

B.1 Equipment Set-Up:



B.2 Chemicals:

- Water
- Diiodomethane
- Cooking oil
- n-Hexadecane

B.3 Procedure:

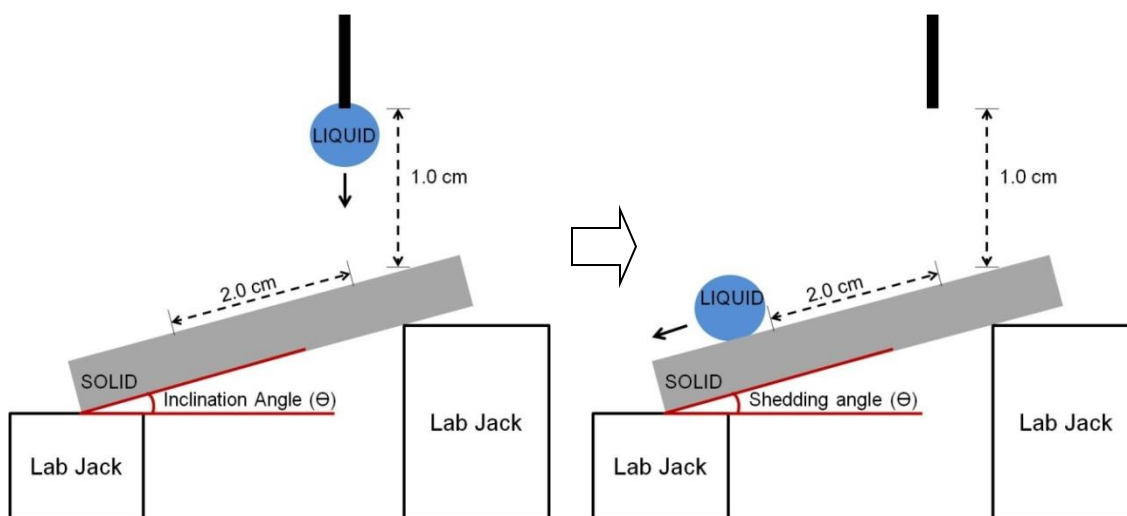
1. Set-up the sliding angle stage at an inclination angle (Θ) of 0° .
2. Put on gloves to avoid contaminating the textile swatches with oils/liquid from hands.
3. Cut out a textile swatch (minimum size 2.5 x 2.5 cm) and attach it to a glass slide using double-sided adhesive tape. If necessary, place tape around the edges of the swatch to make the swatch as flat as possible or place a weight on the swatch to help flatten it.
4. Place the slide on top of the sliding angle stage and tape it to the plate.
5. Add a weight at the bottom of the plate to keep it from moving.
6. Fill a syringe/pipette with the test liquid chemical such as 5.0 or 10.0 μL .
7. Dispense a 5.0 μL droplet of the test liquid and place it on the textile swatch. 10 μL could also be used for testing. If the sample is large enough three droplets could be measured at the same time.

8. Gently tilt the swatch until the droplet/s slides off the surface of the swatch.
9. Measure the angle of the plate (Θ) using a protractor. Alternatively, the height (H) and length (L) of the plate can be measured using a ruler. These values can then be used to calculate the angle of the plate (Θ) using $\Theta = \arctan (H/L)$.
10. If only one droplet was measured, repeat the process until three different positions on the swatch have been measured in total.

Shedding angle measurement procedure

The shedding angle is measured as the lowest inclination angle at which all the drops completely rolled off or bounced off the surface.

C.1 Equipment Set-Up:



C.2 Chemicals:

- Water
- Diiodomethane
- Cooking oil
- n-hexadecane

C.3 Procedure:

1. Set-up the shedding angle stage at an inclination angle (Θ) of 85° . The inclination angle can also be set-up lower if liquids are known to roll off the sample, for example the angle can be set-up at the sliding angle. Measure the angle of the plate (Θ) using a protractor. Alternatively, the height (H) and length (L) of the plate can be measured using a ruler. These values can then be used to calculate the angle of the plate (Θ) using $\Theta = \arctan (H/L)$.
2. Put on gloves to avoid contaminating the textile swatches with oils/liquid from hands.
3. Cut out a textile swatch (minimum size 2.5 x 2.5 cm) and attach it to a glass slide using double-sided adhesive tape. If necessary, place tape around the edges of the swatch to make the swatch as flat as possible or place a weight on the swatch to help flatten it.
4. Place the slide on top of the shedding angle stage and tape it to the plate.




5. Add a weight at the bottom of the plate to keep it from moving.
6. Fill the syringe/pipette with the test liquid chemical such as 10.0 or 5.0 μL .
7. Mount the syringe above the textile swatch (or hold it in your hand) at a distance of 1.0 cm between the needle tip and the surface of the swatch and at least 2.0 cm from the bottom edge of the swatch.
8. Release a 10.0 μL droplet of test liquid. If possible also use a 5.0 μL droplet for testing.
9. Observe the behaviour of the droplet.
10. Move the swatch and release another droplet onto this new position. Repeat until three different positions on the swatch have been measured in total.
11. If all three droplets completely bounced off or rolled off 2.0 cm. Reduce the inclination angle by 5 or 10°. Measure the angle of the plate (Θ) using a protractor. Alternatively, the height (H) and length (L) of the plate can be measured using a ruler. These values can then be used to calculate the angle of the plate (Θ) using $\Theta = \arctan (H/L)$.
12. Repeat the procedure until one or more droplets do not completely roll off the surface or become stuck at the impact point.
13. The last angle where all the droplets rolled off the swatch is the shedding angle.



Photographs of results

D.1 Standard test methods

D.1.1 ISO 4920

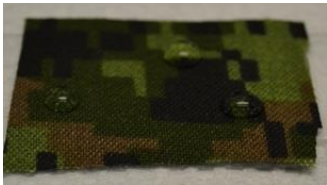
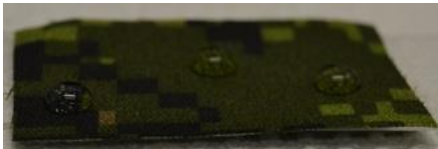
Table 6: ISO 4920 results.

Sample	Image	Result
H		ISO 5 (100)
R		ISO 5 (100)
S		ISO 4 (90)

Sample	Image	Result
N		ISO 5 (100)
F		ISO 4.5 (95)

D.1.2 AATCC 193

Table 7: AATCC 193 aqueous solution 6 results.

Sample	Image	Result
H		A
R		A

UNCLASSIFIED // NON-CONTROLLED GOODS



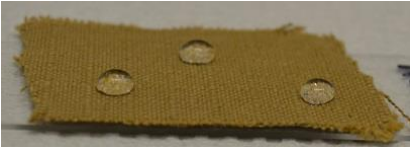


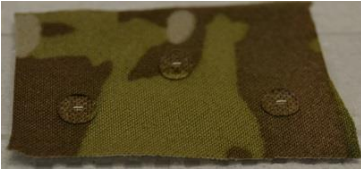
Sample	Image	Result
S		A
N		A
F		A

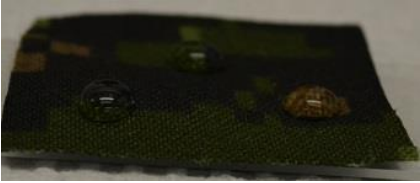

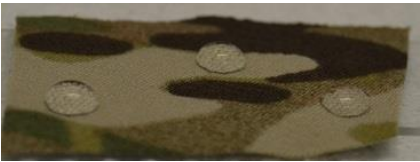
Table 8: AATCC 193 aqueous solution 7 results.

Sample	Image	Result
H		A
R		A
S		A

UNCLASSIFIED // NON-CONTROLLED GOODS

Sample	Image	Result
N		A
F		A

Table 9: AATCC 193 aqueous solution 8 results.

Sample	Image	Result
H		A
R		A
S		A

UNCLASSIFIED // NON-CONTROLLED GOODS





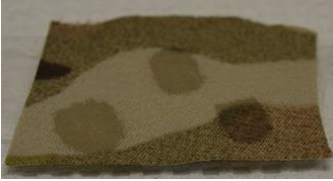
Sample	Image	Result
N		A
F		A

Table 10: Isopropyl alcohol results.


Sample	Image	Result
H		C
R		A
S		D

UNCLASSIFIED // NON-CONTROLLED GOODS

Sample	Image	Result
N		C
F		C

D.1.3 AATCC 118

Table 11: AATCC 118 oil 3 n-hexadecane results.

Sample	Image	Result
H		A
R		A
S		A

UNCLASSIFIED // NON-CONTROLLED GOODS


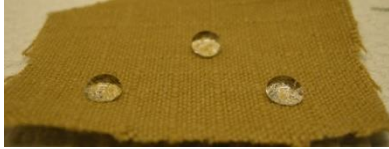




Sample	Image	Result
N		A
F		A

Table 12: AATCC 118 oil 4 n-tetradecane results.

Sample	Image	Result
H		A
R		A
S		A
N		A

UNCLASSIFIED // NON-CONTROLLED GOODS


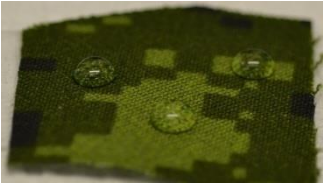



Sample	Image	Result
F		A

Table 13: AATCC 118 oil 5 n-dodecane results.

Sample	Image	Result
H		A
R		A
S		D
N		A

UNCLASSIFIED // NON-CONTROLLED GOODS


Sample	Image	Result
F		A

Table 14: AATCC 118 oil 6 n-decane results.





Sample	Image	Result
H		B
R		A
N		B
F		C (but borderline on B)

Table 15: AATCC 118 oil 7 n-octane results.

UNCLASSIFIED // NON-CONTROLLED GOODS





Sample	Image	Result
H		C (but borderline on B)
R		B
N		D
F		D

Table 16: AATCC 118 oil 8 n-heptane results.

Sample	Image	Result
H		D

UNCLASSIFIED // NON-CONTROLLED GOODS


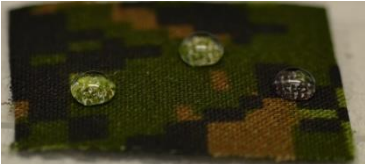
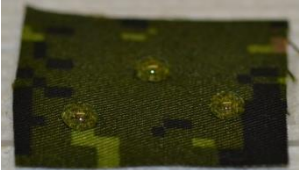


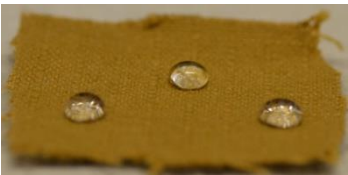
Sample	Image	Result
R		C

Table 17: Methyl salicylate results.

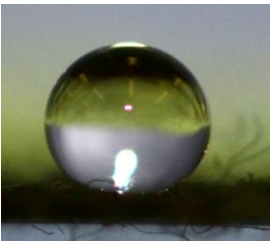
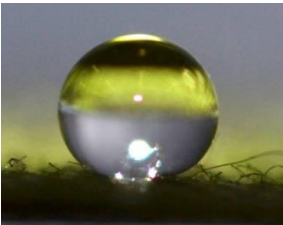

Sample	Image	Result
H		A
R		A
S		A
N		A

Sample	Image	Result
F		A

D.2 Research test methods

D.2.1 Contact angle test

Table 18: Water contact angle results.

Sample	Image	Result
H		150
R		154
S		144

UNCLASSIFIED // NON-CONTROLLED GOODS





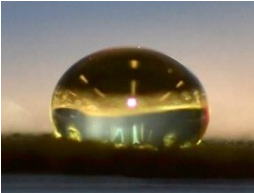
Sample	Image	Result
N		152
F		151

Table 19: Diiodomethane contact angle results.

Sample	Image	Result
H		139
R		119
S		126

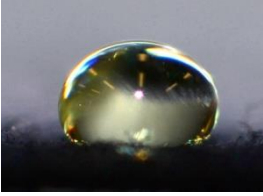
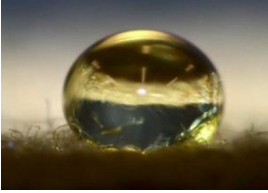
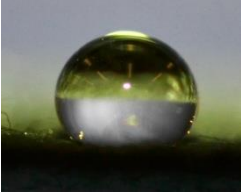
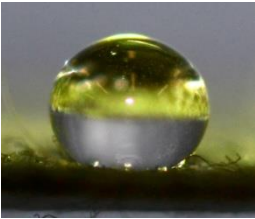

Sample	Image	Result
N		132
F		131

Table 20: Cooking oil contact angle results.

Sample	Image	Result
H		139
R		141
S		132

UNCLASSIFIED // NON-CONTROLLED GOODS




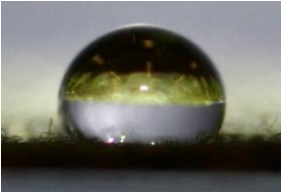
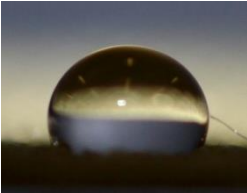


Sample	Image	Result
N		141
F		140

Table 21: n-Hexadecane contact angle results.

Sample	Image	Result
H		123
R		123
S		117

UNCLASSIFIED // NON-CONTROLLED GOODS

Sample	Image	Result
N		121
F		125

List of abbreviations and acronyms

°	Degree
°C	Degrees Celsius
µL	Microliter
AATCC	American Association of Textile Chemists and Colorists
C8	Perfluoroalkyl chains with eight fluorinated carbons (C ₈ F ₁₇ -)
CAN	Canada
CGSB	Canadian General Standards Board
F	Sample firefighter turnout gear fabric
H	Sample CB protective fabric
ISO	International Organization for Standardization
m	Meter
mN	Millinewton
N	Sample CB protective fabric
RMCC	Royal Military College of Canada
R	Sample rain protective fabric
S	Sample CB protective fabric

DOCUMENT CONTROL DATA		
*Security markings for the title, authors, abstract and keywords must be entered when the document is sensitive		
1. ORIGINATOR (Name and address of the organization preparing the document. A DRDC Centre sponsoring a contractor's report, or tasking agency, is entered in Section 8.) Calian Technologies and Dept. of Chemistry and Chemical Engineering Royal Military College of Canada PO Box 17000, Station Forces Kingston, Ontario K7K 7B4		2a. SECURITY MARKING (Overall security marking of the document including special supplemental markings if applicable.) CAN UNCLASSIFIED
		2b. CONTROLLED GOODS NON-CONTROLLED GOODS DMC A
3. TITLE (The document title and sub-title as indicated on the title page.) Evaluation of Liquid Repellent Fabrics		
4. AUTHORS (Last name, followed by initials – ranks, titles, etc., not to be used) Grozea, C.M.		
5. DATE OF PUBLICATION (Month and year of publication of document.) March 2018	6a. NO. OF PAGES (Total pages, including Annexes, excluding DCD, covering and verso pages.) 49	6b. NO. OF REFS (Total references cited.) 16
7. DOCUMENT CATEGORY (e.g., Scientific Report, Contract Report, Scientific Letter.) Contract Report		
8. SPONSORING CENTRE (The name and address of the department project office or laboratory sponsoring the research and development.) DRDC – Suffield Research Centre Defence Research and Development Canada P.O. Box 4000, Station Main Medicine Hat, Alberta T1A 8K6 Canada		
9a. PROJECT OR GRANT NO. (If appropriate, the applicable research and development project or grant number under which the document was written. Please specify whether project or grant.) 06cc—Protect	9b. CONTRACT NO. (If appropriate, the applicable number under which the document was written.) W4938-17-31S	
10a. DRDC PUBLICATION NUMBER (The official document number by which the document is identified by the originating activity. This number must be unique to this document.) DRDC-RDDC-2018-C229	10b. OTHER DOCUMENT NO(s). (Any other numbers which may be assigned this document either by the originator or by the sponsor.) CPT-1803	
11a. FUTURE DISTRIBUTION WITHIN CANADA (Approval for further dissemination of the document. Security classification must also be considered.) Public release		
11b. FUTURE DISTRIBUTION OUTSIDE CANADA (Approval for further dissemination of the document. Security classification must also be considered.)		

12. KEYWORDS, DESCRIPTORS or IDENTIFIERS (Use semi-colon as a delimiter.)

liquid repellency; test method

13. ABSTRACT/RÉSUMÉ (When available in the document, the French version of the abstract must be included here.)

Fabrics coated with liquid repellent finishes can help to protect personnel from both hazardous toxic substances and everyday contaminants. Three standard test methods (ISO 4920, AATCC 193 and AATCC 118) and three test methods developed by university research laboratories (contact angle, sliding angle and shedding angle tests) were set up and used to measure five textiles coated with liquid repellent finishes, in order to establish some baseline expectations for results using these methods at the Royal Military College. The fabrics showed some variations in liquid repellency towards water, various oils, and the chemical warfare agent simulant methyl salicylate. The tests can be further expanded to include more realistic attributes such as movement and pressure.

Les textiles enduits d'une couche résistante aux liquides peuvent aider à protéger le personnel contre les substances toxiques dangereuses et les contaminants quotidiens. Trois méthodes d'essai standard (ISO 4920, AATCC 193 et AATCC 118), et trois méthodes d'essai développées par les laboratoires de recherche universitaires (angle de contact, angle de glissement et angle de chute) ont été mises en place et utilisées pour mesurer cinq textiles enduit d'une couche résistant aux liquides, afin d'établir certaines attentes de base pour les résultats en utilisant ces méthodes au Collège Militaire Royal du Canada. Les textiles présentaient certaines variations de répulsion des liquides vis-à-vis l'eau, diverses huiles et l'agent de guerre chimique simulant le salicylate de méthyle. Les tests peuvent être élargis pour inclure des attributs plus réalistes telles que le mouvement et la pression.