PARTICLE-SIZE ANALYSIS OF SILTS

An Optical Method of Size Analysis for Silt Size Particles

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Abstract

A new optical method for size analysis using a focused laser beam is described. It is rapid and non-destructive. The method can be used on a suspended sediment in any clear liquid. It reads in 16 channels of constant width which can be set to any desired width from 0 to 10 microns. The limits of detection are 1 micron to 10 microns in diameter and 0 particles to 999 particles per milliliter.

Introduction

Particle size analysis is one of the first things done when working with unconsolidated sediments. It gives a basic understanding of what can be expected from the sediment. Traditional methods of size analysis, especially for those sizes less than 63 microns are usually lengthy and the properties being measured are not always known or agreed upon (Swift, Schubel, and Sheldon, 1972). Other drawbacks of these methods concern the sample itself or the way it is treated. Sample sizes need to be large, 15 g/l to 5 g/l, below which analysis becomes very inaccurate (Swift, Schubel, and Sheldon, 1972). Sedimentation tubes commonly used for pipette analysis are not large enough and may produce inaccuracies up to 34% (Gibbs, 1972). The size distribution of the sample itself may effect the results. Studies by Subramanya and Valsangkar (1973) indicate that greater than 0.5% clay content in suspension alters the fluid properties and increases viscosity dependently on the amount of clay. This increases the settling time for silt particles and decreases the apparent size of the particle. In many of these methods the sample is destroyed or made useless for other analysis.

This report deals with a non-destructive optical method of silt size particle analysis (using Spectrex Corporations Laser Particle Counter). The method can be performed quickly and does not require much sample preparation. A minimum of sample handling helps ensure an unbiased measurement. This method is unlike earlier optical methods in that it does not examine particles in a mass after a settling period (Stamm and Svedberg, 1925) or by means of turbidity or absorption (Swift, Schubel, and Shelton, 1972). A single particle is detected by a laser beam. It determines the size and counts the particle without interference from neighboring particles. One particular advantage to this method is that any clean, clear liquid medium can be used, without regard to density or di-electric properties.

Principles

This method of particle analysis can be used because of two assumptions and the conclusions that follow from them. The first is that the particle population is low, less than 999 particles per milliliter, so that the way a particle in the path of the laser beam scatters the incident light is independent of surrounding particles. This means that only one particle at a time is sending a signal to the counter. Low concentration also keeps coincident particles to a minimum.

The second assumption is that the particles are large enough, greater than five times the wavelength of the light, so that true reflection of light is occurring (Stamm and Svedberg, 1925). This means that the light reflected is proportional to the surface of the particle. Light reflected in a near forward direction between 6.34° and 18.95° is detected by the counter. Only the surface of the particle where light impinges between 81.89° and 87.17° from normal to the surface is detected by the unit. This surface is dependent on the size of the particle and may be approximated by the equation:

$$P \propto S = \int_a^b 2\pi / x / dx$$

Where P is the light detected by the unit, S is the surface area detected, a and b are the bounds of the surface detected, and x is the function of the radii of the particle (after Riddle, 1974). This is shown graphically in Figure 1.

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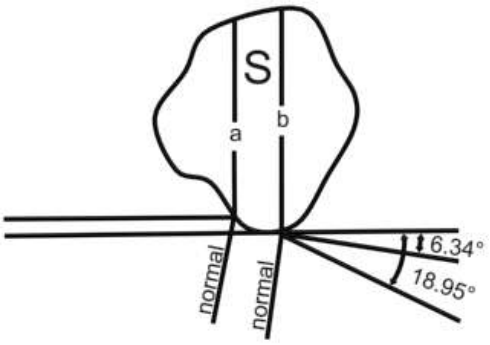

Figure 1

A graphical representation of the surface and bounds of a particle detected by the laser unit. See text for explanation.

METHODS

A focused laser beam is directed through the sample solution. Particles are detected when they pass through the laser in the focus area. Light is scatted from the particle and a photo electric cell detects that portion of the light reflected in the near forward area 6.34° to 18.95° from the light path. The light pulse generated by the particle is analyzed by a detection unit and any abnormal pulse is rejected. An abnormal pulse can be caused by a particle out of the focus area of the laser

or a particle in the focus area which does not pass completely through the laser beam. Particles grazed by the laser reflect less light than the size of the particle would indicate and the pulse of light is shorter than if the particle were hit completely by the laser beam. Short pulse duration is the determining factor for rejection of pulses caused by a particle grazing the light beam. Particles outside the focus area of the laser cause a diffused flash of light on the photo cell and the detection unit rejects these diffused pulses (Spectrex, 1975).

Particle densities should be kept low so there is little chance of coincidence as particles are crossing the laser path. Coincidence would result in one particle being shielded by another and not being counted or the particles combining to give an inordinately large pulse for their size. Shielding by particles outside the focus zone would also cause rejection of particles in the focus zone. Particle density should be low enough so that when a sample is visually examined it should be clear or only slightly clouded.

SAMPLE PREPARATION

Samples for this study were obtained from cores of unconsolidated sediment on the continental shelf in the northeast Gulf of Mexico. They were prepared as for a pipette analysis by wet sieving through a 63 micron screen and retention of the pan fraction from the dry sieving. All samples were brought up to a liter volume. A sample of 20 ml was withdrawn and retained for analysis by the laser unit. Samples were then analyzed by pipette for less than four phi to greater than eight phi by whole phi increments for comparison to the results of the laser unit. Replicates were done for a total weight of the silt fraction.

The 20 ml samples taken for the laser analysis were diluted to 1 : 1000 or about 5 mg/l. Dilutions were done in two stages to allow replicates of the final dilution to be made from the first dilution. The prepared samples were 1 liter of 1 : 1000 and were agitated for uniform dispersion. A 200 ml alloquot was taken from this and analyzed. After analysis the alloquot was returned to the sample and remixed before a new alloquot was taken for analysis. This process allowed an average count to be calculated for each sample or replicate. Replicates are important for turbulence in the sample will present particles to the laser beam in a random process. With repetitive analysis each particle had the same chance of being counted as any other particle.

PRECISION

Precision of this optical method appears to be very good. Replicate samples agree closely with one another (see Table 1). Replicates were taken as separate 20 ml alloquots from the same sample cylinder as the pipette measurements were taken from. The variation between these replicates is less than 5%. Early work to determine the best method of utilizing the equipment showed that the channel to channel reproducibility was good even when total numbers of particles per millimeter varied by an order of magnitude at low concentrations. Precision for alloquots of the final sample dilution is even better. The largest variation from the average count in these determinations was 2% (see Table 2).

Individual channel counts are converted to phi classes by addition of channels within a phi class and a linear interpolation of channels which fall across phi class boundaries. Channels were set to 4.2 micron intervals to 63 microns except channel 1 which ran from 3.9 microns to 4.2 microns. Channel 15 through 9 and 56.00% of channel 8 were 4 to 5 phi interval. The rest of channel 8 through channel 5 and 28.6% of channel 4 were in the 5 to 6 phi interval. The rest of channel 4 and channel 3 plus 14.3% of channel 2 were in the 6 to 7 phi interval.

Table 1. Replicates of sample 242711 and variation.

Channel	Replicate A	Replicate B	Variation
1	1.65 %	1.87 %	.22 %
2	20.91	25.38	4.47
3	18.73	21.11	2.38
4	14.82	14.58	.24
5	11.06	10.27	.79
6	8.26	7.01	1.25
7	6.06	5.26	.80
8	4.87	3.97	.90
9	3.46	2.93	.53
10	2.72	2.11	.61
11	2.08	1.58	.50
12	1.77	1.30	.47
13	1.40	1.05	.35
14	1.18	.88	.30
15	1.05	.72	.32

Table 2. Sample count averages and greatestvariation from mean.

Sample	2536 B		2637 D		2637 F	
Channel	Avg. Count	Variation	Avg. Count	Variation	Avg. Count	Variation
1	1.31 %	.35 %	1.39 %	.29 %	1.39 %	.21 %
2	14.12	1.03	15.81	1.39	15.18	1.25
3	13.62	1.92	14.70	.62	14.26	1.3
4	12.25	.1.50	13.4	.68	12.90	.29
5	11.00	.89	13.02	.72	12.04	.46
6	9.68	.62	11.09	.87	10.76	.58
7	8.75	.64	8.75	.87	9.11	.62
8	7.34	.44	6.57	.69	7.27	.76
9	5.57	.72	4.28	.41	4.93	.50
10	4.63	1.01	3.20	.28	3.53	.40
11	3.51	.98	2.23	.21	2.65	.25
12	2.75	.92	1.89	.24	2.09	.38
13	2.10	.72	1.49	.21	1.59	.21
14	1.73	.57	1.14	.11	1.25	.29
15	1.54	.42	1.04	.11	1.09	.24

Table 3. Particle percentage by Phi size of alloquots of sample 2536 B.

Φ Size	1	2	3	4	5	6	Mean	Variation
4-5 Φ	31.28	22.23	23.86	24.59	29.61	24.40	26.00	5.25
5-6 Φ	34.97	36.56	36.67	37.18	34.39	37.51	36.21	1.82
6-7 Φ	21.25	26.29	26.16	25.08	22.99	24.52	24.38	3.13
7-8 Φ	12.50	14.92	13.31	13.15	13.01	13.57	13.41	1.51

Table 4. Comparison of weight % from pipette with weight % from particle counter with total percentage.

Sample	2637 F		2637 G		2535 J	
φ Size	Pipette	Counter	Pipette	Counter	Pipette	Counter
4-5 φ	62.50	66.11	60.00	59.28	48.48	47.77
5-6 φ	19.64	22.10	21.54	19.70	28.28	21.16
6-7 φ	12.50	11.03	12.31	12.62	13.13	13.13
7-8 φ	5.36	6.19	6.15	7.37	10.10	7.37
Total %	100.00	105.43	100.00	98.97	99.99	89.20

The rest of channel 2 and all of channel 1 were the 7 to 8 phi interval. An Example of the phi class data is given in Table 3. Variation from the mean for phi classes was less than 6%.

Accurate correlation of the particle phi size percent and the weight phi size percent as determined by pipette analysis is difficult. The two methods measure different properties. The laser unit measures particle size only. Pipette analysis measures the hydraulic equivalent size of the particle. This hydraulic equivalent size can depend on the physical size, shape, roundness, and density of the particles and also the density and viscosity of the liquid. The Measurement of the methods is also different. The laser gives a numeric count and the pipette method gives weight counts. This leads to a complex relation between the two methods which is difficult to predict. A relation can be found empirically by the ratios of particle size percent and weight percent but this ratio varies with the sample. Samples which are composed of similar minerals and size distribution can be correlated but this correlation will not be accurate for dissimilar samples. The correlation ratio used in Table 4 was obtained from the 2637 series of samples. This was 3.12 for 4-5 phi, 0.57 for 5-6 phi, 0.43 for 6-7 phi, and .043 for 7-8 phi. Table 4 shows the weight percentage by pipette analysis compared to the weight percentage predicted by the particle size distribution measured by the laser. Samples 2537 F and 2637 G are from the same area and of the same sediment. Sample 2535 J is from a different area and sediment type. The two related samples are close to the weight percent and total weight as measured by pipette. Sample 2535 J has some rather large discrepancies in phi size as well as a 10.8% under estimate of the total weight.

DISCUSSION

As this Particle counter was intended to count particulate impurities in liquids being manufactured it is very sensitive to low concentrations of particles in the size range it is set for. This would seem to make it ideal for measuring particle size and distribution in very dilute suspensions. Samples of open ocean water could be measured for the size distribution and the total particle density of suspended particulate matter without altering the sample in any way. The most that would need to be done to such a sample would be dilution in the event that it contained more than 999 particles per millimeter. Preparation time would be very small and the analysis would take less than 10 minutes. Perhaps an estimate of flocculation could be obtained by measuring the size distribution of a sample and then adding a peptizing agent and measuring the distribution again.

Optical size analysis is also applicable to the testing methods used in this report. Analysis of silt size particles with this machine would be very rapid compared to pipette analysis. The problem would be to set up a conversion factor between particle number percent and weight percent so that the results of a total sediment analysis would be in comparable units. Of course if particle numbers rather than weight percent were desired then this problem would disappear. This would also be the case when silt size particles were the only particles of interest in a sample.

Particle numbers rather than weight percent have many advantages, as statistical theory is based on numerical frequencies and should not be applied to weight percent frequencies (Blatt, Middleton, and Murray, 1972). Also, the randomness of the method is appropriate for statistical treatment. Each particle in a sample has an equal chance of being measured and counted as any other particle in the sample. This would be important when working with very small representative samples which were related in some way, for example a varve series from a lake or flooded area or the fore-set beds of the delta.

CONCLUSIONS

This method should be considered when particle numbers are needed or when a large quantity of similar samples need to have the silt fraction determined in phi intervals. However the need for a calculation of the conversion ratio from numbers to weight percent would rule this out for small groups of samples. The method would need very little extra work to be useful for work with suspended particulate matter.

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