An Evaluation of the Sedigraph as a Standard Method of Sediment Particle Size Analysis

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Abstract

Sediment particle size is an important measure in many facets of hydrological research. Various tests were undertaken to evaluate the applicability of the sedigraph as a standard instrument for particle size distribution analyses. The results show, amongst other things, a good reproducibility, agreement with standard samples but disagreement with presently used techniques which are themselves not in agreement. The absolute results of the instrument are not considered to be the main criterion, but rather the reproducibility of results, the time involved in analyses, compatibility with results from elsewhere and the ease of operation. The sedigraph fulfils all these criteria adequately.

Introduction

Sediment particle size is an important measure in many facets of hydrological research. Examples of fields of research requiring particle size measurement include soil erodibility studies (Wischmeier, Johnson and Cross, 1971); sediment transportation and paleohydraulic studies (Blatt, Middleton and Murray, 1972); reservoir sedimentation studies (Gottschalk, 1964); studies of densities of reservoir sediments (Koelzer and Lara, 1958); studies of sediment-pollutant relationships (Pionke and Chesters, 1973) and studies of sediment-nutrient relationships (Wang, 1974). One of the most common reasons for particle size measurement is to classify sediments.

The determination of sediment particle size is standard practice at institutions concerned with the above problems. Numerous different techniques exist for the measurement of particle size, making the selection of the most suitable method a difficult task. One of the serious problems limiting the number of samples that can be analysed in the particle size range of less than 2 μm, is the long time required for a single analysis. Recently a new technique of particle size analysis in the fine mode has been introduced, namely, the use of a sedigraph.

As the use of this method is relatively new and is not discussed in the literature as frequently as the other methods, a brief description of the instrument and its principle of operation is offered. A more detailed description can be found in Oliver, Hickin and Orr (1970). The sedigraph allows for the rapid automatic measurement of particle size in the fine mode. The rate of sedimentation of particles is recorded and is automatically presented as a cumulative per cent distribution in terms of Stokesian equivalent diameters. The sample to be analysed is introduced as a suspension to the sedimentation cell by means of a peristaltic pump. A finely collimated beam of X-rays is passed through the suspended medium. Radiation is detected as pulses by a scintillation detector on the opposing side of the sedimentation cell to the source. The concentration of sediment in the beam is proportional to the X-ray intensity. The sedimentation cell is continuously moved across the beam to reduce the time needed for analysis. A built-in solid-state digital computer solves Stokes' Law and presents the final result linearly as a cumulative mass per cent distribution.

At the Hydrological Research Institute (HRI) settling velocity methods are used for sizing fine material and sieving for the coarse fraction. Methods compared in the present study include the pipette method, the hydrometer method, the sedimentation balance method, the sedigraph method, sieves and microscope method.

Materials and Methods

The definition of particle size largely depends on the method used. Table 1 summarizes the many different definitions of particle size. Comparison of results using different methods of analyses (and therefore different definitions) can be misleading as the assigned size depends on the method. Possibly the most comprehensive discussion of these methods and their inherent weaknesses is that by Allen (1975). Other useful references include those by Baver (1956), Blatt *et al.* (1972), King (1966), Krumbein and Pettijohn (1958), Kuenen (1968) and Pettijohn, Potter and Siever (1972).

Standard equipment was used and the methods were based on the standard methods prescribed for the instruments. Experiments were carried out to determine the effects of varying concentrations on particle size distribution curves, the reproducibility of particle size distribution curves and the effect of cell size variations on particle size distribution curves. Further tests involved the analysis of standard reference material on the sedigraph and comparisons of the particle size distribution of natural silt and clay samples obtained using various methods.

Experiment to determine the effect of varying concentration on sedigraph results

Kuenen (1968) showed that hindered settling occurred when particles were 6 diameters or less from each other. The solid particles tended to sink at a constant velocity while the liquid flowed upwards. When particles were small distances apart, this upward flow tended to retard the fall of other particles. Hindered settling was said to be absent in suspensions with concentrations lower than 2% (by volume) and negligible in suspensions with concentrations up to 5% (Baver, 1956). The first experiment undertaken was to investigate whether hindered settling occurred within the concentration limits recommended by the manufacturers. A reservoir bottom sediment sample

TABLE 1

DIFFERING DEFINITIONS OF PARTICLE SIZE. (PETTIJOHN, POTTER AND SIEVER, 1972)

Symbol	Name	Definition
d_s	Surface diameter	The diameter of a sphere having the same surface area as the particle.
d_v	Volume diameter	The diameter of a sphere having the same volume as the particle.
d_d	Drag diameter	The diameter of a sphere having the same resistance to motion as the particle in a fluid of the same viscosity and at the same velocity.
d_a	Projected area diameter	The diameter of a sphere having the same projected area as the particle when viewed in a direction perpendicular to a plane of stability.
d_f	Free falling diameter	The diameter of a sphere having the same density and the same free-falling speed as the particle in a fluid of the same density and viscosity.
d_{st}	Stokes' diameter $d_{st} = (d_v^3/d_d)^{1/2}$	The free-falling diameter in the laminar flow region (Reynolds Number <0,2).
d_A	Sieve diameter	The width of the minimum square aperture through which the particle will pass.
d_{vs}	Specific surface diameter $d_{vs} = d_v^3/d_s^2$	The diameter of a sphere having the same ratio of surface area to volume as the particle.

(Spitskop Dam) was prepared. Three runs were carried out on the sample. The first run began with a concentration as close to the minimum required intensity output as possible. Subsequent runs were undertaken on more diluted samples which had a higher intensity output.

Experiment to determine the reproducibility of results on the sedigraph

Four samples with varying particle size distribution were each rerun a number of times. The samples included one of the United States Department of Commerce having a narrow range of particle size and 3 natural samples of varying coarseness. For the finest natural sample, seven runs for different 40 cm³ sub-samples drawn from the same 2-parent sample were undertaken. This was done so that the representativeness of the small sample could be checked.

Experiment to determine the effect of cell size on settling

Stokes' Law assumes that the extent of the liquid is great in comparison to the size of the particles in suspension. Köhn (1928) suggested that particles closer than 0,1 mm to the wall of the settling vessel encountered appreciable resistance to fall. It stands to reason that the smaller the settling vessel, the greater the probability that wall effects will affect results. Comparative tests were therefore undertaken at the Council for Scientific and Industrial Research (CSIR) using their smaller cell and the larger HRI cell on the same sample.

Experiment to determine the accuracy of results by analysing standard samples

Three standard samples were analysed, viz a 12 μm "Micromeritics"* garnet sample, a 3,85 μm "Micromeritics" garnet sample and a United States Department of Commerce standard glass beads sample. Micrographs were also taken of these samples with scales calculated from lens magnification factors.

*The use of trade names does not suggest the recommendation of such products by the Department of Water Affairs, Forestry and Environmental Conservation.

The effect of varying concentration on sedigraph results

A maximum concentration of 5% by volume is suggested by Baver (1956) for methods using settling velocity techniques. The manufacturers of the sedigraph recommend that concentrations should be held to the minimum level consistent with a statistically reliable X-ray output, i.e. a minimum reading of 25 μA on the intensity meter (Micromeritics, 1978). The manufacturers also claim that no particular concentration is needed, as long as the radiation beam intensity is reduced by 60 – 40 %. The recommended procedure is to prepare a sample of approximately 5 % by weight and to add this to pure liquid until an adequate concentration is achieved (Micromeritics, 1978). The results of an experiment designed to evaluate the effect of varying sediment concentration in the sample to be analysed are presented in Figure 1. The curves show very little dissimilarity in particle size distribution at varied concentrations obtained with the sedigraph method.

These results support the claim by the manufacturers that widely variable sample concentrations can be accommodated by this method. It can be concluded that varying concentrations will have no significant influence on the results as long as the suggested concentration limits are adhered to.

The reproducibility of the sedigraph method

Figure 2 depicts the results of the experiment designed to test the reproducibility of results. For the first sample (US Department of Commerce glass beads), results agreed perfectly after four runs. The only time disagreement was found was when the starting diameter was higher than is recommended (defined in Micromeritics, 1978). This curve differs from the mean value for the sample by a maximum of 8% or 1,5 μm, depending on which axis is used as a reference axis.

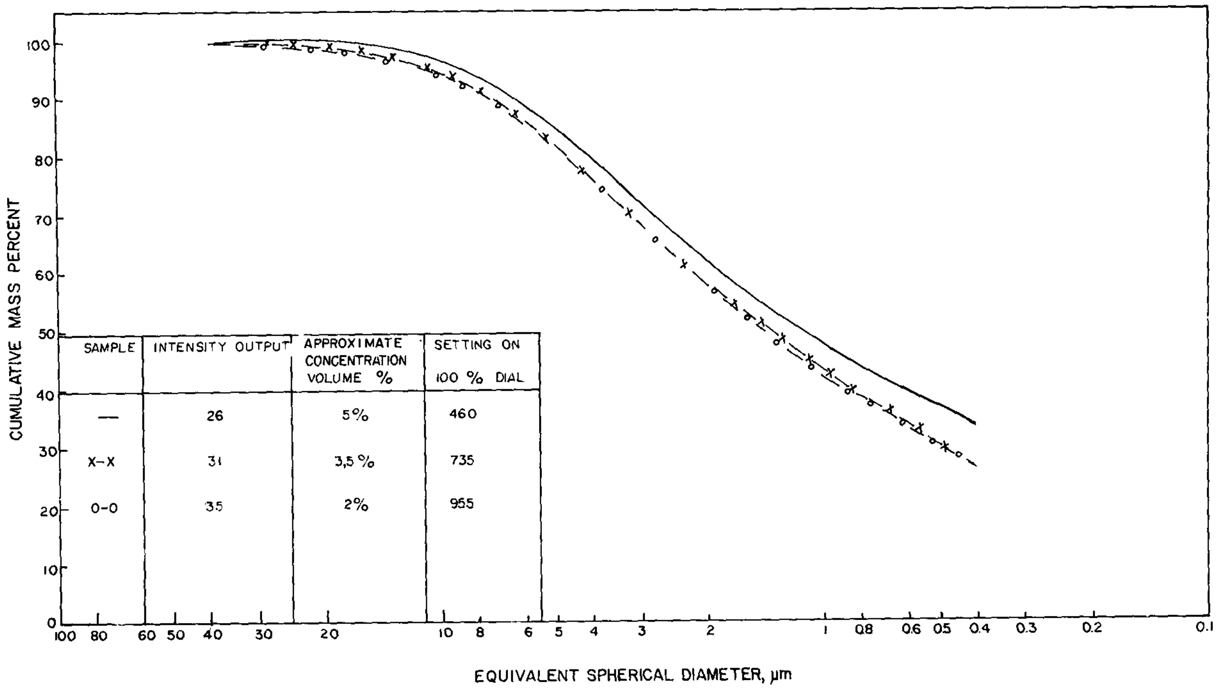

Figure 1
The effect of varying sample concentration on particle size distributions determined with a sedigraph

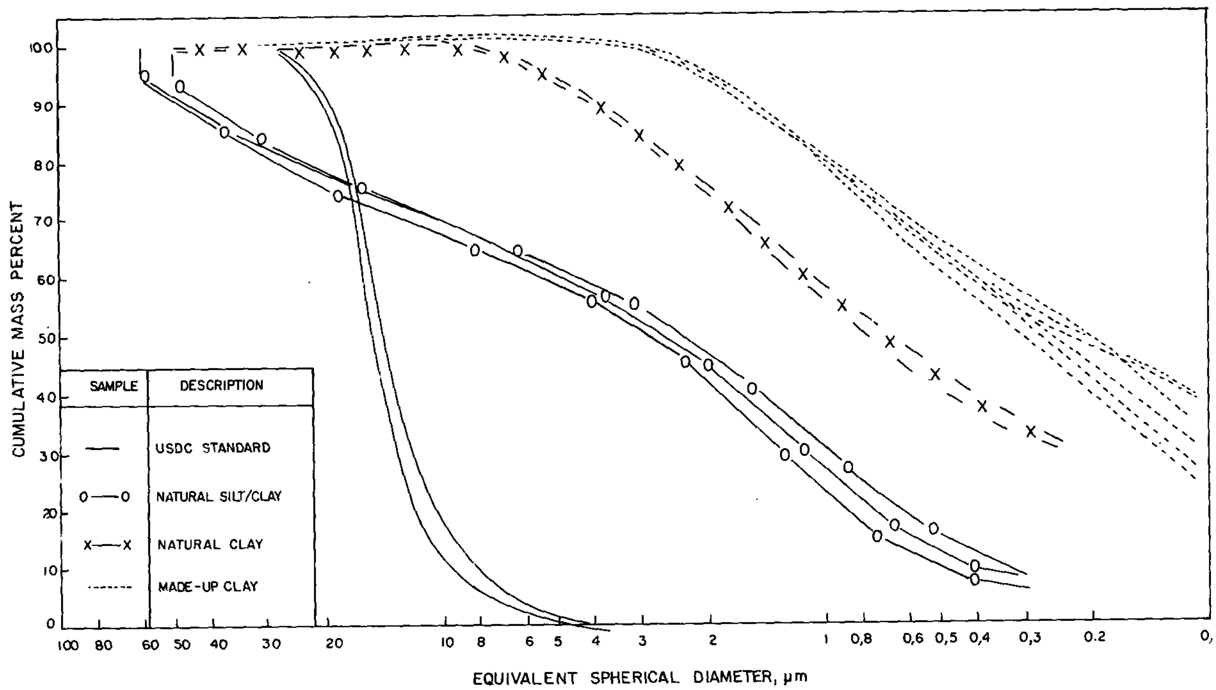

Figure 2
Reproducibility of results on sedigraph for various samples

The variation in results for the remaining samples of natural clay/silt, natural clay and the made up clay is given in Figure 2. Most sedimentological studies categorize sediments fairly broadly and variations such as shown fall well within acceptable ranges.

The similarity in results obtained for each separate 40 cm^3 subsample of the made up clay sample, illustrates the representativeness of the results even though such a small fraction of the parent sample is used.

Experiment to compare sedigraph results with various other methods

The hydrometer, pipette, sedimentation balance and sedigraph methods were used to determine the particle size distribution of the same sample. The sample contained mainly clay with a particle size $< 2\ \mu m$. A sample concentration of 1,5% by volume was used for all methods except the sedimentation balance. Two runs were completed with the hydrometer, pipette and sedimentation balance methods and 7 runs with the sedigraph method. More runs could be done with the sedigraph because of the shorter time needed for sedigraph runs in comparison with other methods. A micrograph of the sample was used for further comparison.

Results and Discussion

The results of the experiments described are in the form of a number of graphs. These graphs have been retraced so that comparisons of curves can be made on the same figure.

The effect of cell size on settling

Figure 3 depicts two curves for a silt sample obtained using different cell sizes in the same sedigraph. It has been pointed out that Stokes' Law assumes a settling vessel of infinite size and that retardation of settling by the walls is negligible. Assuming that particles within 0,1 mm from the walls of the settling vessel are hindered in their settling (Köhn, 1928), the proportion of particles hindered by wall effects can be calculated by expressing the volume of particles within this 0,1 mm zone as a per cent proportion of the total volume of sample obtained in the vessel. Table 2 presents these calculations for the hydrometer and pipette vessels and for the CSIR and HRI cells.

From the data in Table 2, it can be expected that wall effects will be most effective in the smaller sedigraph cell. The differences between the results in Figure 3 are too small to be attributed solely to wall effects as they are of a similar magnitude to variations shown in Figure 2. Oliver *et al.* (1970) calculated diameter correction values for the influence of wall effects. These correction values are presented in Table 3. These values are computed for a sedigraph with a cell half the thickness of the HRI cell. The theoretical diameter correction for this cell will, therefore, be considerably less than shown above. Based on the values given in Table 3 and the close correlation between results obtained with two different sized cells, it is concluded that wall effects are not significantly strong.

The accuracy of sedigraph analyses of standard samples

Plots obtained for the standard garnet samples are presented in Figure 4. The 4 curves include 3 for the 12 μm standard (one on

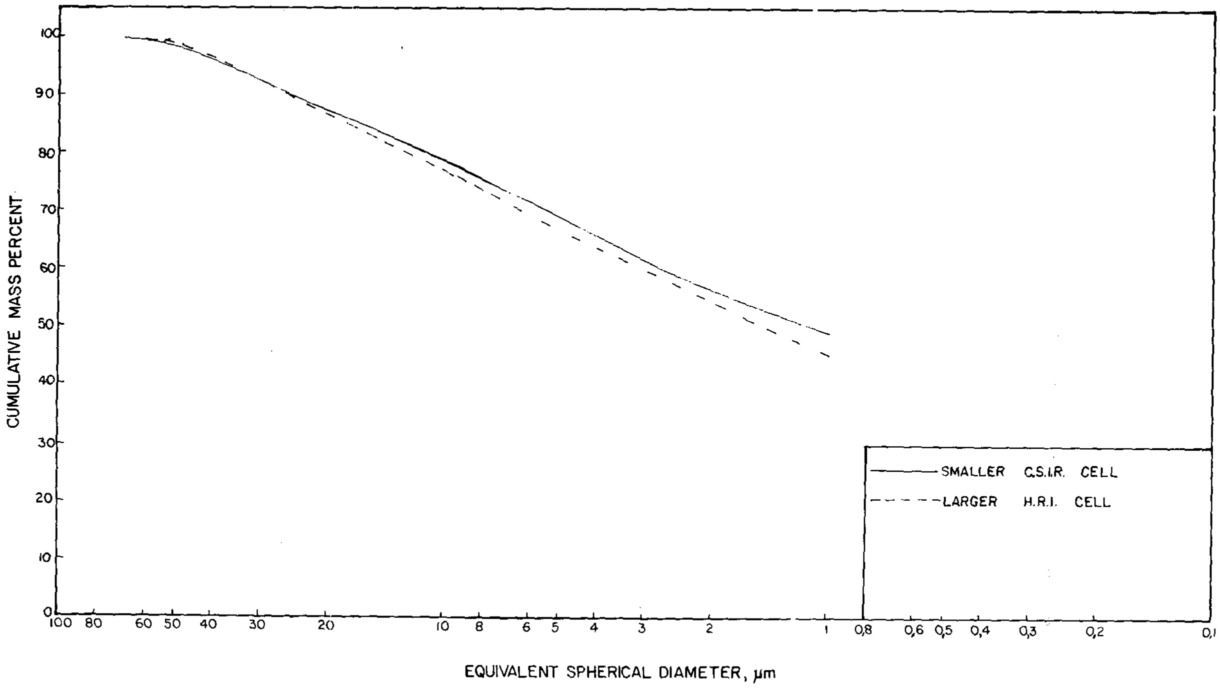

Figure 3
Two runs on the same sample using two different size cells in the sedigraph

TABLE 2
PROPORTION OF PARTICLES SUBJECT TO WALL EFFECTS

Vessel	Capacity of Vessel (cm^3)	% Total sediment subject to wall effects (to nearest 1%)
Hydrometer/ pipette Cylinder	1000	1%
CSIR Sedigraph Cell	1,58	7%
HRI Sedigraph Cell	2,70	5%

TABLE 3
CORRECTION FOR THE INFLUENCE OF THE WALL OF THE SEDIMENTATION RESERVOIR (OLIVER, HICKIN AND ORR, 1970)

Particle diameter (μm)	Diameter correction (μm)
50	0,021
20	0,010
10	0,006
2	0,0014
0,4	0,0003
0,2	0,0002

the HRI sedigraph, one on the CSIR sedigraph and one on the sedimentation balance) and one on the 3,85 μm garnet (HRI sedigraph).

From Figure 4 it follows that the sedigraph is producing the median diameter given by the suppliers. The sedimentation balance, however, produces results considerably coarser than those determined by the suppliers. A micrograph of the 12 μm sample (Plate 1) shows that the particles fall into Powers' "very angular" class of roundness (Blatt *et al.*, 1972). The angularity of the particles makes an accurate determination of diameters from the micrograph difficult.

Figure 5 presents 3 plots for the US Department of Commerce standard glass beads. The plots include one obtained on the CSIR sedigraph, one on the HRI sedigraph and one of the measured diameter of the beads (as given by the suppliers). A micrograph (Plate 2) of the sample shows "well rounded" spheres (Blatt *et al.*, 1972) with easily defined diameters. The sample comprises microscopically measured glass beads issued for use in calibrating equipment and evaluating methods for measuring particle size in the 50 to 30 μm range. Graphs accompanying the sample represent distributions according to volume, weight, Stokes' Law in air and Stokes' Law in water. The plots shown in Figure 5 show a virtually perfect agreement between the standard curve and sedigraph results.

A comparison of sedigraph results with various other methods

Figure 6 depicts curves drawn for results obtained using the sedigraph and sedimentation balance methods on a silt sample. Figure 7 summarizes results using hydrometer, pipette, sedimentation balance and sedigraph methods on a clay sample. The sedigraph consistently gave finer particle size distribution compared with the other methods.

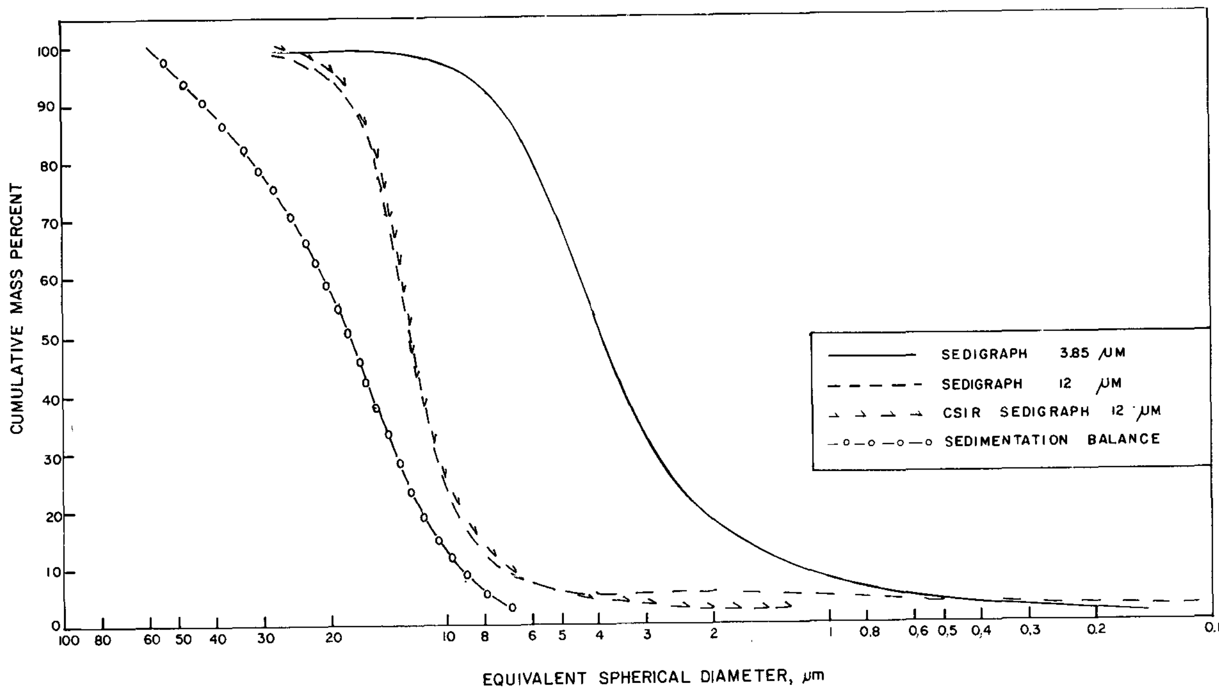

Figure 4
Micromeritics standard garnet samples using sedigraph and sedimentation balance methods

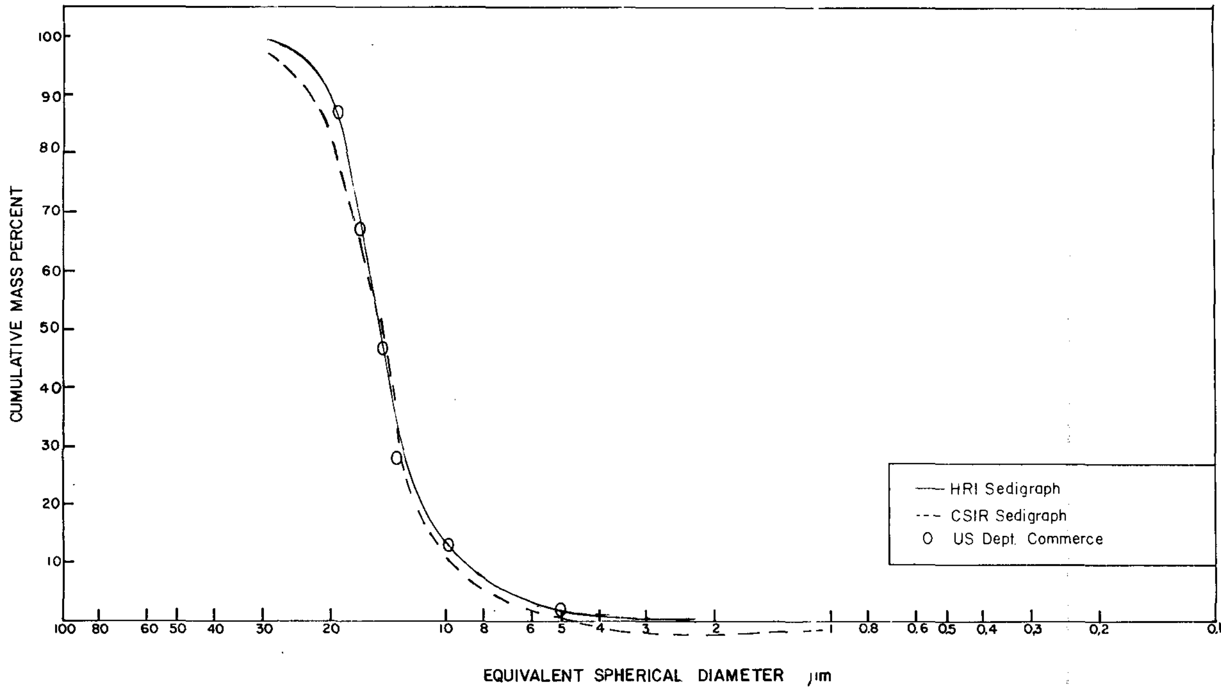

Figure 5
Particle size analyses of U.S. Department of Commerce standard glass beads

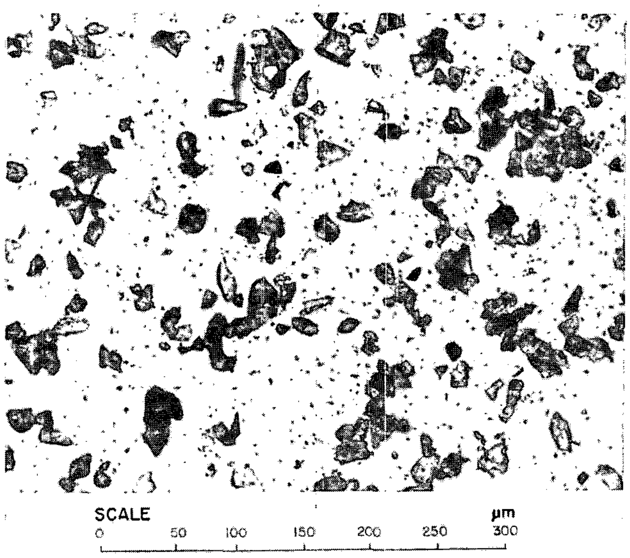

Plate 1
Micromeritics 12 µm standard garnet sample

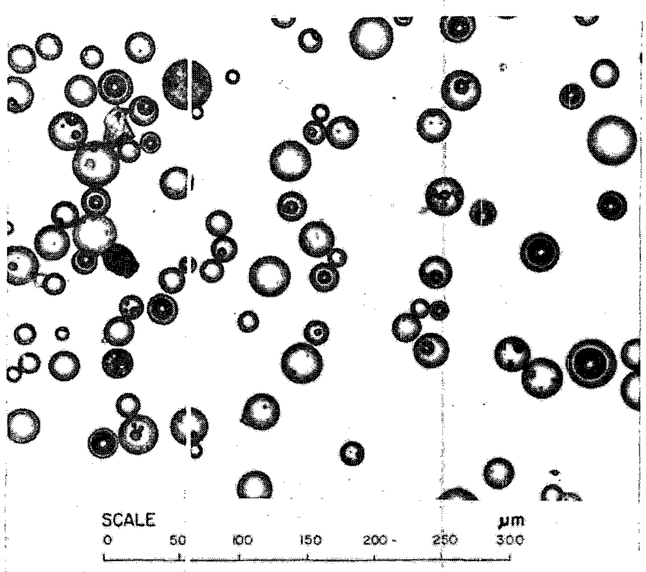

Plate 2
United States Department of Commerce standard glass beads

It has been pointed out that the definition of grain size depends on the method used. The determination of particle size by using settling velocity is referred to as the "equivalent spherical diameter", and is an indirect method. If different definitions of particle diameter (due to the use of different methods) are compared, different results are possible. For example, Head (1979) observed that sedigraph and Coulter Counters tend to show finer results than a micromerograph on the same silicon samples. The tendency for sieves to show finer results than hydrometers was also observed by Weaver (1979). It is for this reason that 4 settling velocity methods are compared in the present experiment. The methods all depend on manual

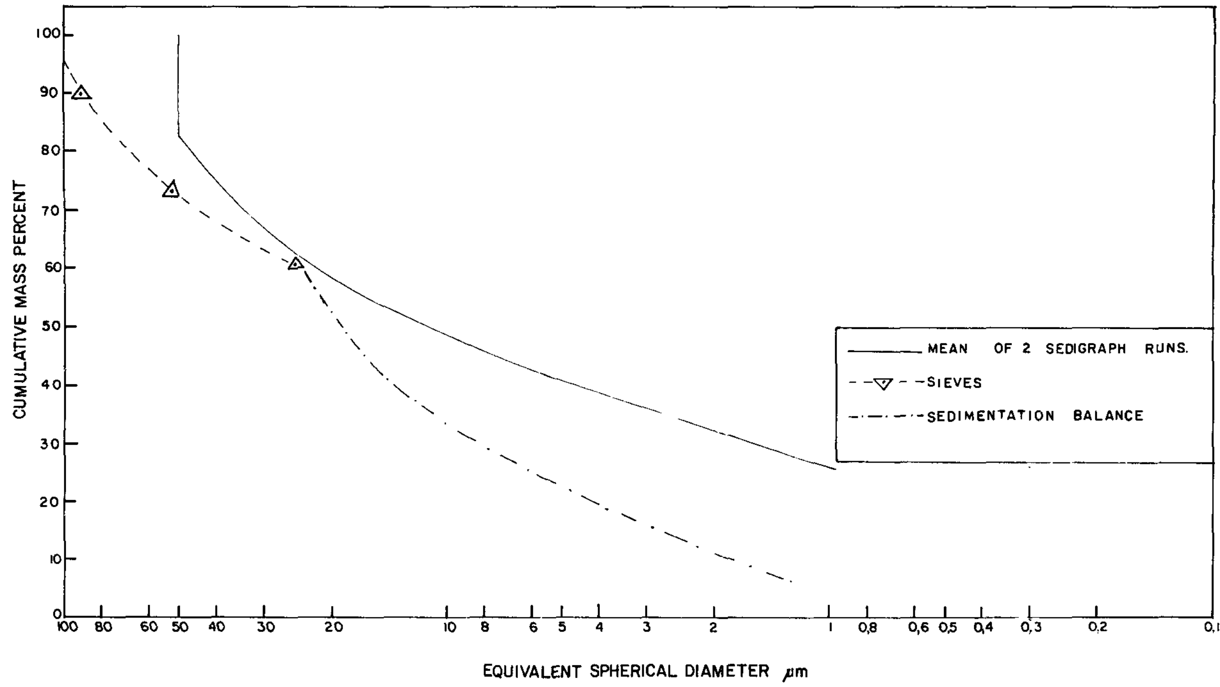

Figure 6
Particle size analysis of natural silt sample using sedigraph, sieves and sedimentation balance methods

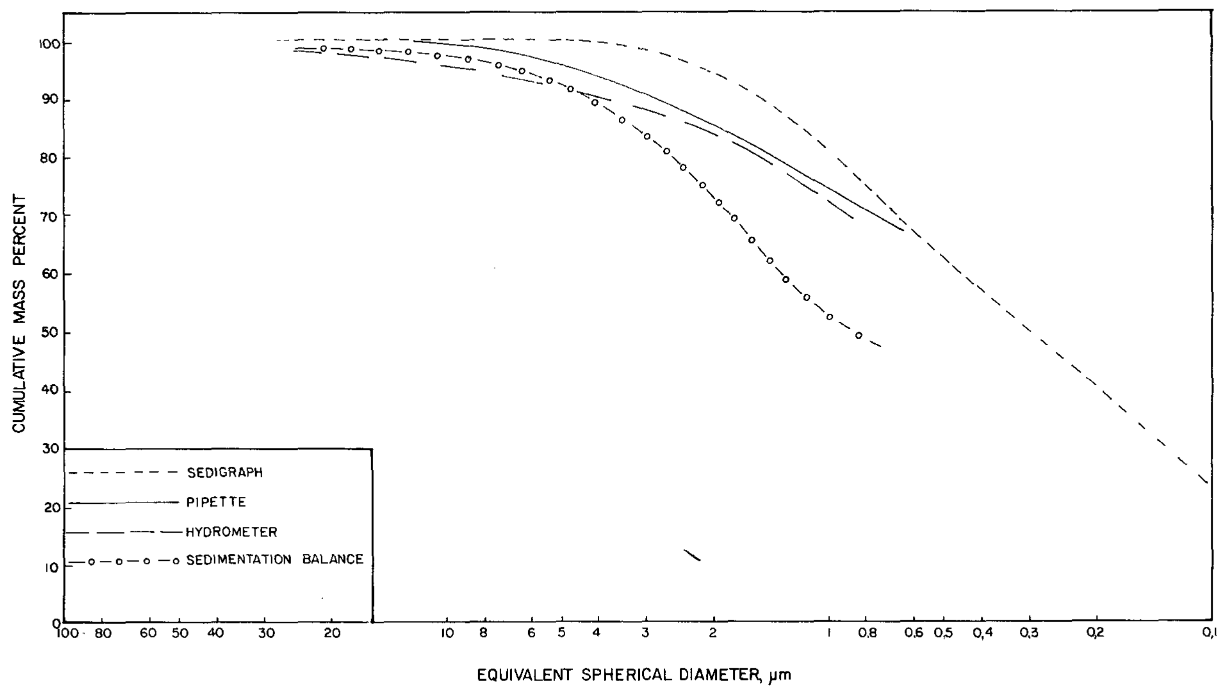

Figure 7
Particle size analysis of natural A clay sample according to various methods

or mechanical computations based on Stokes' Law for the calculation of the equivalent spherical diameter of particles. Results obtained should theoretically be in agreement.

In the sedimentation balance method Scott and Mumford (1971) observed that a certain portion of the sample to be analysed tends to miss the balance pan and the particles bypassing the pan consist of the finer fraction. Du Toit (1979) observed that between 5 and 40% of the sample bypasses the pan. The finer the original sample, the greater the percentage passing the pan.

Scott (1979) suggests that the finer particles are drawn past the sedimentation pan by convection currents set up during the pan readjustment process. A further possible explanation is that the finer particles in the pan repel each other, resulting in the "slipping off" of the finer fraction past the pan.

Although the above explanation could be used to account for the sedimentation balance to show coarser particle size values in Figures 6 and 7, differences between the pipette, hydrometer and sedigraph methods remain unexplained. Further evidence was, therefore, sought from the micrograph of the clay sample (Plate 4) and in comparison with the glass beads (Plate 3), the maximum sized clay particles were considerably fine. Yet the hydrometer and sedimentation balance showed maximum values of greater than 25 µm and the pipette of 12 µm. The maximum sized glass bead (microscopically determined) was 30 µm. Based on a visual comparison of the clay samples and the beads, there were certainly no clay particles approaching 25 µm; even 12 µm appeared to be in excess of those particles depicted in Plate 3. It appears therefore that the 100% cut-off points on this graph for these three methods (sedimentation balance, pipette and hydrometer) is suspect. Even though it can be argued that microscope and sedimentation methods define particle size differently, it is unlikely that such large discrepancies will result by comparing these two methods. Taking the microscopic evidence into account it can be concluded that the sedigraph method gives a true reflection of the actual particle size distribution of the sample although it differed from the results obtained by the other methods.

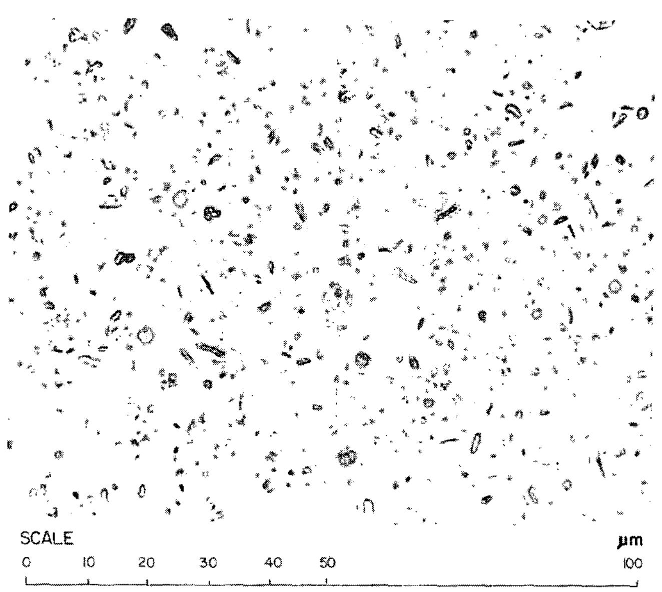

Plate 3
Natural clay sample

Conclusions and Recommendations

1. Measurement of particle size distribution using the sedigraph reduces the time considerably. For example, measurement of particle size down to 0,1 µm using hydrometer, pipette or sedimentation balance methods would take approximately 720 h whereas the sedigraph can complete this analysis in 5 h.

2. The sedigraph gives results with good repetition. The results agree with those obtained on a different sedigraph using a different cell size for the same samples. The sedigraph also produces results in very close agreement with standard samples.

3. Discrepancies in results obtained with the sedigraph, pipette, hydrometer and sedimentation balance methods are disturbing. A microscopic analysis for the same sample tends to support the results obtained with the sedigraph method.

4. The conclusion of this study is not to state that the one technique provides "correct" results and the other not. Such a statement is bound to be misinterpreted and to receive criticism from the manufacturers of the instruments as well as from advocators of traditional methods. It is, however, clear that "different" reults are obtained using various instruments. The choice of instrument will determine the "grain size" measured. This choice will depend largely on convenience, availability, reproducibility and comparability of results to those obtained by other research workers in related fields. The sedigraph fulfils these criteria adequately and it is therefore suggested that it could be employed as standard tool for the measurement of sediment particle size distribution.

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