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MANAGEMENT PERSPECTIVE

It is known that flocculation significantly alters the settling characteristics of fine-grained suspended sediment. This has important consequences for sediment transport modelling of pathways, fate and effects of sediment-associated contaminants. Traditional sediment sizing techniques are indirect methods which make no attempt to maintain the original grain-size distribution. This report provides an inexpensive, non-destructive, direct observation alternate method of sediment sizing. This method has demonstrated that flocculation is an important process occurring in rivers which produces relatively stable flocs.

ABSTRACT

Suspended solids play an important role in the fluvial transport of hydrophobic contaminants. The important components of the suspended solid load for geochemical transport are silts, clays, iron and manganese oxides and organic carbon. Sedimentologists conventionally have assumed that inorganic fine-grained sediment moves as primary particles. Recent findings, however, suggest that primary particles in rivers are frequently and perhaps characteristically transported as larger particles due to flocculation.

Typically floc size has been determined by destructive, indirect laboratory methods or by laborious microscopic measurements. Using the suspended solids from Sixteen-Mile Creek in Southern Ontario a unique direct observation digitizing method of floc characteristic analysis was developed. Using this method of floc handling and analysis it was found that flocculation is a prominent process occurring in the freshwater fluvial system. Results also indicate that flocs are relatively stable within the realms of the sampling and analytical method.

The digitizing method allows for direct observation of floc structures which is impossible with conventional methods of sediment sizing. Results show that the method provides reasonable results with good precision on floc equivalent spherical diameter, surface area, perimeter and circularity. It is however, limited by its labour intensive nature, possibility of sediment overlap, the individual investigator's criteria, and by the photographic and microscopic instruments and techniques used. Comparison of this method to predict

PERSPECTIVE-GESTION

On sait que la floculation a un effet important sur la sédimentation des particules fines en suspension. Ce facteur doit être pris en considération dans la modélisation des voies, du devenir et des effets des contaminants se liant aux sédiments et charriés par ces derniers. Les techniques classiques d'analyse granulométrique sont indirectes et n'essaient pas de maintenir la distribution granulométrique originale. Ce rapport présente une nouvelle méthode non coûteuse d'analyse granulométrique par observation directe qui maintient l'intégrité des échantillons. Cette méthode a révélé que la floculation est un phénomène important dans les rivières et que les flocons ainsi produits sont relativement stables.

floc size distributions between investigators or with other methods may produce different results.

Key Words: Flocculation, River, Freshwater, Suspended Solids.

RÉSUMÉ

Les particules en suspension jouent un rôle important dans le transport des contaminants hydrophobes dans les cours d'eau. Les limons, les argiles, les oxydes de fer et de manganèse et le carbone organique sont les plus importants composants de la charge de particules responsables du transport géochimique. De façon générale, les sédimentologues ont toujours considéré que les sédiments fins inorganiques sont charriés sous forme de particules élémentaires. Des observation récentes laissent cependant entendre que les particules élémentaires des rivières formeraient souvent, voire typiquement, par floculation, des particules plus grosses et seraient transportées sous cette forme.

Habituellement, la taille des flocons est déterminée au moyen de méthodes de laboratoire indirectes et qui les détruisent ou par des mesures laborieuses au microscope. Des échantillons de particules en suspension du ruisseau Sixteen-Mile, situé dans le sud de l'Ontario, ont été utilisés pour mettre au point une méthode originale d'analyse des caractéristiques des flocons. L'analyse se fait par observation directe et numérisation. Cette méthode de manipulation et d'analyse des flocons a révélé que la floculation est un phénomène important dans les eaux douces courantes. Les résultats obtenus montrent aussi que les flocons sont relativement stables, sous réserve des effets possibles de la méthode d'échantillonnage et d'analyse à cet égard.

Cette méthode par numérisation permet une observation directe de la structure des flocons, ce qui est impossible avec les méthodes d'analyse granulométrique classiques. Elle donne des mesures satisfaisantes et suffisamment précises du diamètre sphérique

équivalent, de la surface, du périmètre et de la sphéricité des flocons. Cependant cette méthode a certaines limites : elle demande beaucoup de travail, il peut y avoir chevauchement des sédiments, les résultats peuvent varier d'un individu à l'autre suivant les critères de chacun et les instruments et techniques de photographie et de microscopie ont leurs limites. Si on comparait les résultats d'analyses granulométriques effectuées par différents individus au moyen de cette méthode, ou si on comparait ces résultats avec ceux obtenus avec d'autres méthodes, on observerait peut-être des différences.

Mots clés : floculation, rivière, eaux douces, particules en suspension

INTRODUCTION

The significance of suspended solids in biological and chemical processes in the aquatic environment has long been recognized. They are also an important vector for the transport of contaminants in river systems (Allen, 1986 and Ongley et al., 1988). The important components of the suspended load for geochemical transport tend to be silts, clays, hydrous iron and manganese oxides (micronodules or contings) and organic carbon (Horowitz and Elrick, 1987; Horowitz, 1985; Gibbs, 1973).

Although river sedimentologists refer to suspended sediment as sand, silt or clay size fractions, little is known of the true nature of particles in suspension. Size fractions tend to be operationally defined by the use of conventional dispersion techniques to produce distributions of primary mineral particles, or by sampling and settling techniques that are unlikely to maintain the structure or distribution of sediment aggregates that were originally collected in the field. Generally, river science has lacked instrumentation for in situ and non-destructive measurement of suspended particles. Conventional wisdom of particle transport in rivers typically assumes that fine-grain sediment moves as primary particles. Recent findings illustrate that primary particles are frequently and perhaps characteristically transported as flocculated particles (Droppo and Ongley, 1989; Partheniades, 1986; Kranck, 1984 and 1981).

To improve our understanding of the significance of flocculation in the fluvial environment we studied the suspended particle characteristics of Oakville Sixteen-Mile Creek in Ontario (Figure 1). Our objectives were to: (1) develop a non-destructive, direct observation method for determining floc characteristics such as equivalent spherical diameter (ESD), surface area, perimeter, and circularity; (2) determine the extent to which flocculation is present in the freshwater fluvial system; (3) determine the stability of flocs; and (4) establish the advantages and disadvantages of the sampling and analytical method.

Sampling Site

Sixteen-Mile Creek, situated in Southern Ontario, drains into Lake Ontario between Toronto and Hamilton (Figure 1). The basin is approximately 350 km^2 of which 50 km^2 is urban and the remainder primarily agricultural and forested land.

Two sampling sites were used in the study (Figure 1). Site 1 is a harbour which receives sewage effluent from the sewage treatment plant in the town of Milton upstream. Other sources of particulate matter come from stormwater runoff from urban, agricultural and forested areas. Additional pollution results from extensive pleasure craft traffic during open water periods. Site 2 is upstream and receives only surface and subsurface runoff from forested and agricultural land. This site is relatively clean in comparison, exhibiting lower concentrations of major ions, particulate organic carbon and suspended solids during base flow periods.

MATERIALS AND METHODS

Floc Analysis Overview

The method developed for floc sampling and analysis uses some procedures originally developed for the analysis of plankton;

equipment includes settling chambers, an inverted microscope and a translucent digitizer. In general, a water sample is collected into a settling chamber (Figure 2) where the suspended solids settle under gravity onto an inverted microscope slide. The slide is placed on an inverted microscope and photographs (slides) of the flocs are taken through the microscope. The transparencies are rear-projected onto a translucent digitizer to obtain digital output.

Determination of Settling Chamber Size

The microscope and digitizer are constants in the analysis. The size (volume) of the settling chamber, however, varies with suspended solid concentration. The required volume of the settling chamber is directly proportional to the number of flocs settled which, in turn, is a function of suspended solids (SS) concentration. It is statistically important to utilize the proper chamber volume for a given SS concentration. Too large a volume results in overlapping of the settled sediment particles and increases the probability of flocculation due to differential settling. This may result in an over-estimation of floc size. Too small a volume results in an insufficient number of flocs for statistically significant analysis.

The correct settling chamber volume for field use can be determined directly from measurements of turbidity. The relationship between turbidity and suspended solids (Figure 3) is determined from field measurements. Turbidity was measured with an HF Scientific turbidity meter (Model DRT-15B) and suspended solid concentrations determined by filtration through pre-dried and tared 0.45 μm Millipore filters. For each grab sample, all settling chamber volumes (3 mL, 5 mL, 10 mL, 25 mL, and 50 mL) were filled, settled and viewed under an inverted microscope in order to determine which column, for the given SS concentration, yielded the best results with a minimum of sediment overlap but a significant number of flocs. Appropriate settling chamber volumes are subsequently determined directly from the rating curve (Figure 3).

Floc Sampling

This is presumed to be the most critical aspect of the overall procedure because of the potential for some floc breakage during sampling and transport (Bale and Morris, 1987). Sample handling is minimized to reduce the probability of disaggregation or breakage.

Suspended solids were collected directly into the appropriate settling chamber by placing the settling chamber under the surface of the water, parallel to the direction of flow. The chamber was filled to capacity and capped under water to minimize turbulence in the chamber. The chamber was then transported to the laboratory in an upright position. This method of transportation was found by Cleary et al (1987) to be least destructive to soil aggregates suspended in water. Flocs were allowed to settle for 3 to 4 hours prior to analysis or until the supernatent was clear.

Floc Digital Analysis

The method used to determine floc size is a combination of microscopy, photography and digitization. Once the suspended solids settled out onto the settling chamber slide (Figure 2), inverted microscope (Wild Leitz) photographs were taken of the sediment at 100x magnification. Seventeen evenly distributed stage positions (fields of view) were taken for each suspended solids sample. A transparency of a 1/16 mm^2 standardizing grid scale was used for scale correction. These transparencies were then projected onto a Scriptel translucent digitizer where the floc's perimeter was digitized. The Scriptel digitizer has a resolution of 0.025 mm and an accuracy of +/- 0.64 mm (Scriptel Corporation, Columbus, Ohio, USA 1987). A particle was considered to be a floc if it contained two or more primary inorganic particles. The digital output was converted to floc surface area, floc perimeter, floc equivalent spherical diameter and floc circularity. The equivalent spherical diameter was manipulated to yield grain size distributions by number and by volume.

Equivalent spherical diameter was used as it is the standard unit of measure for most sizing instruments. This allows for comparison of size distributions produced by digitization with those produced by other standard methods based on equivalent spherical diameter. We recognized, however, that equivalent spherical diameter may not be the best determinant of floc size as it eliminates any unique features of a floc in terms of perimeter and shape. In other words, two flocs with the same equivalent spherical diameter may have completely different perimeters and shapes. Equivalent spherical diameter (ESD) was calculated using the equation;

$$ESD_i = \sqrt{4.A_i/\pi}$$

where A_i = area of floc i.

The floc circularity (c) was calculated by the equation;

$$C = (4\pi)\ (\text{AREA})/\text{PERIMETER}^2$$

This ratio of floc area to the area of a circle with the same perimeter as the floc, is close to 1.0 for a circular floc and close to 0.0 for a linear floc.

RESULTS AND DISCUSSION

Floc Stability

Floc stability and the ability of the sampling, transport and analytical method to retain the in situ floc structures are crucial to the validity of the method. From the marine literature it is generally believed that flocs are very fragile and the act of sampling will cause disaggregation (Kranck, 1984; Gibbs and Konwar, 1982; Kranck and Milligan, 1980). Floc development in the marine environment are highly dependent on electrochemical flocculation due to the high salt content which suppresses the electrical double layer thickness. In freshwater fluvial systems, electrochemical forces of flocculation appear to be less important than biological processes in the formation of flocs (Tsernoglou and Anthony, 1971; Pearl, 1975, 1974 and 1973).

This study makes the assumption that the flocs retain their structure during sampling into the settling chamber. Although there is no way of directly testing this assumption, the observations and experiments on floc stability below support the assumption. However, it is possible that the flocs analyzed here may be sub-aggregates of larger structures which were broken during sampling.

In one test, settled flocs were shaken on the settling chamber slide during viewing through the microscope. It was observed that the flocs rolled from side to side in a jelly-like fashion and did not break up during this disturbance. Direct measurements of floc strength are limited; however, from rheological studies it has been found that cohesive sediment suspensions behave like pseudoplastics (van Leussen, 1988). This strength may be highly correlated with the ability of some bacteria to secrete a sticky organic extracellular polymeric substance which helps bond particles together (van Leussen, 1988; Biddanda, 1985; Paerl, 1974). Using 1.0 µm Nuclepore filters and the epifluorescence microscopy techniques of Rao et al. (1984), flocs from Sixteen-Mile Creek had bacterial counts ranging from 10^7 to 10^8 count/mg of suspended sediment (Droppo, 1989). Particulate organic carbon concentrations (using the methods of Environment Canada, 1974) ranged from 0.12 mg/l in late summer to 12.4 mg/l during spring melt (Droppo, 1989). The compact and stable structure of these flocs may also be enhanced by the fact that these flocs were formed in a turbulent environment. According to Tsai et al., (1987), Partheniades, (1986) and Krone, (1987), the larger the shear stress, the smaller and stronger the floc becomes.

The stability of flocs was also seen in their durability during transport. In an experiment, photographs of specific stage positions were taken of flocs settled on a settling chamber slide in the laboratory from a water grab sample. The slide was then driven a normal sampling distance in a car (approximately 40 Kms) and the same stage positions subsequently photographed and digitized. Before and after transport photos revealed no significant difference between the mean floc areas (two sample, two tail T-test - pooled - $\alpha = 0.05$) (Droppo, 1989). Direct microscopic observation also revealed that the flocs did not move from their original settled position on the slide during transport. In a separate experiment flocs were settled in two identical chambers (10 mL) from a water grab sample. Photographs from the same stage positions were digitized for the one chamber with flocs settled in the lab and for the other chamber whose flocs were settled during a normal transport distance. Once again there was no significant difference between the mean floc areas of the two samples (two sample, two tail T-test - pooled - $\alpha = 0.05$) (Droppo, 1989). The results of these observations and experiments lead to the conclusion that flocs in this fluvial environment appear to be stable and that the act of sampling and transporting flocs does not result in significant floc breakage.

Importance of Flocs to Total Suspended Solid Transport

Figure 4 and Figure 5 clearly demonstrate that suspended sediment within Sixteen-Mile Creek is flocculated. Figure 4 illustrates the suspended sediment of Sixteen-Mile Creek in its natural form on March 25, 1989. The same sediment is sonicated in Figure 5 to show the constituent primary particles. The histogram in Figure 6 illustrates the flocculated and dispersed distributions of the same environmental sample from March 15, 1989. The flocculated distribution was significantly shifted into the larger size classes in comparison with the primary particles. Primary grain sizes ranging from 1 to 2.6 microns were entirely incorporated into the matrices of the flocs.

Over the sampling period it was found (by digitizing flocculated and primary mineral particles appearing on the same slides) that while flocs comprised only 10 to 27% of the total number of particles, they represented 92 to 97% of the total volume of suspended sediment (Droppo, 1989). This finding is similar to that of Schubel and Kana (1972) for an estuarine environment where flocs of 3 primary grains or more made up only 11% of the total number of particles but nearly 97% of the total sediment volume. These results demonstrate the importance of flocs to the total suspended solids transported within Sixteen-Mile Creek.

Floc Geometry

Geometric characteristics of flocs are produced here for samples characteristic of each season (summer base flow, winter ice cover and spring peak flow). Figure 7 illustrates the equivalent spherical diameter (ESD) distribution for all three seasons. All three distributions exhibit the same positive skewness and predominant size classes. The positive skewness indicates that the majority of flocs are in the smaller size ranges with fewer flocs in the larger size classes. The predominant sizes are between the 5 and 10 µm class with the median and mean values at the high end of this range. While the mean floc sizes for the three days are all relatively close, a one way analysis of variance ($\alpha = 0.01$) indicates that there is significant difference in floc ESD among the three distributions. This indicates that the winter ice cover conditions possesses the smallest floc size distribution followed by the summer base flow and peak spring melt period respectively.

Flocs in Sixteen-Mile Creek are small in comparison to some estuarine floc studies. For example, Bale and Morris (1987), using a Malvern particle sizer _in situ_, found flocs larger than 188 µm (ESD) in the Tamar Estuary. Freshwater fluvial flocs were expected to be smaller due to the low salt content of the water. Rao _et al_. (1989) found the predominant size fraction in the Yamaska River in Quebec, Canada to be between 20 - 40 µm. Their method of floc sizing (Malvern Particle Size Analyser - Model 2600 C) and handling was, however, entirely different.

ESD, although the standard of measurement for many sizing techniques, ignores the high degree of variability in floc shape. Shape is an important variable in sediment (floc) settling velocity. Figure 8 represents the circularity distribution for the three different flow conditions. Floc shape tends towards a circular shape with mean and median values ranging between 0.64 and 0.66 in the summer to 0.67 and 0.69 in the winter respectively. While the mean values are similar, a one way analysis of variance ($\alpha = 0.01$) indicates that there is a significant difference between the circularity of the flocs among the three seasonal days with winter flocs being more circular than in summer.

Figures 9 and 10 illustrate similar floc surface areas and perimeter distributions for the three seasonal days with the largest number of flocs between 20-40 μm^2 and 20-40 µm respectively. A one way analysis of variance ($\alpha = 0.01$) indicates that the perimeters of flocs are significantly different among the three days but surface areas are not. The data are all positively skewed.

Evaluation of the Digitizing Method

This system has the advantage of being an image analysis system. It allows for the direct observation of the flocs in the microscope and on the digitizing tablet. As seen in Figure 11, inorganic and organic grains as well as inter-grain pores may be viewed. Direct microscopic observation also reveals microorganism grazers feeding on the flocs and swimming in the fluid. There were no instances where grazers broke up the flocs over the many hours of viewing. While 100x magnification was used throughout this study, different magnifications can be useful for examining floc structure.

Although the manual tracing of flocs is time consuming, it does allow the investigator to trace only those flocs which meet the study criteria. Automated image analysis systems work on the basis of pixel light intensity which operates on the assumption that similar objects will reflect the same range of light intensity provided their compositions are the same. Because flocculated material is composed of both organic and inorganic material, its light reflecting properties may vary widely. Often non-floc material will also have the same light intensity as the flocculated material and thus error may be introduced into the data set by automated floc selection.

The digitization method is also useful in characterizing floc circularity, perimeter, equivalent spherical diameter and surface area. Although percent finer by volume distributions produced by the digitizing method overestimate the importance of a small number of larger particles, we believed this method is useful for characterizing changes in floc size both temporally and spatially.

The digitizing method has a high degree of precision. Three replicate digitizations of 500 flocs from Site 1 on August 5, 1988 (Table 1) illustrated that the reproducibility of the method for equivalent spherical diameter was statistically acceptable. A one way analysis of variance on the 3 replicate digitizations yielded a F statistic of 0.05, indicating that the null hypothesis (means of the samples are the same) can not be rejected (α=0.05). Therefore, the samples are statistically identical.

While the digitizing method is believed to provide an accurate representation of floc characteristics, it is limited by its dependence on the individual criteria of the investigator. It can also be limited by the photographic and microscopic instruments and techniques used (Li and Ganczarczyk, 1986). In this case the magnification used did not allow for the sizing of particles smaller than 1 μm.

The digitizing method is also very labour intensive which results in small sample sizes of approximately 500 flocs. This number is minimal compared to automated sizing instruments such as the Malvern laser particle sizer which we use to measure size distributions of particle suspensions.

Because of the small floc sample sizes in this study, size classes may not be accurately represented in the percent by volume distribution. For example, the size distributions (in percent by volume) of four different samples were plotted as histograms in Figure 12. Although the last size class generally possesses the largest percent by volume of sediment they generally represent less than 0.55% of the total number of particles digitized. The small sample size therefore over-emphasizes the importance of one or two flocs in a distribution.

CONCLUSIONS

Using the direct observation digitizing method we have demonstrated that suspended solid flocculation is quantitatively, an important process occurring in the fluvial system. Greater than 90% of the total sediment volume is transported as flocs. These flocs appear to be relatively stable within the limits of the sampling and analytical methods used here.

The flocs analyzed from Sixteen-Mile Creek (over one season) are generally much smaller than those analyzed in the marine environment, ranging from approximately 2 to 60 μm in equivalent spherical diameter. Median sizes are generally around 9 μm. The flocs tend towards a circular shape with a median circularity ratio close to 0.7. Analysis of variance revealed a significant difference (α = 0.01) between the three seasonal days for equivalent spherical diameter, circularity and perimeter. No significant difference was found for surface area.

The digitizing method, as it is a direct observational method, is useful for floc structural analysis. It can accurately provide individual floc geometric characteristics with good precision. The method is, however, limited by the individual investigator's criteria, possibility of sediment overlap, photographic and microscopic instruments and techniques, and by its labour intensive nature. Small floc sample sizes restricts the digitizing method from predicting accurate percent by volume distributions. Thus, comparison of this method to predict grain-size distributions between investigators or with other methods may produce significantly different results.

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Figure and Table Legends

Figure 1 Oakville Sixteen-Mile Creek and study sites.

Figure 2 An example of a settling chamber. A - top cap, B - bottom cap and inverted microscope slide, C - 50 ml column.

Figure 3 Rating curve of suspended solid concentration versus turbidity. The optimal column volume is noted on the Y axis.

Figure 4 Naturally flocculated suspension (March 25, 1989, Site 1).

Figure 5 Disaggregated suspension after sonification (March 25, 1989, Site 1).

Figure 6 Comparison of particle-size distribution for a flocculated and dispersed sediment sample (March 15, 1989 Site 1).

Figure 7 Particle size distributions of flocculated suspended solids.

Figure 8 Circularity distributions of flocculated suspended solids.

Figure 9 Surface area distributions of flocculated suspended solids.

Figure 10 Perimeter distributions of flocculated suspended solids.

Figure 11 Natural flocculated suspension (November 17, 1887).

Figure 12 Floc size distributions of four individual samples.

Table 1. Precision test of the digitizing method.

Equivalent Spherical Diameter (μm)			
Sample No.	Mean	Median	Standard Deviation
1	8.815	7.270	5.318
2	8.719	6.951	5.369
3	8.806	7.235	5.309
Mean	8.780	7.152	5.332
SD	0.053	0.175	

SD = standard deviation

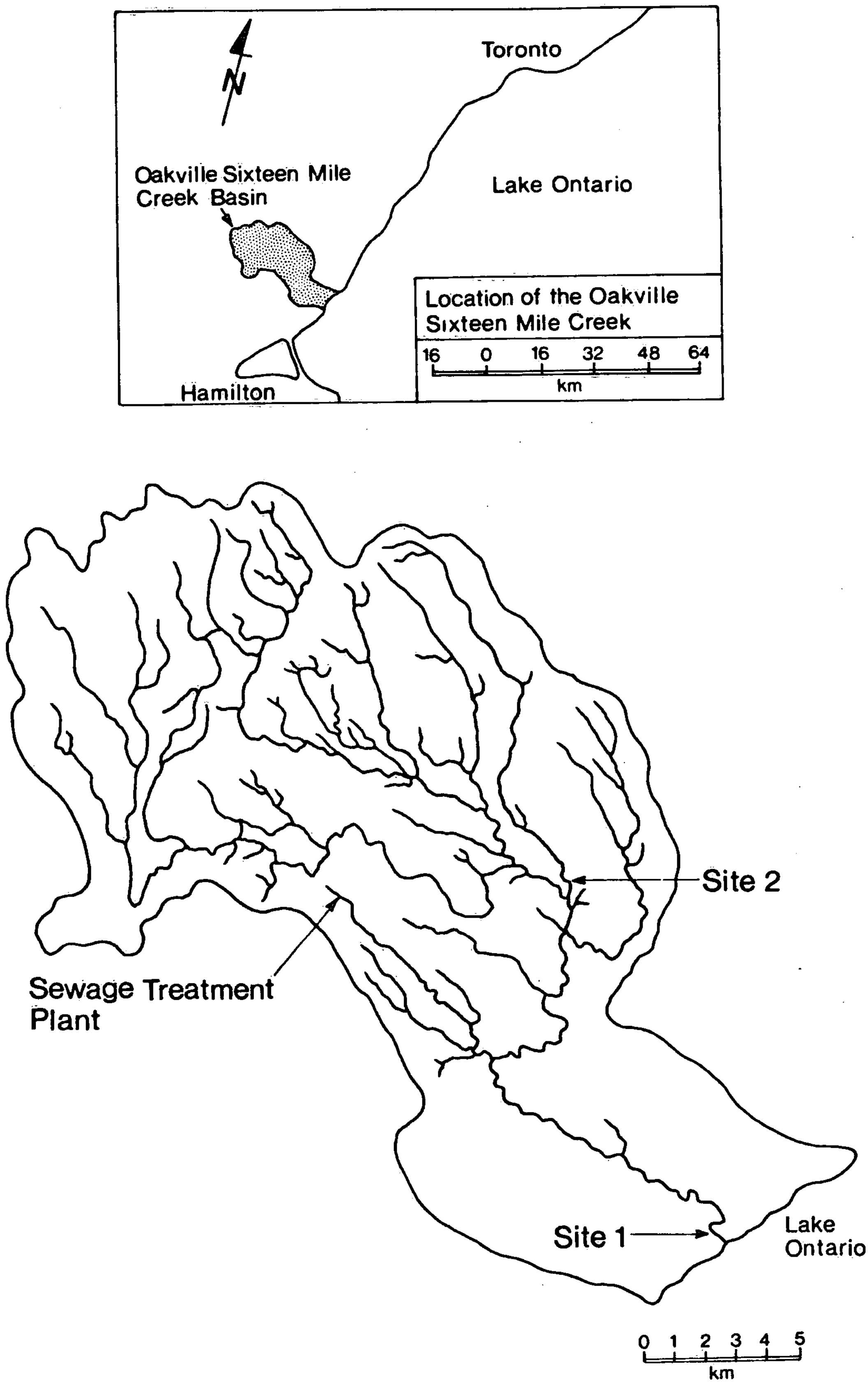

Figure 1 Oakville Sixteen-Mile Creek and study sites.

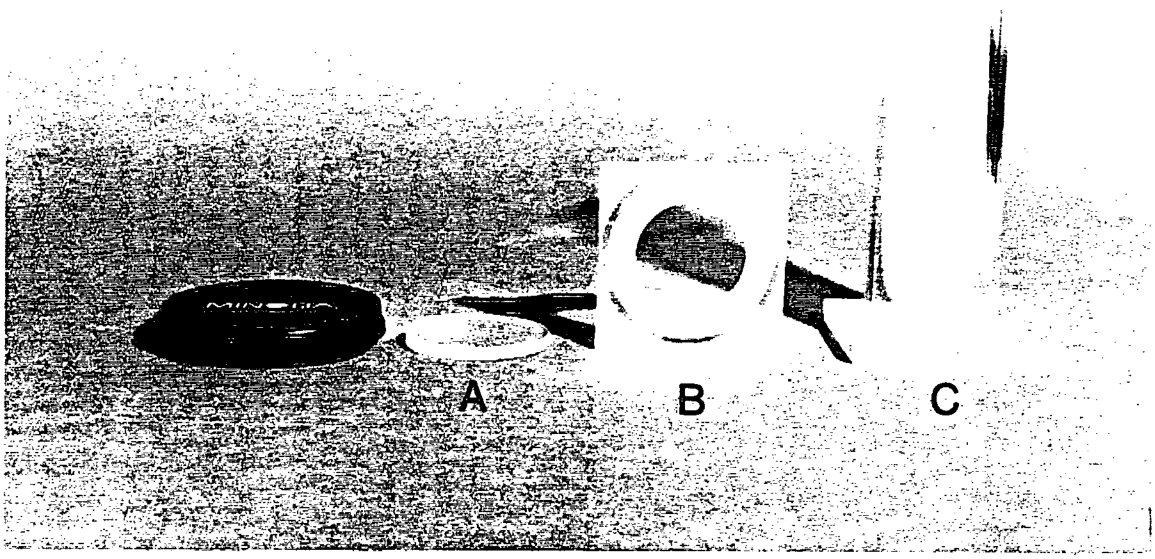

Figure 2 An example of a settling chamber. A - top cap, B - bottom cap and inverted microscope slide, C - 50 ml column.

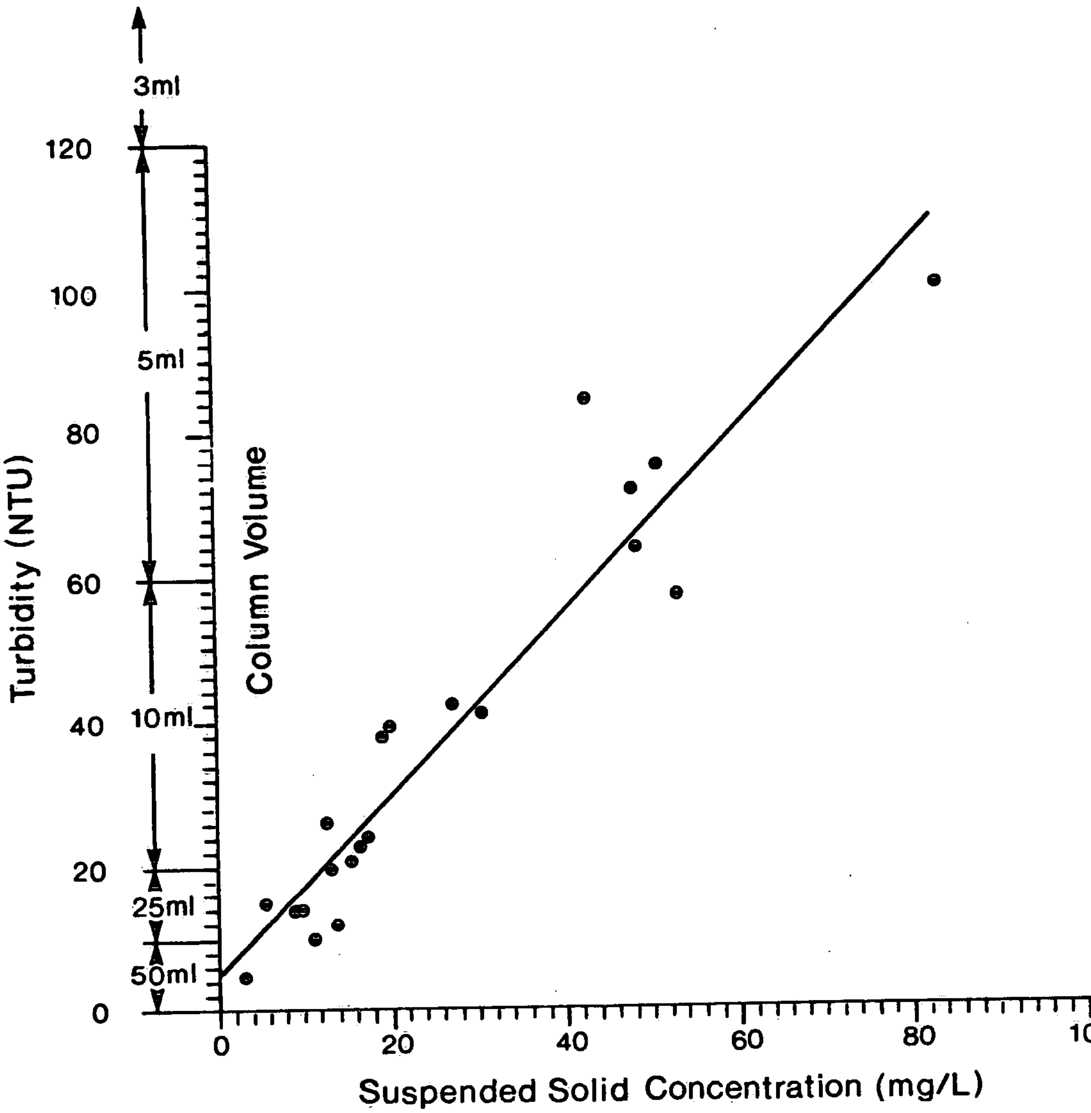

Figure 3 Rating curve of suspended solid concentration versus turbidity. Used to determine the optimal column volume.

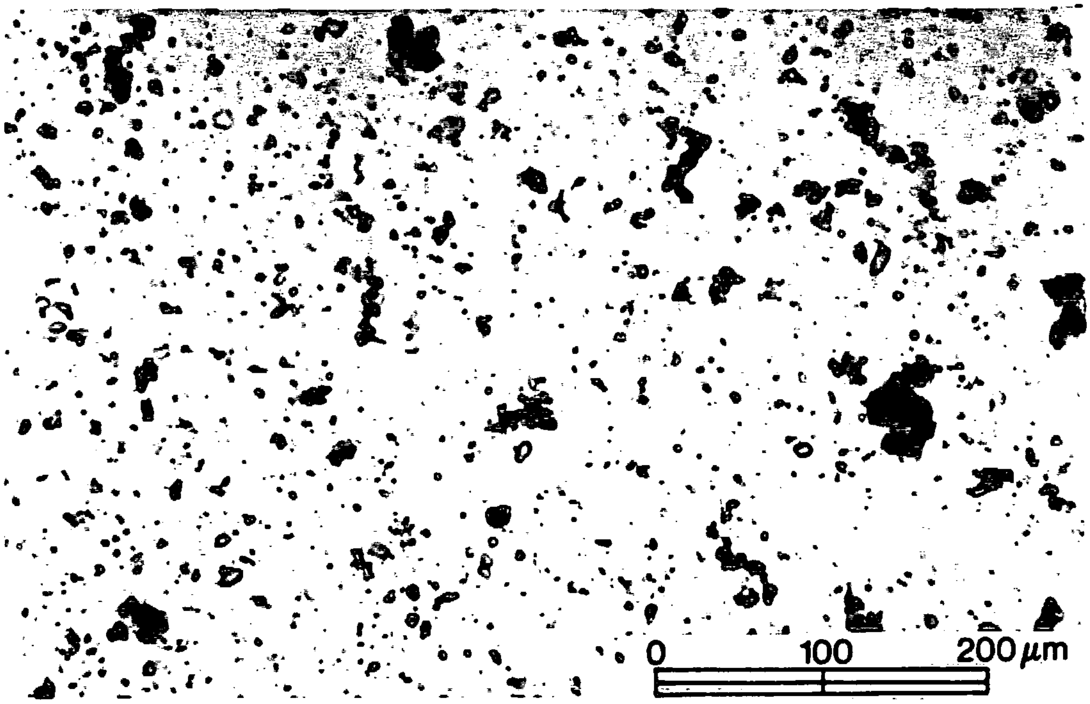

Figure 4 Naturally flocculated suspention (March 25, 1989, Site 1).

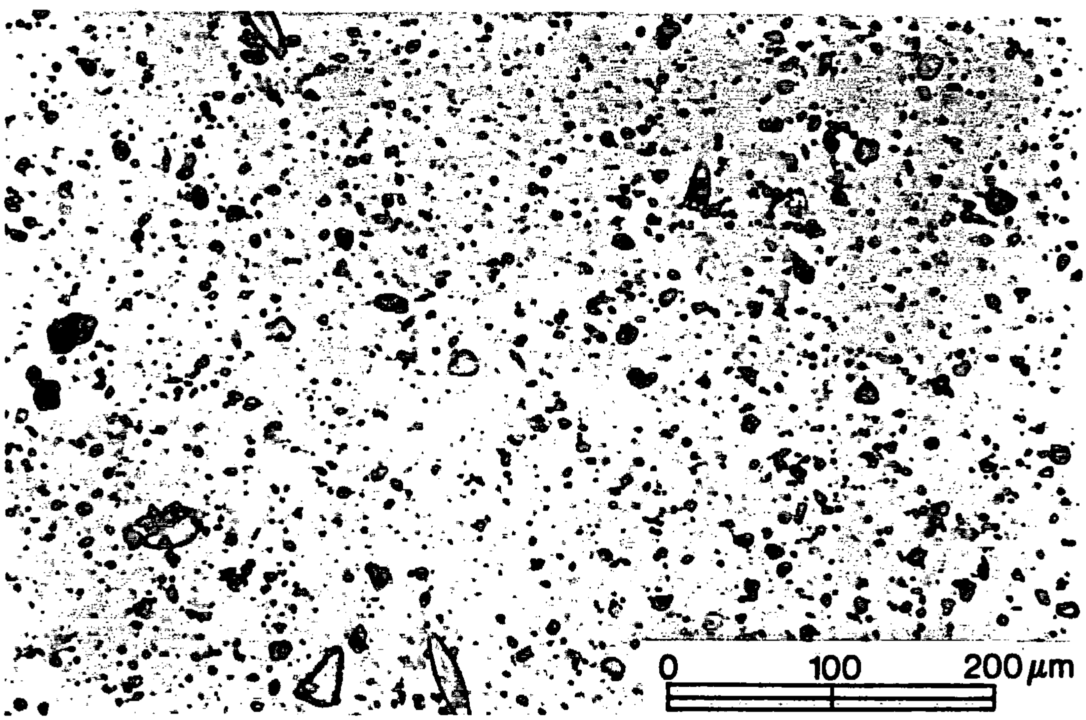

Figure 5 Disaggregated suspension after sonification (March 25, 1989, Site 1).

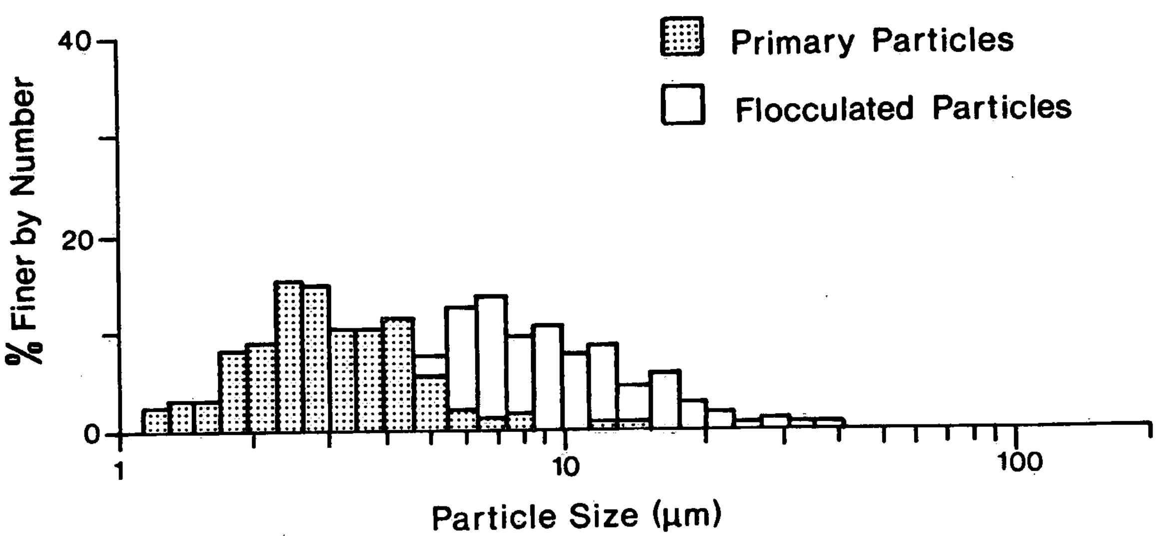

Figure 6 Comparison of a flocculated and deflocculated sediment sample (March 15, 1989 Site 1).

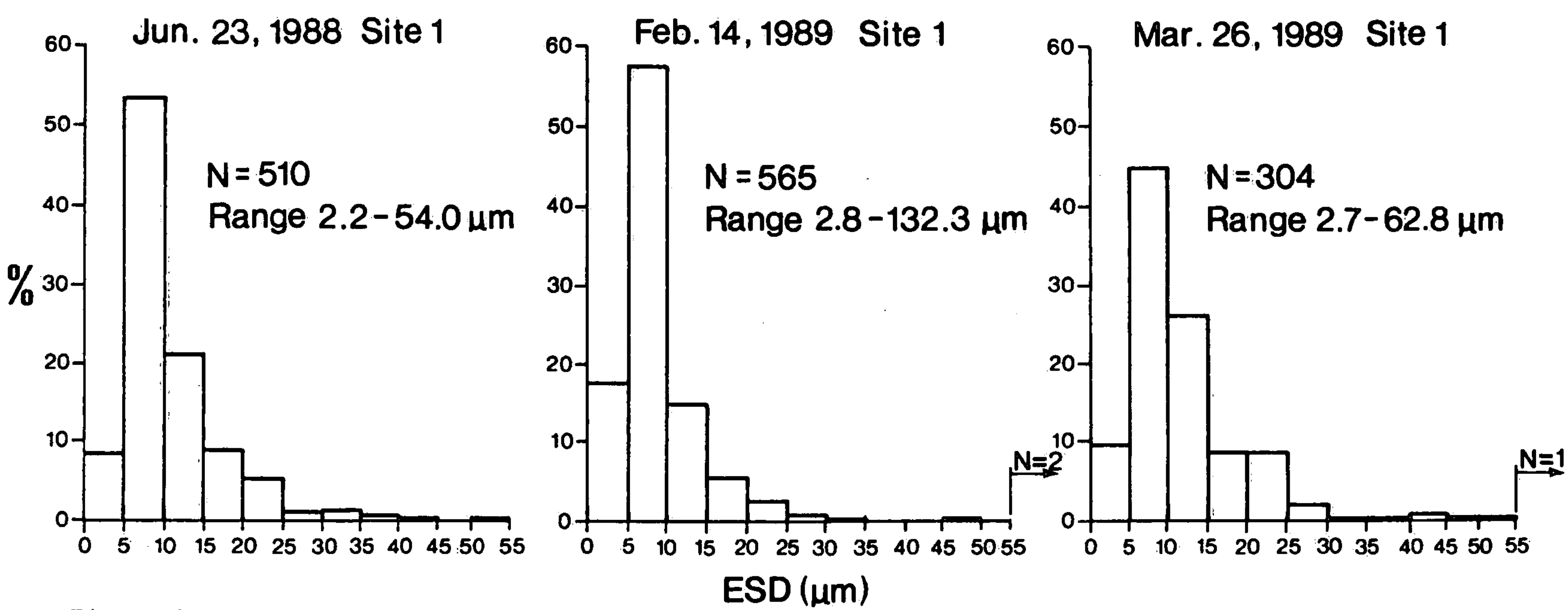

Figure 7 Equivalent spherical diameter distributions of flocculated suspended solids.

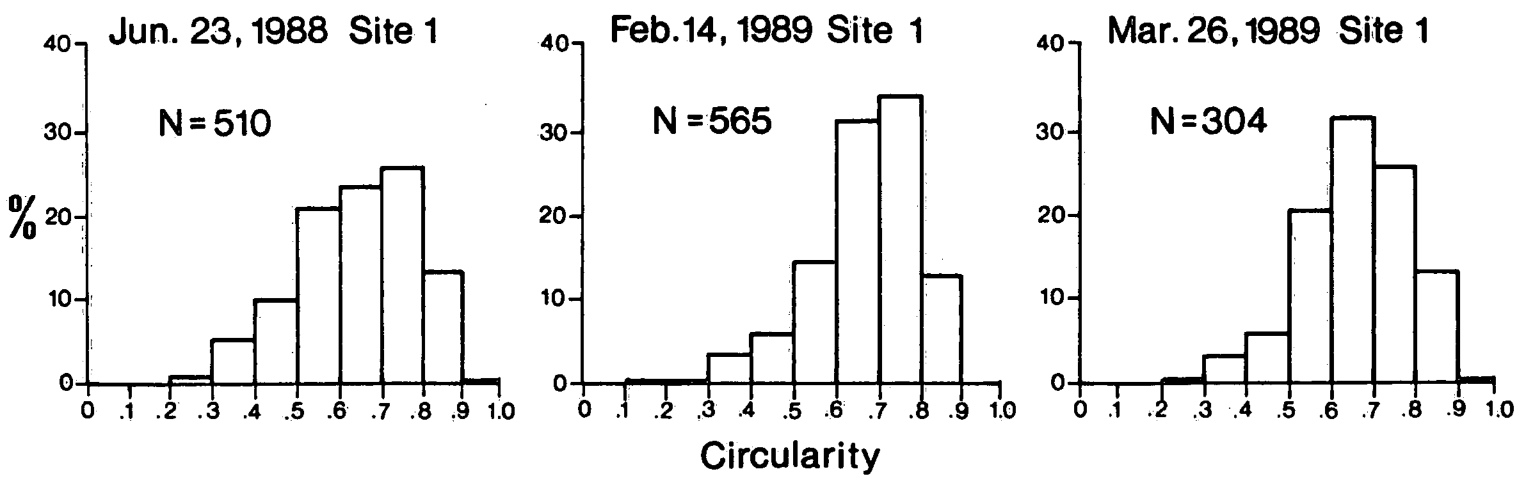

Figure 8 Circularity distributions of flocculated suspended solids.

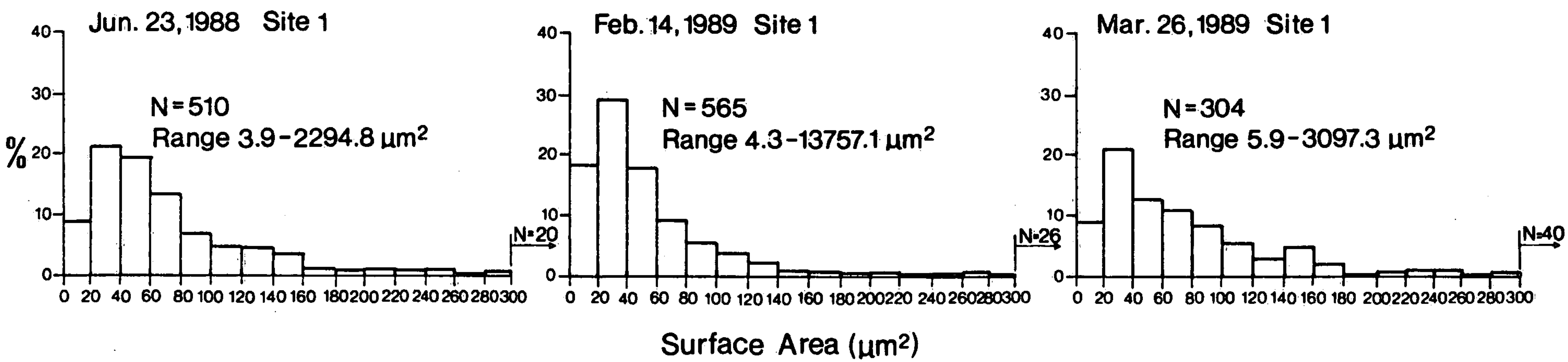

Figure 9 Surface area distributions of flocculated suspended solids.

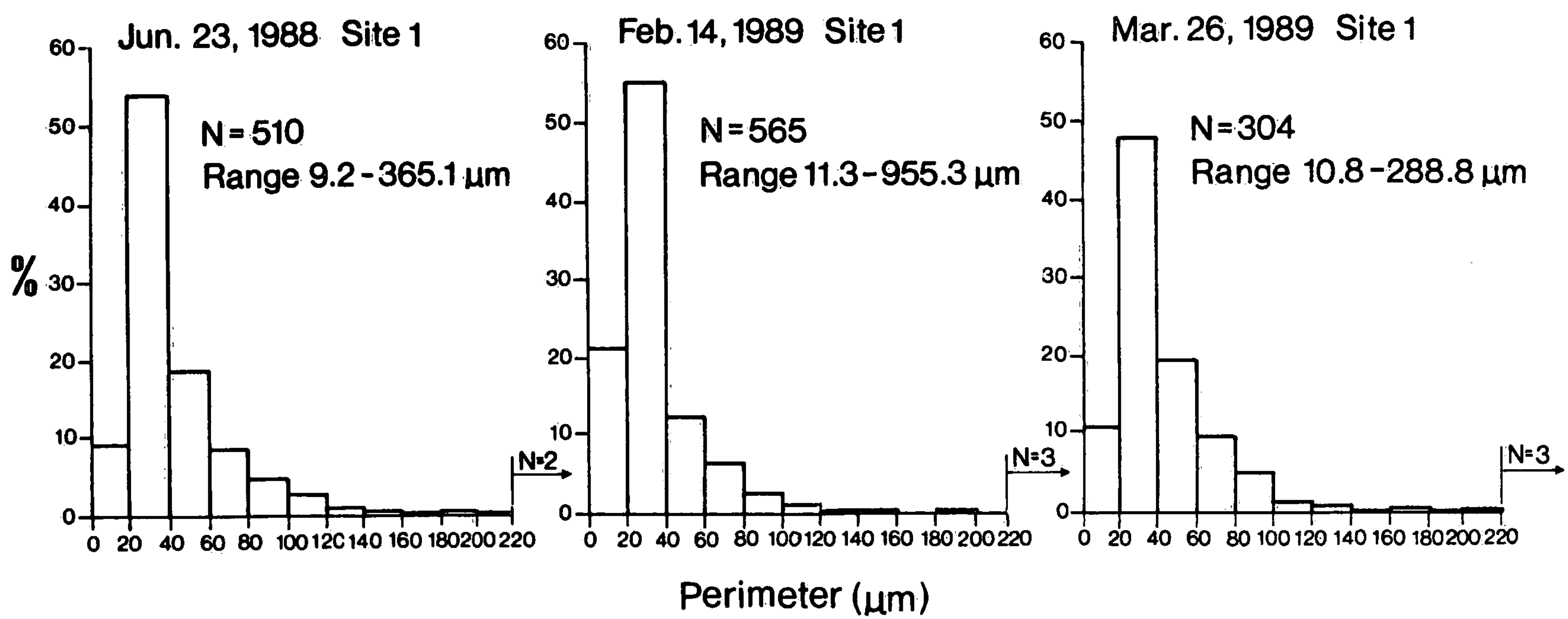

Figure 10 Perimeter distributions of flocculated suspended solids.

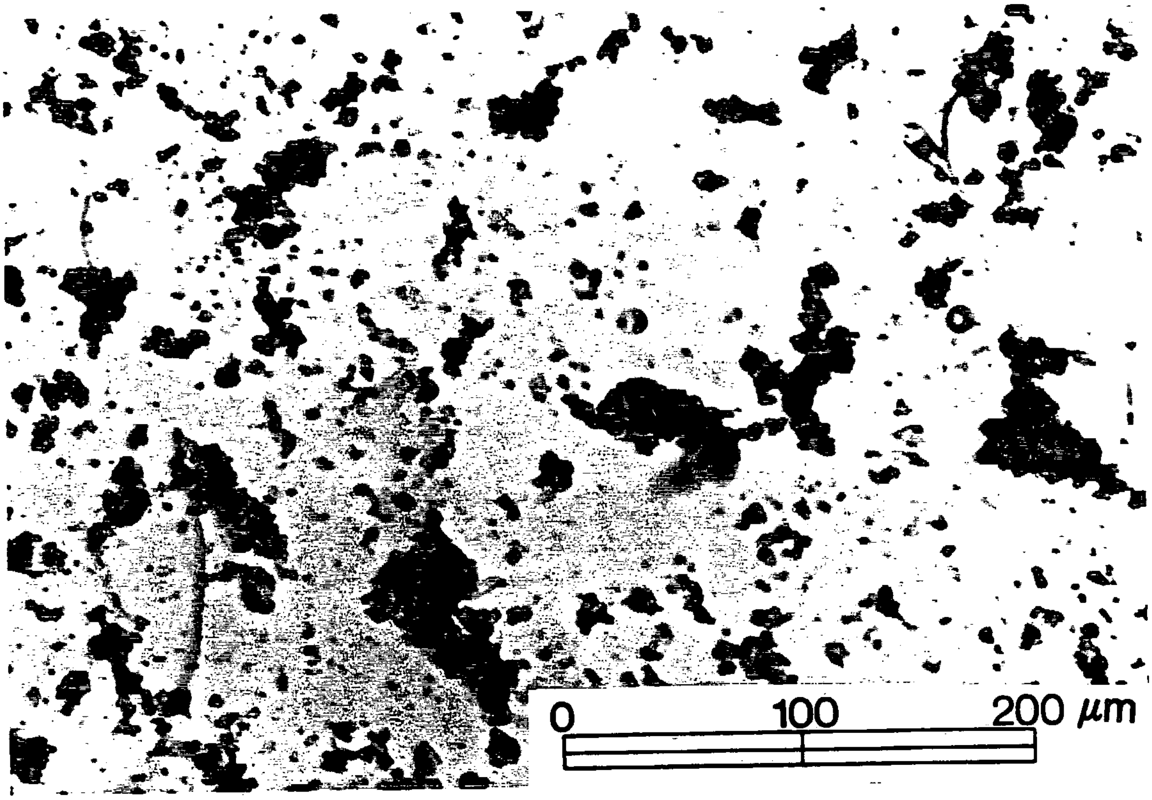

Figure 11 Natural flocculated suspension (Nov. 17, 1987, Site 1).

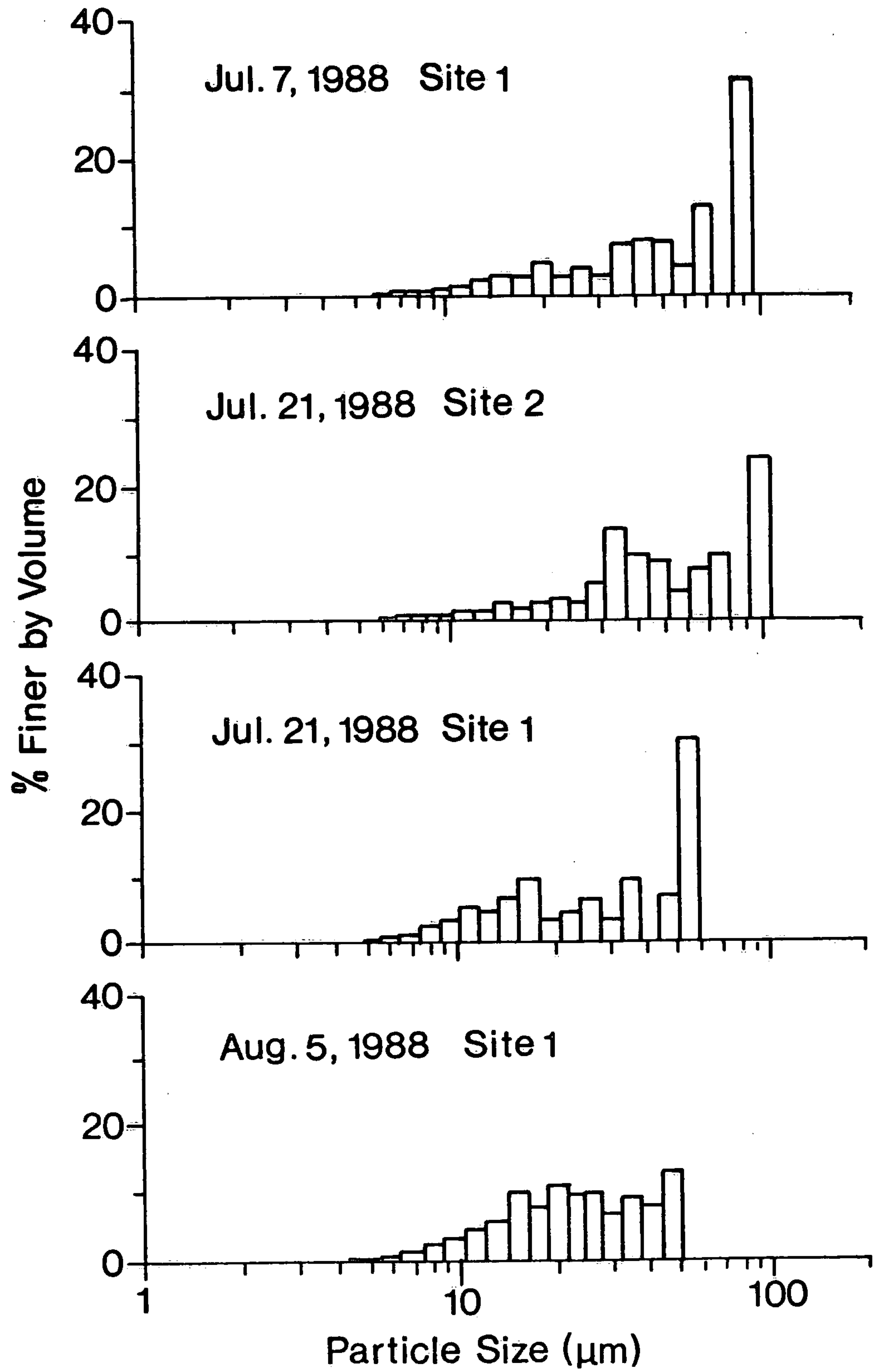

Figure 12 Floc size distributions of four individual samples.

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