Error Estimates in Size Analysis of Marine Sediments

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Repetitive size analysis of 28 marine sediment samples, carried out according to established procedures, indicates systematic and cumulative errors up to 18%. Percentages of errors at different stages of pre-treatment of the sample are located. By adopting differential pre-treatment of the marine samples, size parameters vary significantly. Instead of the presently followed elaborate pipette analysis for determination of the sand-silt-clay ratios, a simpler procedure is suggested wherein the error is reduced to 3%.

Review of various methods of size analysis—pipette analysis, Ro-tap, electronic particle counting—reveals that each technique has its advantages and disadvantages. The accuracy of size analysis is also determined with different diameters of the settling tube. A critical study has been made to see how the shape of the grains will modify grain size parameters. Modified methods and new techniques are evolved in an attempt to minimise the errors and to increase the reproducibility of size analysis. Since weight and cumulative percentages are computed on the basis of total weight of different fractions, till now the percentage of errors is not estimated in any of the earlier works even though they are known to exist.

The results of replicate size analysis indicate that the errors are systematic and cumulative, and tend to increase with decrease in the quantity of the sample. Though the convenient sample size is 25 to 30 g, in case of marine sediments work has to be done with only 10 g of sample. In this paper, size analysis on marine sediment samples is given and errors at different steps of the accepted procedure are discussed and suggestions made to minimise the errors.

Pre-treatment—Grain size analysis of 28 shelf sediments from parts of the east coast of India is carried out by sieve analysis and pipette analysis after pre-treating the sample.

The sample is thoroughly washed to remove salts present in it. But in the case of marine samples the fine clay which is present in considerable quantities is also washed off along with the salts, as the suspended fine clay will not settle before 14 h of standing (if centrifuge is not used). The amount of fine clay lost is estimated by taking the difference in weights of the sample before and after 3 or 4 times of decantation (after 14 h of standing).

After the removal of carbonates and organic matter, sand is separated from silt and clay fractions by wet sieving, during which a very small fraction of fine material (−230 fraction) still adheres to sand in the sieves. This fine fraction separates out during subsequent sieving of the dried sample and this should be added to the silt and clay. Otherwise the maximum error will be about 3% in the sandy samples. The percentage of error in the sand is calculated from the weight of the −230 fraction obtained after sieving the dried sample from which silt and clay fractions have been separated by wet sieving.

Errors in pipette analysis—The quantity of the sample is chosen such that the dispersed silt-clay fraction should not exceed 2% in the medium to avoid coalescence. After a settling time of 25 sec at 27°C, 20 ml of suspended material is drawn with a pipette. The amount of material finer than 4 μ can thus be estimated. It takes minimum 6 sec to pipette out 20 ml during which time some of the coarse silt settles down. Consequently the results show lower percentages of silt-clay fractions than the actual values. The error depends on the quantity of 4 to 5 μ size range present in the sample and shows a maximum of 15% in silty samples. If only 10 ml is pipetted out instead of 20 ml the duration of the operation is reduced nearly to half and the error is reduced to 3%. The error is estimated from the difference in the weight fraction calculated from the suspended material when 20 and 10 ml are pipetted out respectively. Settling times according to the diurnal variation of temperature must also be taken.

<table>
<thead>
<tr>
<th>Nature of sediment</th>
<th>No. of samples analysed</th>
<th>Av. % of clay finer than 10 μ</th>
<th>Error range, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sandy</td>
<td>9</td>
<td>-</td>
<td>0.2</td>
</tr>
<tr>
<td>Silty</td>
<td>11</td>
<td>3.5</td>
<td>4.4</td>
</tr>
<tr>
<td>Clayey</td>
<td>8</td>
<td>6.0</td>
<td>7.6</td>
</tr>
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</table>
The calculated error in the settling time is - 2.4% for every increase of 1°C. A change in the value of acceleration due to gravity (g) at the laboratory set-up can bring about an error of 0.5% (calculated) of the settling time which is insignificant.

The same sediment is subjected to different pre-treatments: (a) original sample without any pre-treatment; (b) organic and calcareous matter decomposed with 30% H₂O and 1 NHCl; (c) clay finer than 10 φ separated and organic matter destroyed and (d) clay finer than 10 φ separated and calcareous and organic matter decomposed with 1 NHCl and 30% H₂O₂.

The analysis shows that the maximum error is about 4% in sandy and 18% in clayey sediments (Table 1). The results (Table 2) show unequivocally that the error is less for samples in which fine clay is separated during the pre-treatment. The size parameters of the same sample are calculated and presented in Table 3. The lower value of the φ mean size, of samples A and C (compared to B and D, respectively) in Table 3 is due to the presence of coarser carbonate grains which are dissolved during acid treatment in samples B and D. The skewness has decreased significantly after pre-treatment of the sample D compared to A-C and this is also due to the removal of coarser carbonate material during acid treatment. The pre-treatment of the samples changes the grain size distribution and consequently the sorting and kurtosis values reflect the changes in the grain size distribution.

**Estimation of sand, silt and clay**—Their content in samples can be determined rapidly, by pipette analysis following the procedure: (1) sand coarser than 4 φ is allowed to settle and suspended silt-clay finer than 4 φ in 10 ml will give the amount of silt plus clay as well as sand percentage; and (2) silt coarser than 8 φ is allowed to settle and from the amount of suspended clay finer than 8 φ in 20 ml of aliquot percentages of silt and clay are calculated.

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**References**