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MANAGEMENT PERSPECTIVE

The determination of primary (dispersed) grain-size distribution is frequently required for contaminant transport and sedimentological studies. Sediment programs often require more concentrated samples than those naturally occuring in the field. Here, we examine the utility of concentrating inorganic suspended sediment by filtration and the biases that may occur using this technique. The results indicated that the filtration and resuspension of sediment from two different types of filters has no significant effect on the primary grain-size distribution. Thus filtration may be used effectively to concentrate samples for grain-size distribution analysis with instruments such as the Malvern Particle Size Analyzer. This is an inexpensive and efficient alternative to other concentration techniques such as bench centrifugation or settling.

SOMMAIRE À L'INTENTION DE LA DIRECTION

Il est souvent nécessaire de connaître la distribution granulométrique primaire (dispersée) pour des études sur le transport des contaminants et des études de sédimentologie. Dans le cas des programmes de sédimentologie, il faut souvent des échantillons plus concentrés que ceux que l'on trouve sur le terrain. Ici, nous étudions l'utilité de concentrer des sédiments inorganiques en suspension par filtration et les biais qui peuvent survenir avec cette technique. Les résultats ont révélé que la filtration et la remise en suspension des sédiments effectuées au moyen de deux types différents de filtres n'avaient aucun effet important sur la répartition granulométrique primaire. La filtration peut donc être utilisée efficacement pour concentrer des échantillons aux fins de l'analyse de la distribution granulométrique au moyen d'instruments comme l'analyseur de la granulométrie Malvern. Il s'agit d'une solution peu coûteuse et efficace à d'autres techniques de concentration comme la centrifugation ou la décantation sur table.

ABSTRACT

The influence of concentrating suspended sediments by filtration for analysis with the Malvern Particle Sizer is determined. Low concentration solutions with known primary grain- size distributions (determined by a Malvern Particle Sizer) were filtered onto two types of filters (0.4 µm Nuclepore polycarbonate plastic membrane filter and 0.45 µm Millipore cellulose membrane filter). The sediments were resuspended by a distilled water wash to produce higher concentration solutions, sonicated, and again analyzed by the Malvern Particle Sizer for their primary grain-size distributions. The comparison of the initial and resuspended grain size distributions revealed minimal difference between the two. There was some evidence of filter disintegration and pore clogging, however, their influence on the distributions was minimal. It is expected that the errors induced by pore clogging may increase with samples containing significant amounts of organic matter.

RÉSUMÉ

On détermine l'effet de la concentration des sédiments en suspension par filtration aux fins d'analyse au moyen de l'analyseur de la granulométrie Malvern. Des solutions à faible concentration et à distribution granulométrique primaire connue (établie par l'analyseur Malvern) ont été filtrées au moyen de deux types de filtres (membrane filtrante de polycarbonate Nuclepore de 0,4 um et membrane filtrante de cellulose Millipore de 0,45 um). Les sédiments ont été remis en suspension par lavage à l'eau distillée afin d'obtenir des solutions à concentration plus élevée, traités par les ultrasons et analysés de nouveau au moyen de l'analyseur Malvern afin de déterminer la répartition granulométrique primaire. La comparaison des répartitions granulométriques initiales et aprés remise en suspension a montré une différence minimale entre les deux. Il y avait des signes de désintégration du filtre et de colmatage des pores, cependant, leur influence sur les répartitions était minime. On s'attendait à ce que les erreurs induites par le colmatage des pores puissent augmenter dans les échantillons contenant des quantités importantes de matiére organique.

INTRODUCTION

Suspended solids play an important role in the biological and chemical balance of the aquatic environment. They are also identified as important components for the transport of contaminants in river systems (Allen, 1986 and Ongley et al., 1981). Because many contaminants demonstrate a high affinity for the fine-grain fraction of sediments the primary grain-size distribution is often of interest for sediment and contaminant transport studies. The accurate determination of suspended grain size distributions by traditional methods is often difficult if sediment concentrations are low. The Malvern Particle Sizer (series 2600c), however, allows for the determination of grain-size distributions on a very small amount of sediment. The minimum concentration for optimum operation of the Malvern Partical Sizer depends on the size of particles and on the path length of the laser beam. For an average particle diameter of 10 microns and path length of 10 mm, the minimum volumetric concentration is 3.4 ppm (Weiner, 1984). However, even with this method it is often necessary to concentrate the suspended solids to obtain a large enough concentration for analysis. Evaporation of large volumes is time consuming and may alter the grain-size distribution by precipitation of dissolved solids. Horowitz (1988) discusses four other methods of sediment concentration for direct metal analysis. However, if the end result of analysis is only primary grain-size distribution and not suspended solid's concentration or chemistry, then filtration and resuspension of the sediment from the filters may be suitable for analysis with the Malvern Particle Sizer. It is the objective of this technical note to determine if the process of filtering and resuspending the sediment off of the filters results in a significant change of the primary grain-size distribution due to sediment retention and disintegration of the filters.

MATERIALS AND METHODS

Sample Preparation

The sediment used for the study was a bulk bottom sediment collected from Lake Erie off shore of Port Stanley and Port Burwell. The sediment was fully dispersed, wet sieved through a 62 μm mesh to ensure a size distribution in the silt and clay range and then freeze dried. Organic content was 2.9% as determined by loss on ignition. Rather than produce six seperate solutions with six subsamples of the dry sediment, one solution was produced and six subsamples were drawen off to allow for a more even distribution of particale sizes between subsamples.

One hundred milligrams of the bulk sediment was suspended in one litre of 10% solution of sodium hexametaphosphate in distilled water. The solution was sonicated for 2 minutes to disperse the sediment into its primary particles then placed on a magnetic stirrer to keep the particles in suspension. The solution was divided into six samples by pipetting aliguots of 10 ml successively into six beakers until the original suspension was depleted. The pipette (10 ml) was held at the same depth within the original solution beaker for each round of six withdrawals to account for any possible segregation of size classes within the suspension. Additional distilled water was added to retrieve any particles deposited on the bottom of the original solution beaker and distributed evenly among the six samples. Each sample was approximately 170 ml with a calculated concentration of 16.7 mg/l.

A small proportion of each of the six samples (representing the initial prefiltered solution) was removed by pipet for particle sizing with the Malvern Particle Sizer. The initial sub-sample was then filtered through the respective filter (3 on Millipore and 3 on Nuclepore) and the sediment immediately washed off with a distilled water spray before the filter and sediment could dry. The resuspended solutions were once again sonication and vigorously shaken prior to particle size determination. Through repeated measurements, the precision of the Malvern readings of median grain size was found to be within one micron.

Filters

Two common filter types (Millipore and Nuclepore) were used to determine if there is any difference in their ability to retain sediment or to degrade during the sediment recovery process and thus affect the grain-size distribution. Nuclepore Corporation does not produce a filter with a nominal pore size of 0.45 μm (the conventional boundary between the dissolved and particulate phase) and, therefore, the closest pore size option was used. The Nuclepore filter is a polycarbonate plastic membrane filter with a nominal pore size of 0.4 μm. The filter has cylindrical pores (within $\pm 29°$) normal to the surface and randomly distributed. The pore size deviation is +0 to -20% of the nominal pore size. The majority of sediment is retained on the surface of the filter due to its flat smooth surface (Nuclepore Corporation, 1989). The Millipore filter is a membrane filter composed of cellulose acetate and cellulose nitrate fibres woven into a matrix with a nominal pore size of 0.45 μm. This filter is a weave of fibres with many flow channels and has no specific upper limit on the size of particles which pass through the matrix. Unlike the Nuclepore filter this type of filter retains particles within its matrix of fibres as well as on the surface (Millipore Corporation 1983).

Particle Sizing

Primary particle grain-size distributions were produced with the Malvern Particle Sizer. The Malvern comprises a 3 mV laser, a receiving optics assembly and an electronic circuitry to interface with a microcomputer. Particle size distributions are derived from measurements of the near-forward Fraunhofer diffraction spectrum that is provided by a particle group randomly distributed in a sample cell mounted in the beam path between the laser source and the detector array (Bale and Morris, 1987). A more complete description of the Malvern Particle Sizer can be found in Krishnappan et al. (1990).

Results and Discussion

All initial primary grain-size distributions (by volume) of the six samples are plotted in Figure 1, indicating an envelope of variability for particle size distribution between samples. A one-way analysis of variance (α = 0.01) reveals no significant difference between the means of the distributions of the six samples. Any variations between samples are likely due to random factors, including unequal particle size sampling (splitting) from the original one litre suspension and instrumental factors. Variance in particle frequency was greatest in the larger particle size classes and decreased as particle size decreases. This reflects the smaller number of larger particles in the distribution yielding greater variability in size determinations. Particles in the distribution above 62 μm may reflect flocculation occurring in the suspension prior to analysis, or elongated particles which have passed longitudinally through the sieve but whose long axis was sized by the Malvern yielding a larger particle size.

Distributions of the resuspended suspensions are plotted in Figures 2 and 3 for their respective filter type and compared to their initial sample distributions. Once again visual observations reveal variation in the resuspended distributions to be greatest in the larger size classes and decreases with size.

Figure 2 and 3 illustrate significant overlap in the envelopes of the resuspended and initial distributions. This is also evident from Tables 1 and 2 where the means and standard deviations of the percent volume in each size class for the initial and resuspended sediment are very similar. Visual observation of the Millipore resuspended suspension revealed some fibrous material present from the filter structure suggesting filter disintegration. This may explain the higher percent by volume of particles in the larger size classes as compared to the initial sediment. This problem may be minimized by pre-washing the filters. The presence of particles above the 62 μm (silt) particle size may also be a result of sediment flocculation in suspension or resuspension of coagulated sediment from the filter which was not broken up by sonication. Within the smaller size classes there tends to be slightly fewer particles by volume in the resuspended sediment than in the initial sediment suspensions. This is indicative of some pore clogging and sediment retention by both filters. Visual observations of both filter types revealed discolouration induced by sediment retention. This phenomenon was, however, more pronounced in the Millipore filter due to its weave construction. The difference between the initial and resuspended sediment distributions for both filters is, however, quite minimal even with the difference in nominal pore size. Although sediment retention is occurring, its impact on the grain-size distribution is insignificant as a one way analysis of variance ($\alpha = 0.01$) reveals no significant difference between the means of the initial and resuspended distributions. This may be explained by the fact that there is a larger number of smaller particles than larger particles in the distribution. As it is the small sediment sizes which are most likely to be trapped by the pores of the filters, the impact of their loss to the distribution appears to be unimportant. The filtration of the sediment suspensions onto filters and their subsequent resuspension for sediment concentration, therefore, appears to have little effect on changing the initial primary grain-size distribution.

The use of this method for environmental samples that contain large amounts of organic matter may be inappropriate. There may be a higher probability of pore clogging and sample retention due to the more cohesive nature of organic material.

For operational purposes, sample dispersion is not necessary or desirable for the initial sample as the fewer the number of small particles in the initial suspension, the lower will be the sediment retention of the filters due to fewer particles being imbedded into the pores or matrix of the filters. High vacuum pressures (>5 psi) may bind particles to the filters as well as suck particles through the filter.

CONCLUSIONS

From the above experiments we conclude that primary grain-size distributions of inorganic sediments filtered on and resuspended from both Millipore (0.45 μm) and Nuclepore (0.4 μm) filters are not significantly different from the initial primary grain-size distribution. While some filter disintegration and pore clogging occurred, their influence on the primary grain-size distribution was minimal. However, an increase in grain-size distribution errors may result for environmental samples that contain a high proportion of organic matter.

ACKNOWLEDGEMENT

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Table 1. Initial Sample vs. Resuspended Sample Distribution Variations for Millipore Filters.

Size Class	Initial Samples N = 3		Resuspended Samples N = 3	
	Mean % in Class	Standard Deviation of % in Class	Mean % in Class	Standard Deviation of in Class
118.4 - 54.9	12.3	2.6	16.5	3.1
54.4 - 33.7	23.3	2.7	26.3	1.1
33.7 - 23.7	10.2	0.7	10.4	1.1
23.7 - 17.7	9.9	0.5	8.8	0.1
17.7 - 13.6	6.5	0.6	5.9	0.7
13.6 - 10.5	4.9	0.8	4.4	0.3
10.5 - 8.2	5.4	0.6	4.3	0.6
8.2 - 6.4	5.0	0.7	4.1	0.5
6.4 - 5.0	4.3	0.7	3.9	0.6
5.0 - 3.9	4.6	0.7	4.0	0.7
3.9 - 3.0	4.4	0.8	3.7	0.7
3.0 - 2.4	3.1	0.6	2.8	0.6
2.4 - 1.9	1.9	0.4	1.6	0.4
1.9 - 1.5	1.1	0.2	0.8	0.2
1.5 - 1.2	0.8	0.1	0.6	0.2

Table 2. Initial Sample vs. Resuspended Sample Distribution Variations for Nuclepore Filters.

Size Class	Initial Samples N = 3		Resuspended Samples N = 3	
	Mean % in Class	Standard Deviation of % in Class	Mean % in Class	Standard Deviation of in Class
118.4 - 54.9	18.6	2.8	21.1	1.9
54.9 - 33.7	30.6	2.7	31.4	0.4
33.7 - 23.7	10.2	1.6	10.2	0.7
23.7 - 17.7	9.0	0.5	8.7	0.3
17.7 - 13.6	5.4	0.8	4.9	0.7
13.6 - 10.5	3.7	0.8	3.5	0.6
10.5 - 8.2	3.7	0.5	3.5	0.5
8.2 - 6.4	3.2	0.6	3.0	0.4
6.4 - 5.0	2.9	0.5	2.8	0.2
5.0 - 3.9	3.1	0.4	2.9	0.2
3.9 - 3.0	3.1	0.4	2.6	0.1
3.0 - 2.4	2.1	0.3	1.9	0.2
2.4 - 1.9	1.2	0.2	1.1	0.2
1.9 - 1.5	0.8	0.1	0.7	0.2
1.5 - 1.2	0.8	0.1	0.6	0.2

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FIGURES

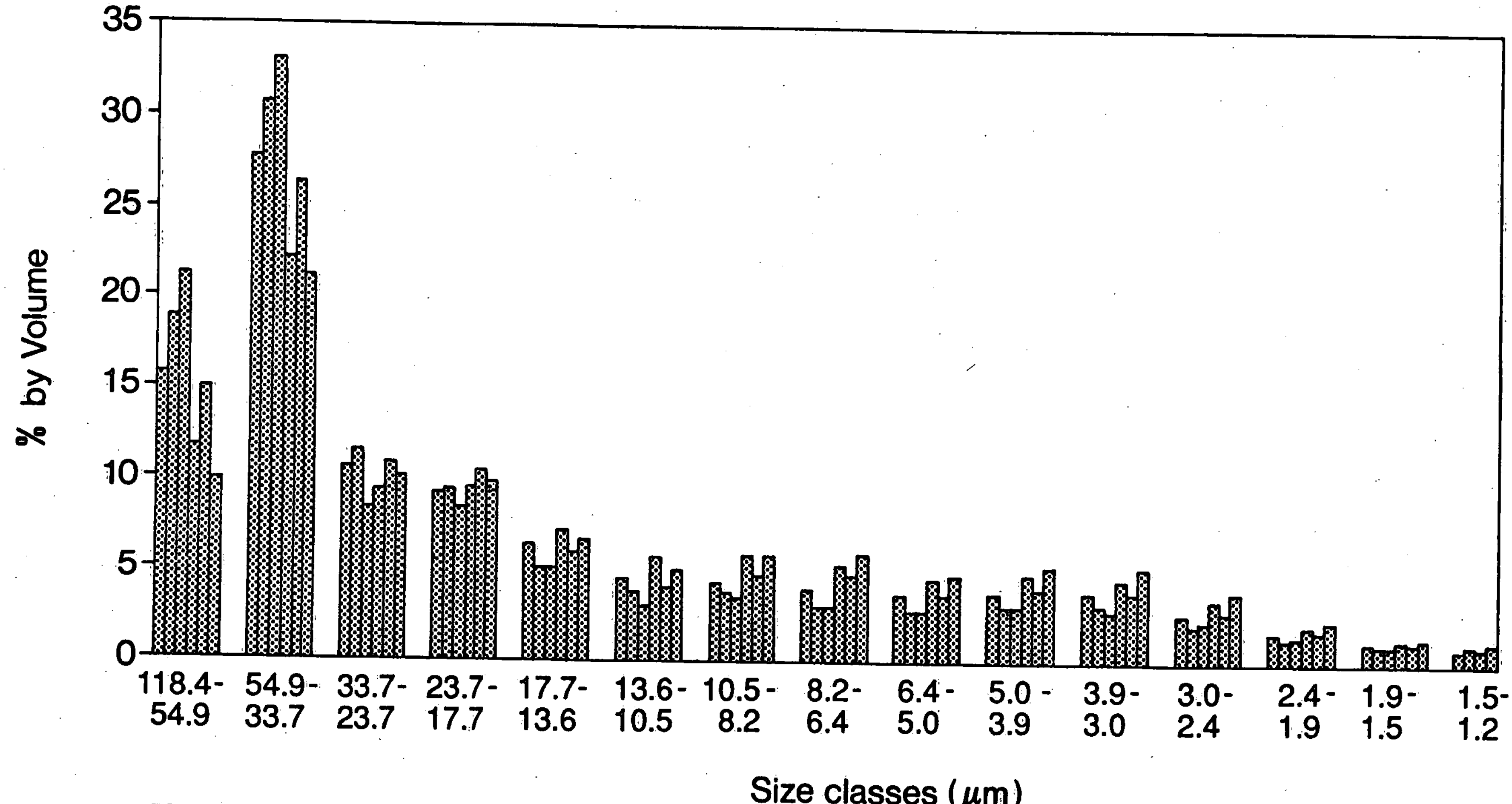
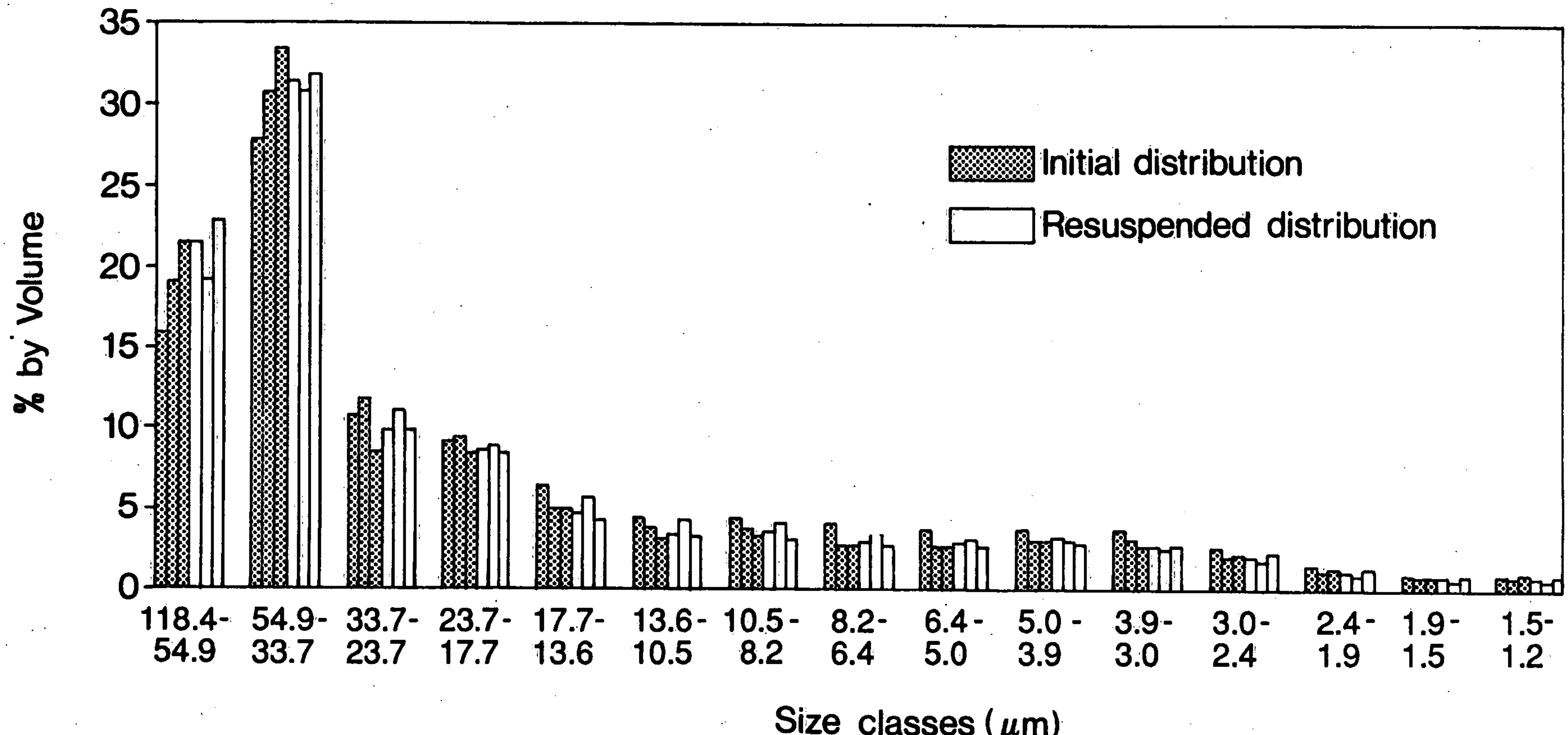

% by Volume
35
30
25
20
15
10
5
0
118.4- 54.9
54.9- 33.7
33.7- 23.7
23.7- 17.7
17.7- 13.6
13.6- 10.5
10.5- 8.2
8.2- 6.4
6.4- 5.0
5.0 - 3.9
3.9- 3.0
3.0- 2.4
2.4- 1.9
1.9- 1.5
1.5- 1.2
Size classes (μm)

FIGURE 1



35
30
25
20
15
10
5
0
% by Volume
Initial distribution
Resuspended distribution
118.4- 54.9
54.9- 33.7
33.7- 23.7
23.7- 17.7
17.7- 13.6
13.6- 10.5
10.5- 8.2
8.2- 6.4
6.4- 5.0
5.0 - 3.9
3.9- 3.0
3.0- 2.4
2.4- 1.9
1.9- 1.5
1.5- 1.2
Size classes (μm)

FIGURE 2

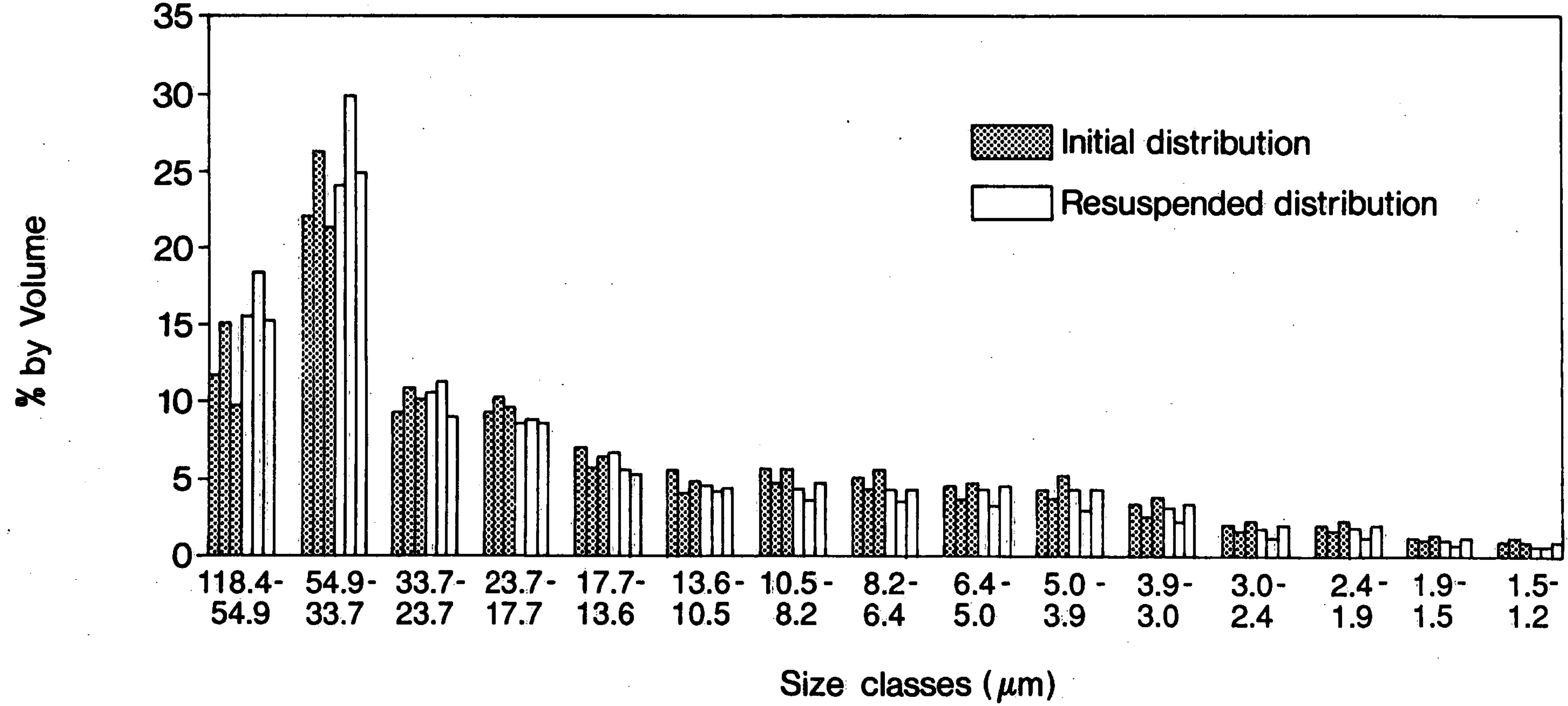

% by Volume
35
30
25
20
15
10
5
0
Initial distribution
Resuspended distribution
118.4- 54.9
54.9- 33.7
33.7- 23.7
23.7- 17.7
17.7- 13.6
13.6- 10.5
10.5 - 8.2
8.2- 6.4
6.4 - 5.0
5.0 - 3.9
3.9- 3.0
3.0- 2.4
2.4 - 1.9
1.9- 1.5
1.5- 1.2
Size classes (μm)

FIGURE 3

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