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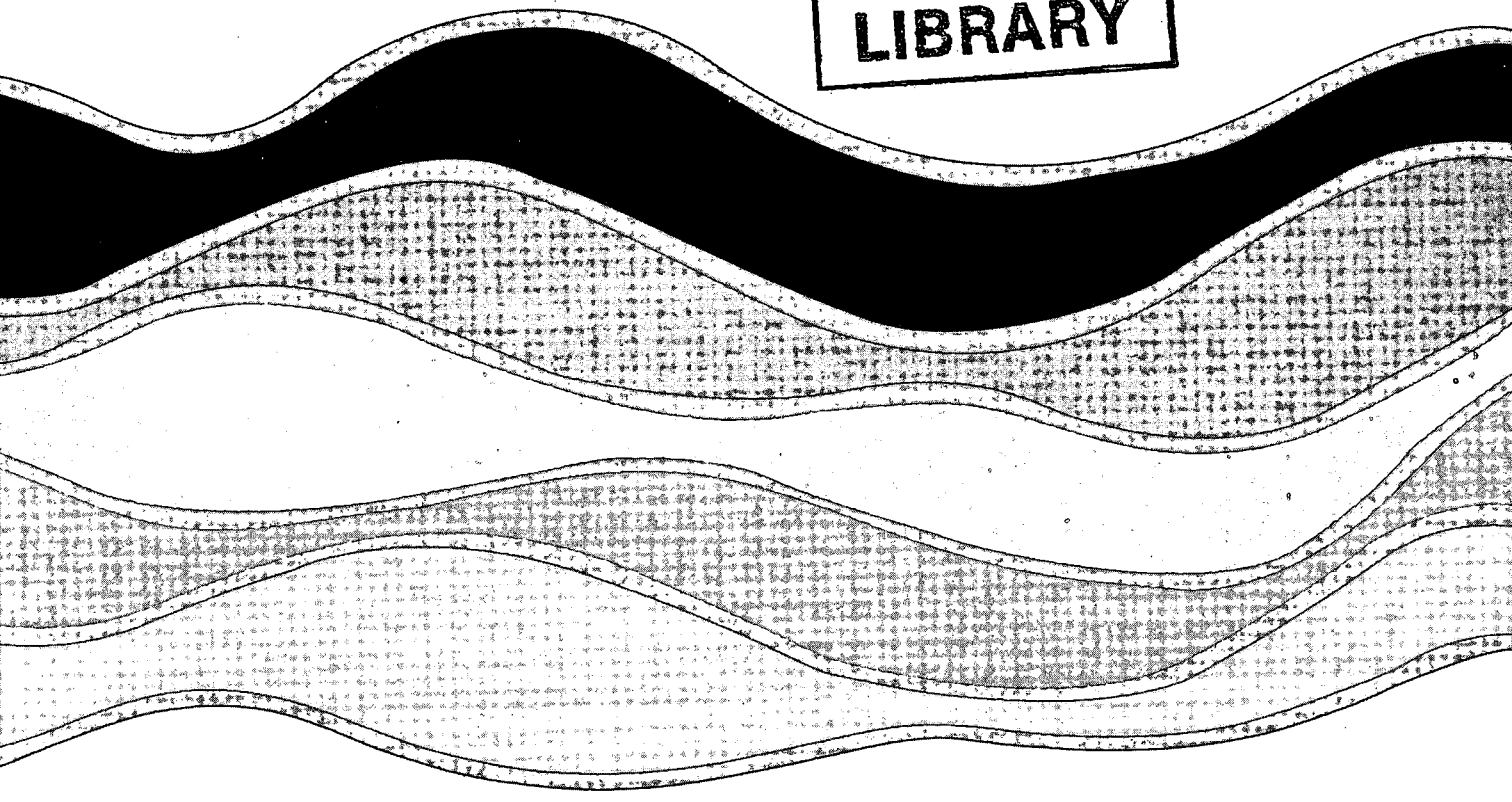

TESTING OF A FILTER FRACTIONATION TECHNIQUE USING MALVERN PARTICLE SIZE ANALYZER

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MANAGEMENT PERSPECTIVE

The fractionation of a sediment sample into a number of size classes is frequently needed to study the effect of sediment size on a number of sediment-contaminant interaction processes. In this paper, a laser particle size analyzer was used to test the effectiveness of the filtration technique that is often used for fractionation. The results indicate that the filteration technique is not adequate even for a fully dispersed inorganic sediment. The fractionation of environmental samples that are likely to flocculate need innovative approaches.

PERSPECTIVE GESTION

Bien souvent, il faut fractionner un échantillon de sédiment en un certain nombre de fractions granulométriques, pour étudier les effets de la taille des particules de sédiment sur certaines interactions sédiment-contaminant. Dans cette communication, on décrit l'utilisation d'un analyseur granulométrique à laser pour évaluer l'efficacité d'une technique de filtration fréquemment utilisée pour le fractionnement. Selon les résultats obtenus, cette technique de filtration n'est pas appropriée, même avec un échantillon de sédiment totalement dispersé. Il faudra mettre au point de nouvelles techniques pour fractionner les échantillons environnementaux susceptibles de floculer.

ABSTRACT

In this paper, a methodology to test the effectiveness of a filtration technique to separate a fully dispersed inorganic sediment sample into a number of size classes is described. The filtration technique involves passing of sediment sample through a series of filters arranged in a descending order of filter pore sizes. The testing was carried out by measuring the size distributions of sediments retained on the filters using a laser particle size analyzer manufactured by Malvern Instruments Ltd. The results of the test show that fractionation using filtration is not perfect even for a dispersed inorganic sediment sample. The filters retained a spectrum of sediment sizes greater and smaller than the nominal pore sizes. The filter clogging and the variability of filter pore sizes are the main reasons for such a result. The fractionation of environmental samples that are likely to flocculate is even more difficult using the filtration technique.

RÉSUMÉ

Dans cette communication, on décrit une méthode permettant de vérifier l'efficacité d'une technique de séparation d'un échantillon de sédiment inorganique totalement dispersé, en un certain nombre de fractions granulométriques. On passe l'échantillon de sédiment dans une série de filtres disposés en ordre de porosité décroissante. Au cours de l'essai, on mesure la distribution granulométrique des sédiments retenus sur les filtres, à l'aide d'un analyseur granulométrique à laser fabriqué par la Malvern Instruments Ltd. Les résultats de l'essai indiquent que la filtration ne permet pas de fractionner parfaitement l'échantillon, même s'il s'agit d'un échantillon de sédiment inorganique dispersé. Les filtres retiennent une gamme de particules de sédiment plus grosses et plus petites que leur porosité. Cette fourchette s'explique surtout par l'encrassement des filtres et par leur porosité variable. Il est encore plus difficile de fractionner, par cette technique de filtration, les échantillons environnementaux susceptibles de floculer.

INTRODUCTION

Investigations into the effect of particle size on the adsorption of contaminants and association of microbial organic matter with fluvial sediment and the concomitant toxicity require that the sediment be separated into a number of size fractions. Such separation or fractionation can be effected in a number of ways. Some of the common fractionation methods include centrifugation (Lammers, 1968), cyclosizer (Kelsall and McAdam, 1963), air elutriation (Horowitz, 1986) and cascade filtration (Munawar et al., 1983, Rao et al., 1988). Each of these methods has certain limitations and drawbacks. In this paper, we test the effectiveness of the cascade filtration method for fractionation of inorganic sediments consisting of silt and clay size particles using a Laser Particle Size Analyzer.

Fractionation using the cascade filtration technique consists of passing sediment water mixture through a series of filters arranged in a descending order of filter pore sizes. The system used in this study consisted of five filters of nominal pore sizes 60, 40, 20, 10 and 8 microns. The first four filters were Nitex sheet filters and the last one was a Nuclepore polycarbonate filter. Filtration through the first four filters was gravity fed whereas the last filter was under a vacuum of 5 psi. The testing of the cascade filtration technique was carried out by measuring the size distribution of sediment fractions (primary particles) retained on individual filters of the system using a Laser Particle Size Analyzer manufactured by Malvern Instruments Ltd. The particles are reasonably spherical (see Fig. 1) and hence allow for a comparison between the nominal size of the filter pores and the equivalent spherical diameter of the particles as measured by the Malvern Particle size analyser.

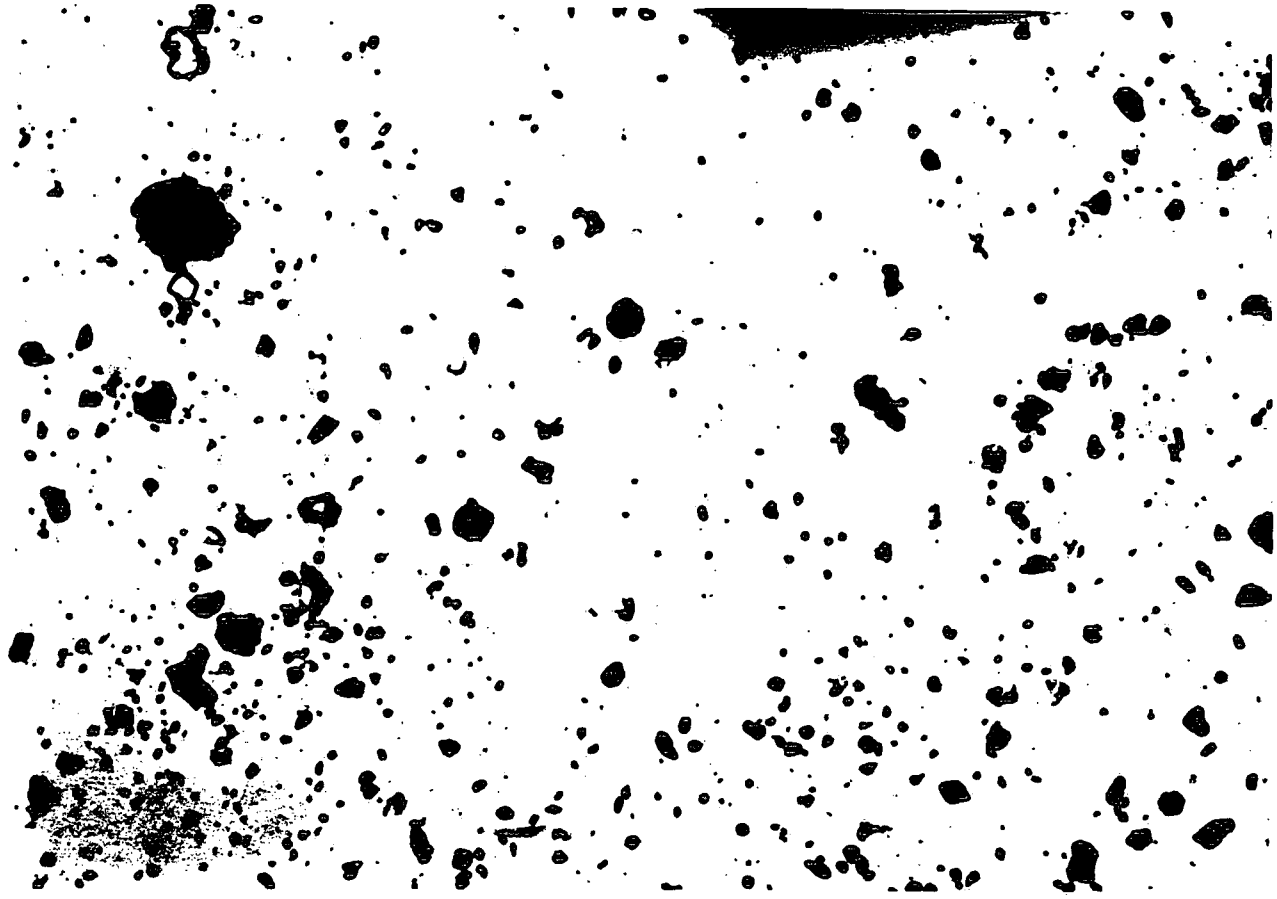

Fig. 1 Photograph of sediment sample
as seen through a microscope

SAMPLE PREPARATION

The sediment used for the study was a fully dispersed inorganic bottom sediment collected from the Great Lakes. The organic content was 2.9% as measured by the process of ignition of 3 g of sediment at 500°C for 3 hours. The sediment was wet sieved through a 62 μm mesh to ensure a size distribution in the silt and clay range and freeze dried.

Three quarters of one gram of the bulk sediment was suspended in 300 mL of 10% solution of sodium hexametaphosphate in distilled water. The suspension was then sonicated for 2 minutes and placed on a magnetic stirrer to keep the particles in suspension. The solution was subsampled by pipetting 100 mL of solution into each of three consecutive flasks until the original suspension was depleted. The pipette (10 mL) was held at the same depth within the original solution flask for each round of three 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 flask and distributed evenly among the subsamples. The three subsamples were then made up to 200 mL with 10% sodium hexametaphosphate resulting in an approximate concentration of 1250 ppm. These three subsamples were then run through the cascade apparatus.

CASCADE FILTRATION PROCEDURE

Prior to filtration, the subsamples were sonicated again for 2 minutes to break up any aggregates which may have formed through

flocculation during settling. Subsamples were passed through each successive filter from largest to smallest pore size. The particles collected on each cascade filter were removed for subsequent size and weight analysis by rinsing the filters with distilled water into clean flasks. Filtrate from the 8 µm filtration was also retained for size analysis and to determine the fractional sediment weight for a mass balance calculation.

Each sediment fraction washed from the individual cascade filter (>60µm, 60-41 µm, 41-20 µm, 20-10 µm, 10-8 µm and <8µm) was subjected to size analysis by a Malvern Particle Size Analyzer (2600 series). The fractions were then filtered on tared 0.45 µm Millipore filters and dried at 104°C. The filters were then reweighed to determine the fractional sediment weights. It was observed that out of the original 0.75 g of sediment, 18% was not accounted for during the filtrations. The sediment which was lost was presumably retained within the pores of the cascade filters and on the sides of the flasks, filter cups and Malvern sample cell.

DESCRIPTION OF MALVERN PARTICLE SIZE ANALYSER

The Malvern Particle Size Analyzer (series 2600c) used in his study consists of a 2 mW He-Ne laser as a light source and a receiver unit mounted on an optical bench (Figure 2). The laser beam is expanded to 9 mm using a beam expander and is collimated. The receiver unit consists of a Fourier transform range lens and a detector plate mounted at the focal plane of the lens. The detector consists of a series of photodiodes mounted in semi-circular arcs at different radii and measures the near-forward Fraunhofer diffraction

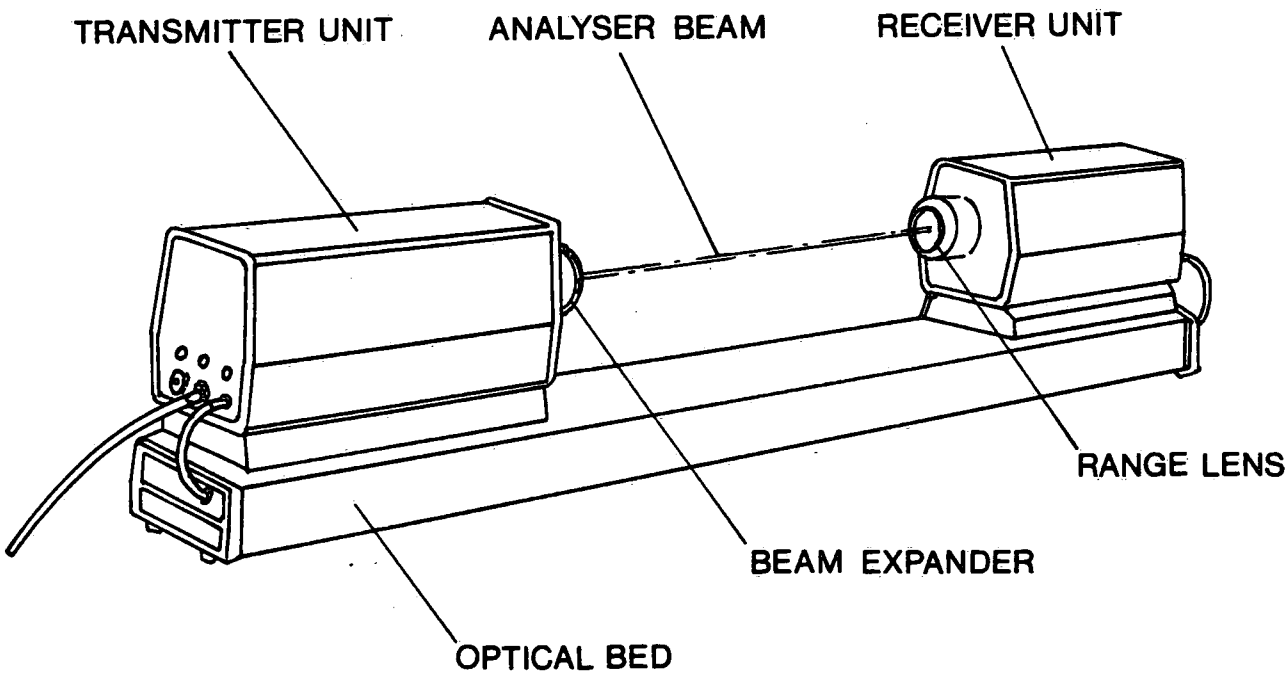

Fig. 2 Malvern particle size Analyser: 2600 series

spectrum produced by randomly distributed particles in a sample cell placed in the path of the laser beam between the laser unit and the range lens. The receiver unit also contains an electronic circuitry which interfaces with a micro computer (not shown in Figure 2) to facilitate the computation of particle size distribution based on the Fraunhofer diffraction theory from the measured light energy distribution.

The Malvern Particle Size Analyzer uses three different range lenses. Each lens covers a specified particle size range. For example, the 63 mm focal length range lens covers a particle size range of 1.2 to 118 μm. The particle size ranges for the 100 mm and 300 mm focal length lenses are 1.9 to 188 μm and 5.8 to 564 μm respectively. Each size range is divided into fifteen size bands, which are listed in Table 1 for the three different lenses. The selection of a range lens depends on the size range of the sediment to be measured.

Two types of sample presentation systems are available for the 2600 series: a recirculating cell and a small volume cell. The small volume cell was used in the present experiment. This cell has glass windows on either side and can be mounted in the path of the laser beam using a mounting block on the optical bench. The cell contains a magnetic stirrer to keep the particles in suspension during measurement.

Each measurement involves taking light energy distribution readings without and with particles in the sample cell and the difference in the light energy distribution is used to calculate the particle size distributions. The instrument has a very fast response.

Table 1: Particle size ranges in micron in each of the size bands and three different focal length range lenses of Malvern Particle Size Analyzer

	63 mm Focal Length		100 mm Focal Length		300 mm Focal Length	
Band No.	Upper	Lower	Upper	Lower	Upper	Lower
1	118.4	54.9	188.0	87.2	564.0	261.6
2	54.9	33.7	87.2	53.5	261.6	160.4
3	33.7	23.7	53.5	37.6	160.4	112.8
4	23.7	17.7	37.6	28.1	112.8	84.3
5	17.7	13.6	28.1	21.5	84.3	64.6
6	13.6	10.5	21.5	16.7	64.6	50.2
7	10.5	8.2	16.7	13.0	50.2	39.0
8	8.2	6.4	13.0	10.1	39.0	30.3
9	6.4	5.0	10.1	7.9	30.3	23.7
10	5.0	3.9	7.9	6.2	23.7	18.5
11	3.9	3.0	6.2	4.8	18.5	14.5
12	3.0	2.4	4.8	3.8	14.5	11.4
13	2.4	1.9	3.8	3.0	11.4	9.1
14	1.9	1.5	3.0	2.4	9.1	7.2
15	1.5	1.2	2.4	1.9	7.2	5.8

It takes only 35 milliseconds to sweep all photodiode rings and to obtain a set of light energy distribution. A large number of sweeps, usually about 500, is used and the readings averaged to cover a representative sample of randomly oriented particles from all size classes in one measurement. Even with this number of sweeps, a measurement of particle size distribution for a sample can be made within a minute.

Another important consideration in the operation of the Malvern Particle Size Analyzer is the concentration of particles in the sample cell. High concentration of the sample will give rise to multiple diffraction which is not accounted for in the theory. It is, therefore, important to use dilute suspensions. The instrument measures the concentration of the particles by measuring the attenuation of the light due to the presence of particles in the sample volume and warns the user if the concentration has exceeded the optimum value derived from theory and experiment.

The Malvern Particle Size Analyzers have been used by a number of investigators such as Tsai et al. (1987), McCave et al. (1986), Bale et al. (1984), Mohamed et al. (1981) and Weiner (1984). The instrument was also tested against other instruments such as settling tubes, sedigraphs, hydrophotometers and Coulter Counter (Singer et al. 1988).

RESULTS AND DISCUSSION

The sediment fractions were introduced into the sample cell of the Malvern Particle Size Analyzer and the size distributions of these fractions were measured. The results are given in Figures 3 to 8.

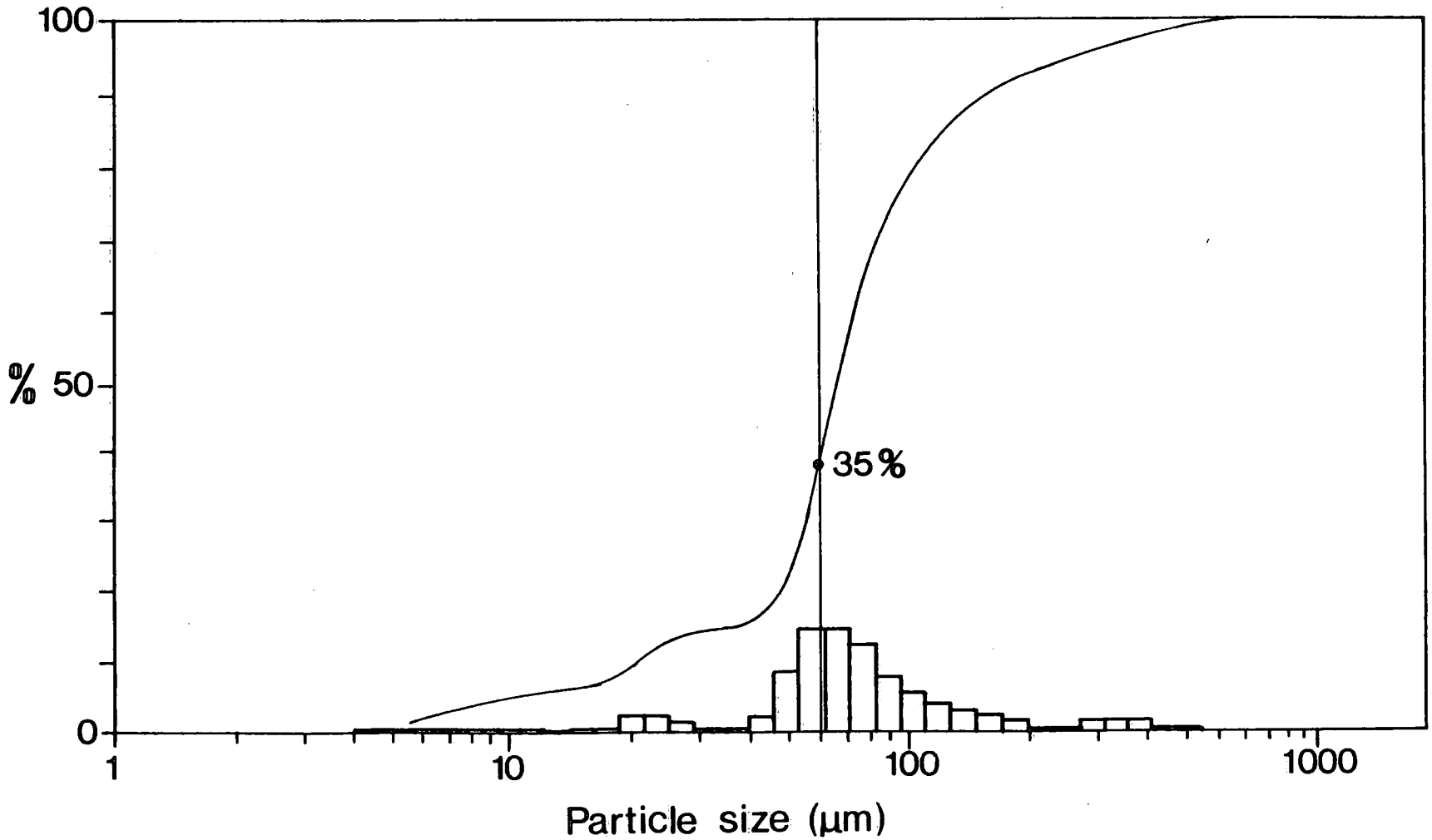

Fig. 3 Size distribution of sediment fraction retained on 60 micron filter.

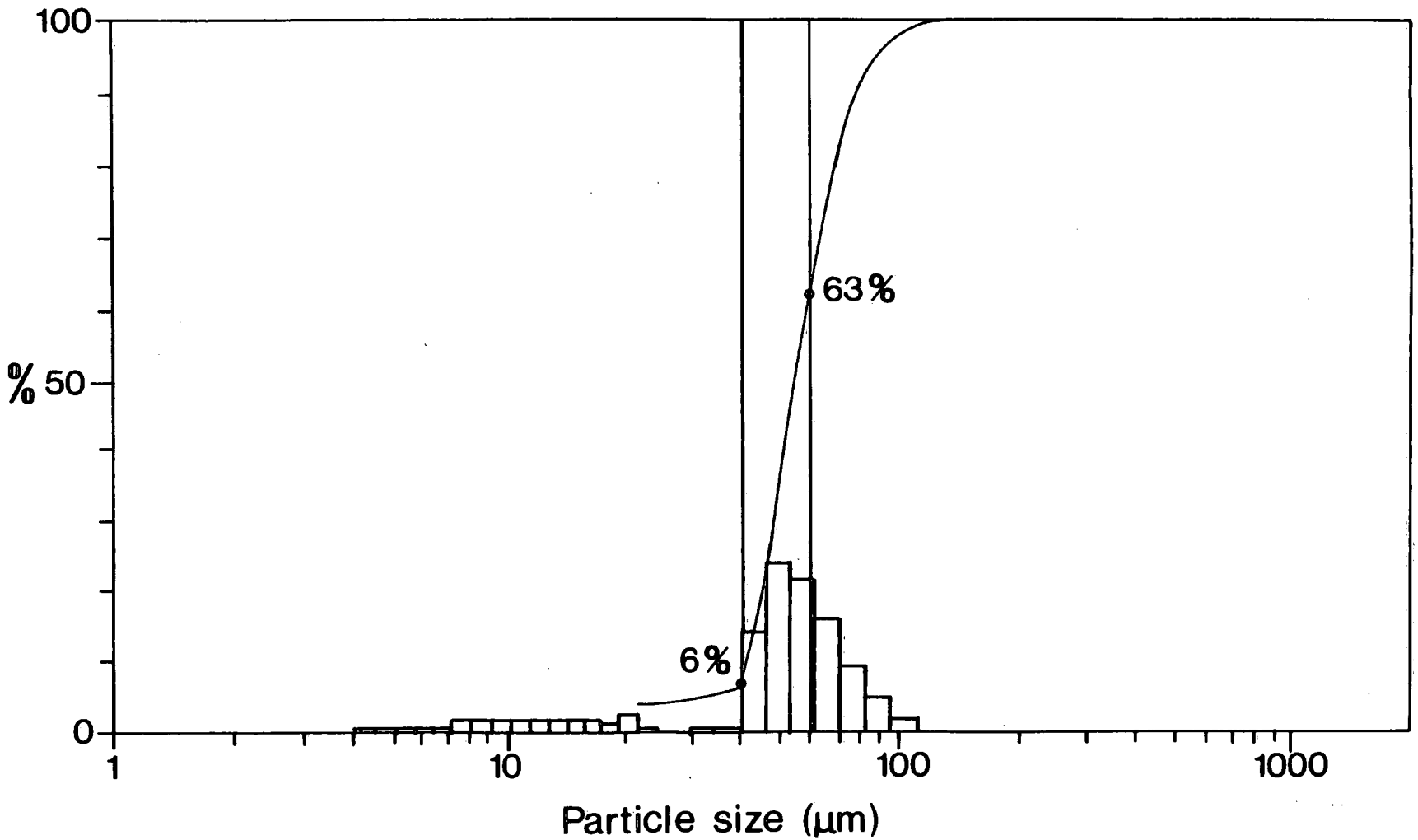

Fig.4 Size distribution of sediment fraction passing through 60 micron filter and retained on 40 micron filter.

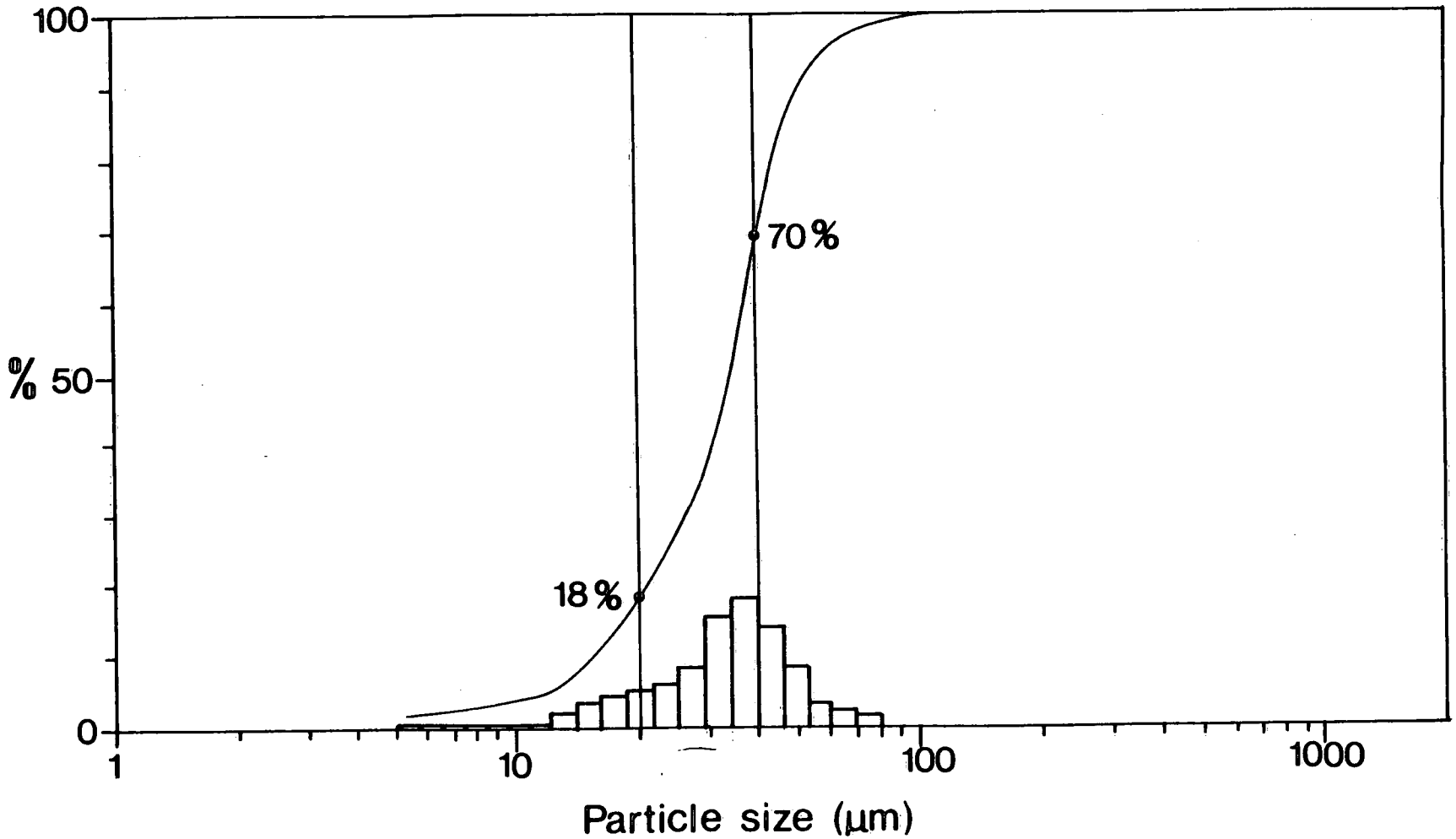

Fig. 5 Size distribution of sediment fraction passing through 40 micron filter and retained on 20 micron filter.

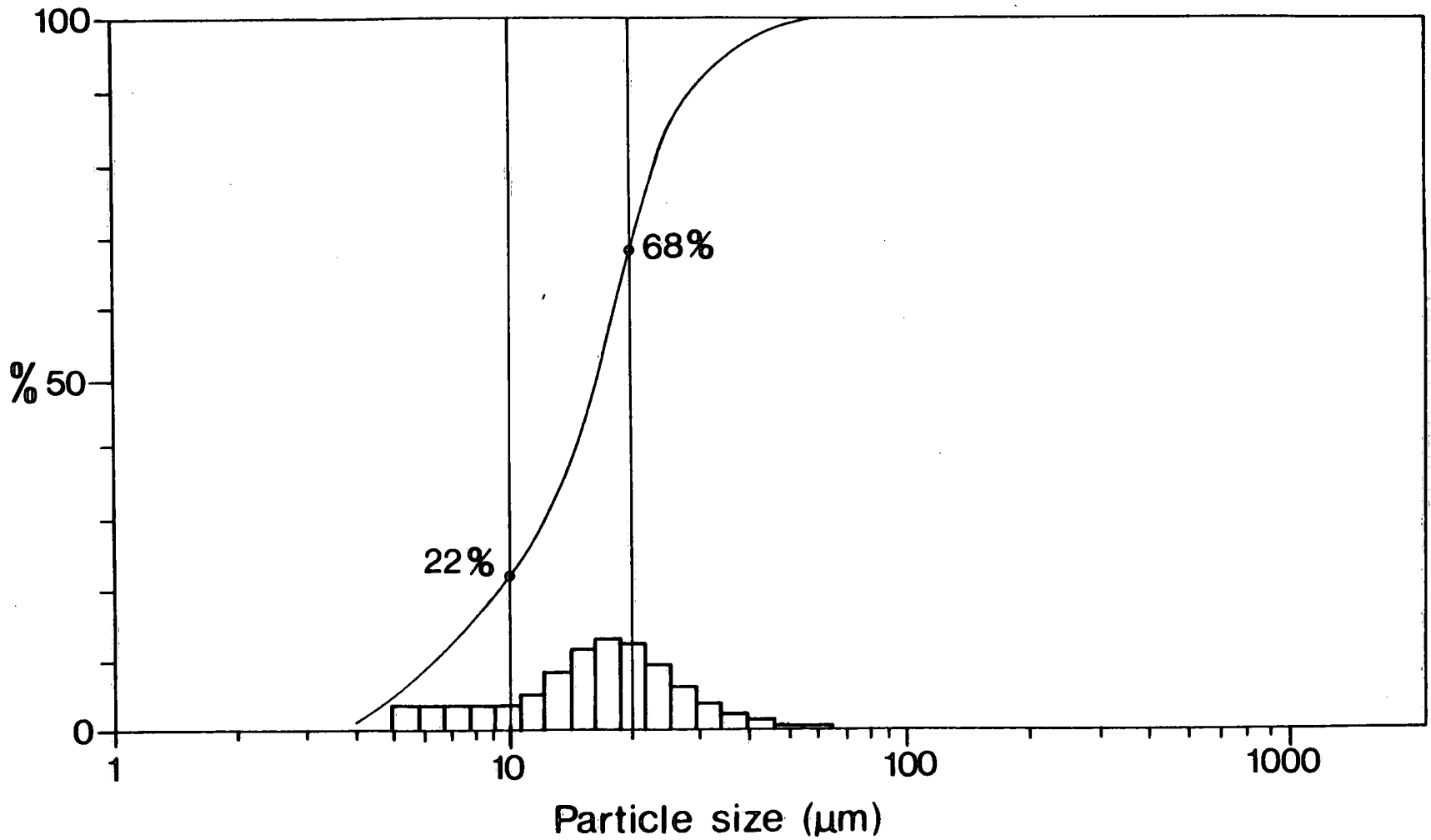

Fig.6 Size distribution of sediment fraction passing through 20 micron filter and retained on 10 micron filter.

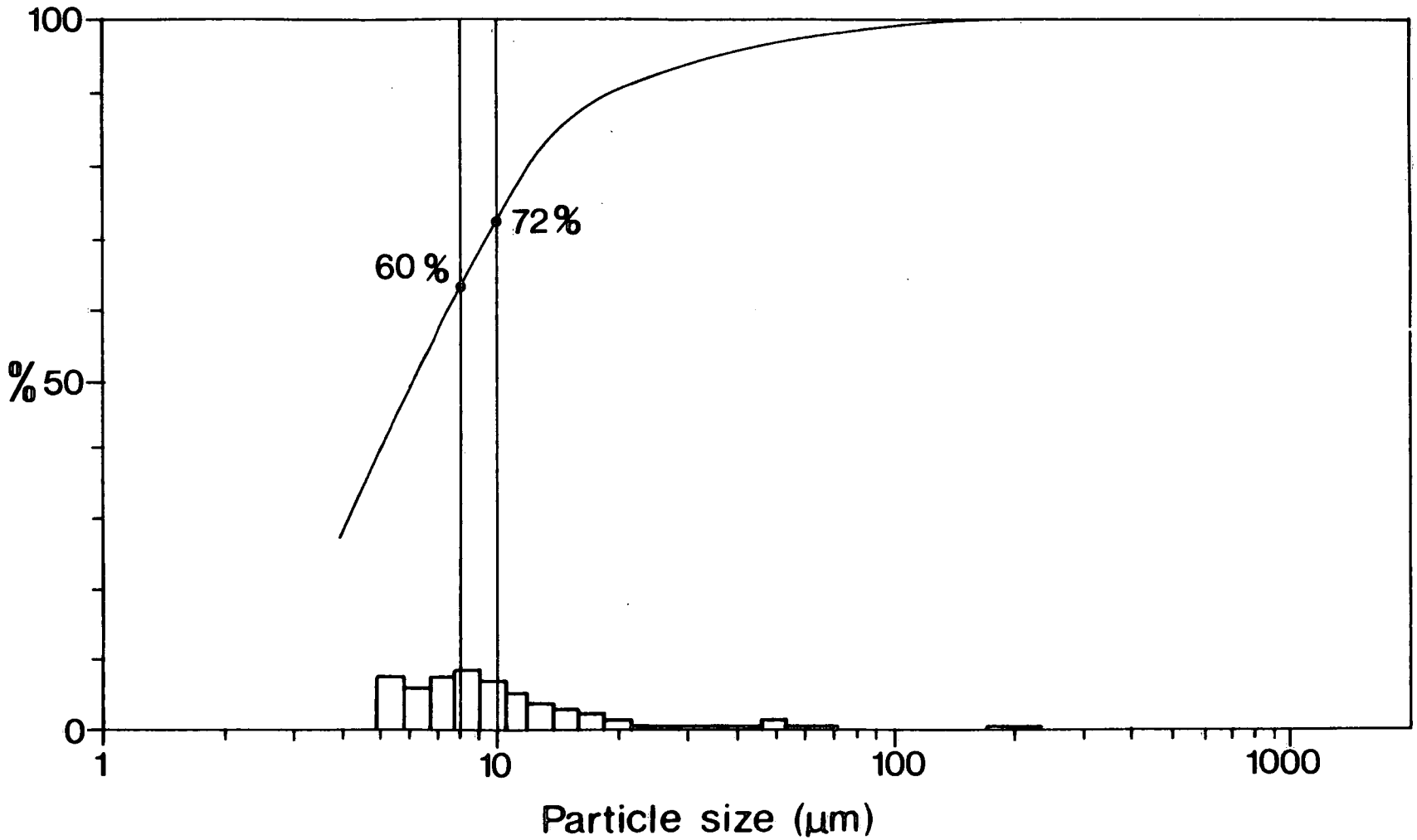

Fig. 7 Size distribution of sediment fraction passing through 10 micron filter and retained on 8 micron filter.

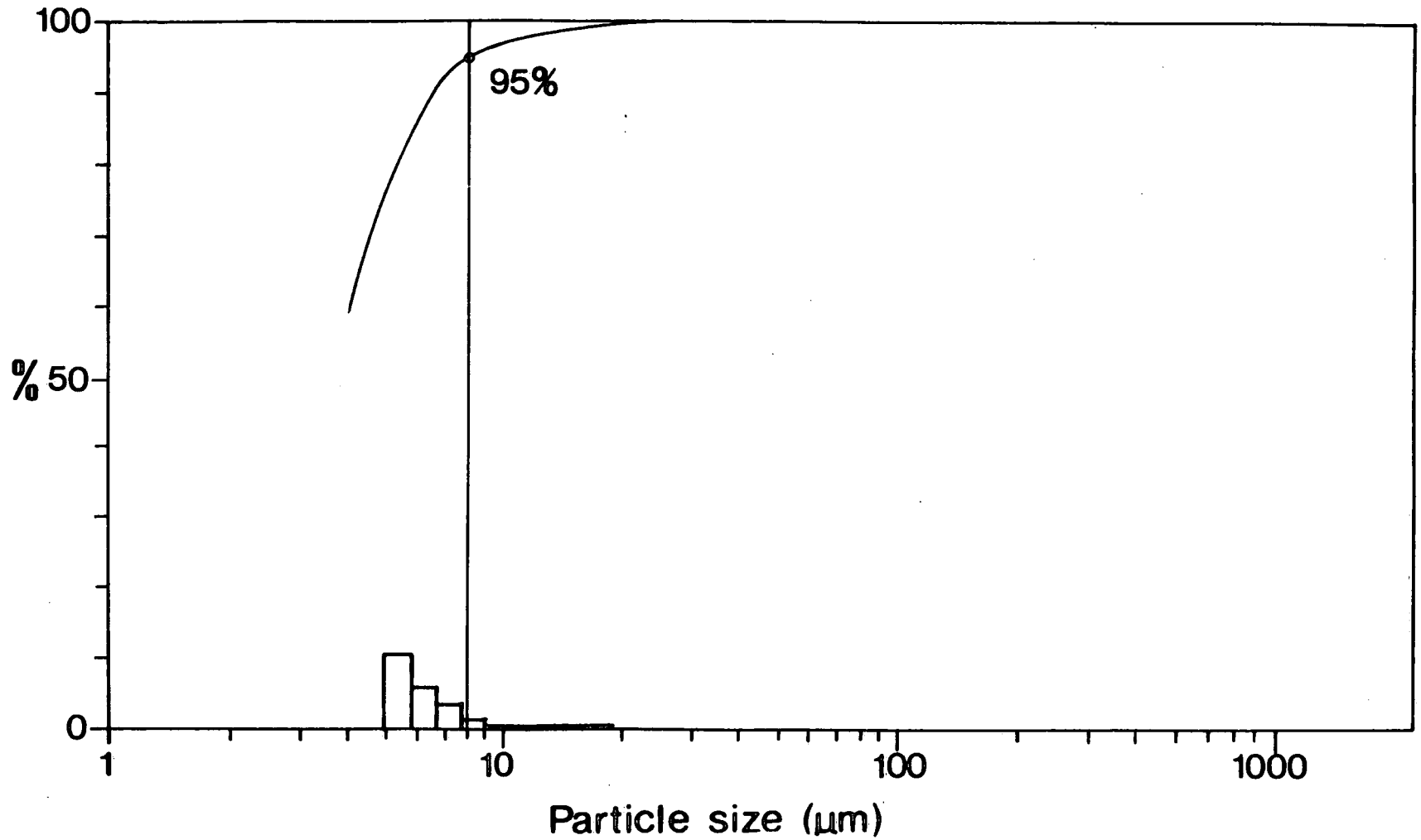

Fig.8. Size distribution of sediment fraction passing through 8 micron filter.

The distributions are plotted as histograms as well as percent finer. Figure 3, which shows the size distribution of sediment retained on the 60 micron filter, indicates that there is a considerable amount (approx. 35%) of sediment less than 60 microns. This may be due to clogging of the filter or flocculation during filtation. We minimized the amount of flocculation during filtration, however, by adding a dispersing agent (10% solution of hexametaphosphate). Flocculation of sediment in the sample cell of the Malvern Particle size analyses may also not be a factor because of the dispersing agent and the shearing action of the magnetic stirrer used to suspend the particles.

Figure 4 shows the size distribution of the sediment passed through the 60 micron filter and retained on the 40 micron filter. From this figure, it can be seen that a substantial amount (approx. 37%) of sediment contains particles larger than 60 microns. This is possible if some of the pore sizes of the 60 micron filter are in fact larger than 60 microns. Attempts to obtain specifications of the pore sizes of the nylon filters from the manufacturer were not successful. Evidence of filter clogging is also present for this filter because of the presence of particles less than 40 microns, although, the amount is small (6%).

Similar results were obtained for sediment fractions retained on 20, 10 and 8 microns. But, as the filter size decreases, the clogging appears to increase. Figure 6 which shows the result for sediment fraction passing through the 10 microns and retained on 8 micron filter indicates that 60% of the sediment is finer than 8 microns. This result is partially explained by the physical characteristics of the polycarbonate filter. The Nuclepore pores are generally cylindrical and have a random distribution normal to the surface. The

pores, however, cover only approximately 10% of the filter surface area. Deviations within the pore size is +0 to -20% of the rated pore size (Nuclepore Corporation). Thus the low porosity and existence of pore sizes smaller than the nominal pore size helps explain the large error seen with the 8 μm filter.

CONCLUSIONS

From this study we conclude that fractionation of inorganic sediment using cascade filtration technique is not exact and that filters retain a spectrum of sediments both greater than and smaller than the nominal pore size. The error induced by pore clogging is accentuated as pore size decreases. The amount of sediment larger than the nominal pore size retained on the filters increases from 35% and 37% for the 60 μm and 40 μm filter respectively to 60% for the 10 μm filter. Sediment larger than the nominal pore sizes found in the filtrate may be explained by the possibility that some of the pores of the filters could be larger than the normal pore size as specified by the manufacturer. The problems associated with filter fractionation techniques in general becomes far more severe for environmental samples which are likely to contain organic matter and bacteria. These are believed to be important controlling factors for the process of flocculation in fresh water systems (Droppo & Ongley, 1990).

To deal with the pore clogging problem, Rao and Kwan (1989) modified the cascade filtration procedure for environmental samples by resuspending the material collected in each filter by gently dipping the filter surface into the filtrate and thereby allowing a "backwash" of the filter to minimize filter clogging. However, the flocculation

can still be a problem. Furthermore, sampling of sediment by traditional methods is likely to alter the size distribution of sediment flocs and fractionation using any of the methods mentioned previously may not yield a true picture of various size fractions that exists in a flow field. Further research is needed to address the problem of size distribution of suspended aggregates from natural river systems and its effect on contaminant adsorption.

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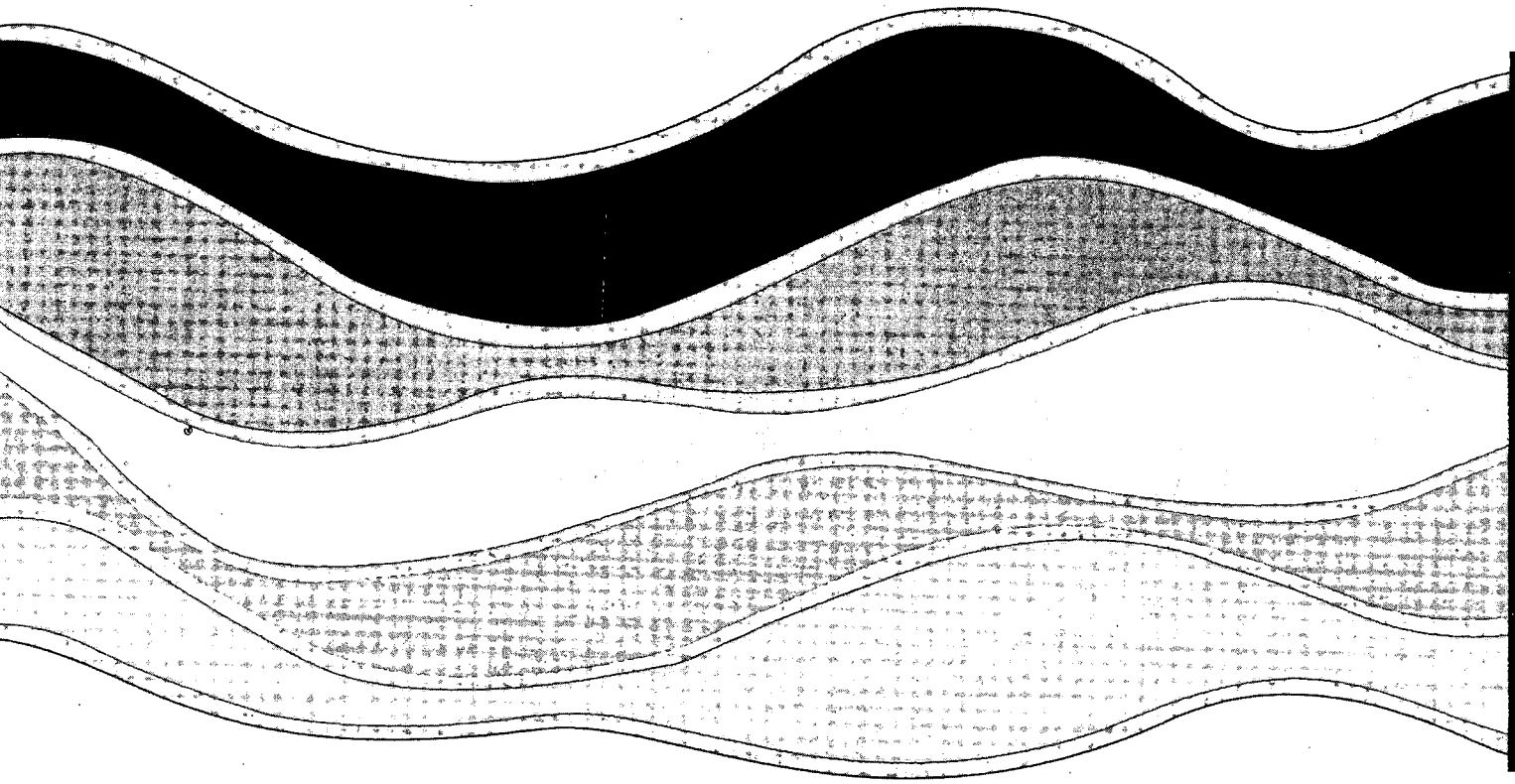

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