

REVIEW OF PARTICLE SIZE DISTRIBUTION ANALYSIS METHODS

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INTRODUCTION

Particle size distribution analysis (PSDA) is a measurement of the size distribution of individual soil/sediment particles, sand, silt and clay, which can be used to understand soil genesis, to classify soil or to define texture (Soil Survey Staff, 1993). The USDA classification of soil texture is based on the proportion of sand 2.0-0.05 mm, silt 0.05-0.002 mm and clay < 0.002 mm particles (Table 1). Geologists and sedimentologists have been on the forefront of analyzing marine and submerged sediments. Demas was one of the first to take a soil science approach to study the sediments of shallow coastal systems (Demas et al, 1996). A soil science approach to sediments would necessitate the classification of these subaqueous soils into a unified taxonomic system with the Natural Resource Conservation Service's (NRCS) terrestrial soil taxonomy (Soil Survey Staff, 1999). Thomas Reinsch of the NRCS' National Soil Survey Center explained that PSDA data will be collected for subaqueous soils in regards to existing policies and methods with the terrestrial soils (personal communication with Dr. Thomas Reinsch). PSDA is a major criterion used to describe these soils and their characteristics.

PSDA of subaqueous soils will be consistent with the terrestrial standards of the established NRCS methods. The current procedure accepted by the NRCS for PSDA is the pipette method (Gee and Bauder, 1986). The USDA uses this method because it is reproducible on many different types of soils (Soil Survey Staff, 1999). This procedure has been relatively unchanged in variations used since 1922 (Muller et. al., 2009). In contrast geologists and sedimentologists have begun to rely almost exclusively on the

instrumental methods which are more time and cost effective as well as more reproducible (Mudroch et al., 1997; Molinaroli et. al., 1999; Last and Smol, 2004; Muller et. al., 2009).

SUMMARY OF METHODS

The two classes of methods of determining the particle size distribution (PSD) of a given sample consist of classical and instrumental, both of which rely on physical segregation of particles followed by quantification by mass (Mudroch et al., 1997). Examples of classical methods are sieving and pipette methods. As previously mentioned, instrumental methods tend to be faster and more reproducible (Welch et. al., 1979; Mudroch et al., 1997). Examples of instrumental methods are optical determination of particles, electrical sensing zone or electroresistance particle counting (Coulter Counter), X-ray sedimentation (Sedigraph) and laser diffraction (Mudroch et al., 1997; Molinaroli et. al., 1999; Last and Smol, 2001; Goossens, 2008).

Optical determination or direct measurement of particles is one of the oldest methods, where each particle is directly measured with calipers or through magnified digitized photos (Last and Smol, 2004). This process is time consuming and requires a great deal of particles counted to reach desirable confidence intervals (Last and Smol, 2004). Electroresistance particle counters were originally designed to count blood cells, but have been used in earth sciences enough to be established in the American Society of Testing Materials (Last and Smol, 2004). They measure particles on the basis of electrical resistance. When particles pass between electrodes, the machine records the resistance, which is proportional to particle size (Mudroch et al., 1997; Molinaroli et. al., 1999; Last and Smol, 2004). They are popular because they can analyze samples rather quickly, approximately 10-100 seconds per sample and can be used for small quantities of sample; drawbacks are the measuring tube tends to clog and there is no way to know

if more than one particle is passing through the electrodes (Mudroch et al., 1997; Last and Smol, 2004). X-ray sedimentation and laser diffraction particle size instruments are based on sedimentation rates and Stoke's Law, as is the pipette method (Mudroch et al., 1997; Molinaroli et. al., 1999). X-ray sedimentation measures the density of the suspension with a cumulative curve of the percentage mass of the silt and clay size fraction versus the logarithm of equivalent diameter (Buchan et. al., 1993). This technique is quick and reproducible, but is limited to the fine particles, generally <63 μm , and may be influenced by the different densities of particles (Buchan et. al., 1993; Last and Smol, 2004; Muller, 2009). Laser diffraction spectroscopy uses the same basic principle as the X-ray, but instead uses the intensity of the light scattered by the particle to determine its size (Mudroch et. al., 1997, McCave et. al., 2006; Taubner et al., 2009). All of these methods have distinct advantages and disadvantages and no one procedure gives exact results due to the nature and definition of soil particle size (Mudroch et al., 1997; Molinaroli et al. 2000; Last and Smol, 2004; Goossens, 2008).

With the pipette method, samples are usually pre-treated to avoid interferences, by flocculation and aggregates (Welch et. al., 1979; Mudroch et al., 1997). Standard pretreatment and dispersion procedure outlined in the Soil Survey Laboratory Methods Manual is to remove organic matter, carbonates, iron, silica, and to disperse soil aggregates (Soil Survey Staff, 2004). These pre-treatment methods were designed for the use in terrestrial soils and may not be suitable for some of the subaqueous soils. For instance one of the pre-treatment procedures outlined in the Soil Survey Laboratory Methods Manual is to remove carbonates (Soil Survey Staff, 2004). This is not appropriate in soils where the majority of the parent material is composed of

carbonaceous material, such as many of our South Florida Subaqueous Soils. The importance of the appropriate pre-treatment is as important as the technique for measuring PSD (Vassma, 2008).

ORGANIC MATTER REMOVAL AS A PRE-TREATMENT

To determine PSD of soil, the organic matter is removed as to not interfere with the mineral components of the sample. There are some associated challenges with the removal of organic matter depending on the type of pre-treatment and on the sample type. For instance exfoliation of mica, dissolution of manganese dioxide, dissolution of carbonates, dissolution of iron and aluminum and the formation of artifacts such as calcium, aluminum and ferric oxalate which can bind particles together (Gee and Bauder, 1986, Mikhail and Briner, 1978, Anderson, 1961). The most common pre-treatment methods for the removal of organic matter is oxidation. Four frequently used methods of oxidizing organic matter are achieved using hydrogen peroxide (H_2O_2), sodium hypochlorite ($NaOCl$), disodium peroxdisulfate ($Na_2S_2O_8$), and by combustion or loss on ignition (LOI); each of which have associated problems if to be used for PSDA. Mikutta et al. (2005) reviewed the use and results of these reactants with the mineral and organic constituents of soils, and many procedures have been documented depending on the nature of the soil as well as the determination to be run on the sample.

Of the four treatment types removal of organic matter by LOI and $Na_2S_2O_8$ are not typically used for textural analysis. LOI removes the organic matter through combustion, is quick and effective, but also partially removes carbonates, and damages and aggregates small particles (Vassma, 2008). Disodium peroxdisulfate can take as little as 16 hours for removal of 93% of the organic content, but has been reported that after a 2 day reaction time organic carbon was not sufficiently removed (Mikutta et. al.,

2005). The use of disodium peroxodisulfate has little to no effect on the structural components of Ca and Mn containing minerals, which eliminates the concern of forming oxalates, though is not practical for PSDA because of the large amounts of reactant needed and the variable reaction time (Mikutta et. al., 2005).

The use of H_2O_2 to remove organic matter is the NRCS standard pretreatment for PSDA (Gee and Bauder, 1986). The reaction of H_2O_2 can be highly variable depending on many different factors; for instance the structure of the organic compound, pH, and concentration of the solution (Mikutta et. al., 2005). At a pH of 9-10 the reaction only removes 5-20% of organic carbon, but 50-90% at a pH of 6 and 7.5 (Mikutta et. al., 2005). It takes a considerable amount of time sometimes several weeks for H_2O_2 to oxidize the organic matter. H_2O_2 also reacts with Ca forming oxalates, leaving residual carbon which can complex with the surface of other minerals to form silt sized particles (Anderson, 1961, Mikutta et. al., 2005). Hydrogen peroxide has also been shown to exfoliate and weather mica, vermiculite, and biotite through the destruction of Mn oxides (Mikutta et. al., 2005).

NaOCl is more efficient in the destruction of organic matter and does not form oxalates as readily as with H_2O_2 . This procedure using NaOCl was proposed by Anderson (1961) for mineralogical analysis. The sample is heated for 15 minutes with NaOCl then centrifuged at 2000 rpm for a total of three treatments (Anderson, 1961). It is limited to 15 minute intervals because of the relatively fast decomposition of NaOCl at high temperatures (Anderson, 1961; Mikutta et. al., 2005). Sodium hypochlorite at a pH of 8-9.5 has been used for mineralogical analysis because of the relatively fast reaction times and less affected by the presence of carbonates (Mikutta et. al., 2005; Mikhail and

Briner, 1978). It does not dissolve Mn oxides and Fe and Al as readily as with H_2O_2 which eliminates the concern of forming silt sized particles in the sample (Mikutta et. al., 2005). NaOCl also acts as a dispersing agent which eliminates the use and extra step of adding $(\text{NaPO}_3)_6$ typically added when using H_2O_2 as an oxidant (Omueti, 1980).

There has been considerable work on the abundance of analysis techniques and the methods for pretreatments on a diversity of minerals and physically different soils and sediments. There are also recommendations on what the best and most efficient procedure for the removal of organic matter for PSDA. With this it is my opinion that sodium hypochlorite is the most efficient and least detrimental option for the removal of organic matter in subaqueous soils for PSDA. However there are concerns that have arisen with the use of NaOCl.

PROPOSAL FOR RESEARCH

Introduction

In preliminary studies I performed in the University of Florida Soil and Water Science Department Environmental Pedology Laboratory, most samples required more than required spinning which may mechanically form clay sized particles (Discussions with Graduate Committee). The procedure also calls to decant the supernatant after each treatment. It was found that the supernatant is rarely clear after the first two treatments which could imply the suspension of clay and silt sized particles in the supernatant.

In these studies the majority of the concerns arose from the Key Largo, FL subaqueous soils. These soils mainly consist of calcium carbonate parent materials. The fine-grained soils are mainly calcium carbonate in the form of aragonite and calcite (Figure 1). In the removal of OM through NaOCl and centrifuging there is concern of forming clay sized particles through physical abrasion from coarser fragments of shell and coral. When the fine-grained soils (silt + clay) are spun the supernatant is rarely clear after centrifuging at 2000 rpms for 5 minutes as recommended (Anderson, 1961). The first order of business is finding the correct spin speed and time to consistently get a clear supernatant on these soils. With increased speeds and time intervals in conjunction with the presence of coarse shell and coral fragments physical destruction and abrasion may form smaller sized particles skewing the results of PSDA, showing incorrect clay and silt fractions numbers.

Aragonite and calcite are the two crystallographic forms of calcium carbonate. Aragonite, a polymorph of calcite, has an orthorhombic structure while calcite is trigonal. Unlike calcite, aragonite's carbonate ions lie in two planes pointing in opposite directions where as calcite, the carbonate ions lie on a single plane pointing in the same direction. Aragonite is considered unstable at normal surface temperatures and pressures and will spontaneously convert to calcite at 400 degrees C. Aragonite will still preferentially form if conditions are right, such as the magnesium and salt content and turbidity of the crystallizing fluid. Most bivalves, corals and many sea creatures secrete aragonite for their shells laying them in several layers of aragonite. Most of Florida's subaqueous soils are dominated by quartz, though there are calcium carbonate rich soils in South Florida that are made up of the aragonite form of calcium carbonate.

Hypotheses and Objectives

Objective 1) To find the correct spin velocity and duration to obtain a clear supernatant for two South Florida subaqueous soil types; calcium carbonate rich and quartz dominated soils for the analysis of PSD.

Hypothesis 1) RPM and duration of speed will have to be increased in order to get a clear supernatant free of suspended particles.

Rationale: South Florida subaqueous soils tend to have significant amounts of fine textured (silt + clay) particles easily dispersed into the water column. At higher velocities the particles will flocculate more effectively leaving a clear supernatant and minimal suspension of soil material.

Objective 2) Compare the percent clay of two naturally occurring subaqueous soil types. I will determine if spin time and speed mechanically produce clay in subaqueous soils of quartz versus carbonaceous parent material.

Hypothesis 2) The carbonaceous soils will have an increase in percent clay after treatment, while the quartz soils will not be affected.

Rationale: Aragonite crystals preferentially form in the carbonaceous South Florida subaqueous soils. Aragonite is of orthorhombic structure forming acicular needles making aragonite less stable and more susceptible to comminution. Quartz is hard and characteristically spheroidal making it more physically stable mineral, therefore not being as affected.

Methods

Sites: We have two South Florida samples site where the samples will be taken from. One is from the Indian River Lagoon in Fort Pierce where quartz is the dominant mineral in the subaqueous soil. The other is from Lake Surprise in Key Largo dominated by calcium carbonate soil material. Both are representative of South Florida's subaqueous soils (Figure 2).

Procedure

We will be using a modified version of the procedure for moist soil samples, as well as the Pipette method from the NRCS Soil Survey Laboratory Methods Manual. The modifications are the use of NaOCl instead of Hydrogen Peroxide to avoid the creation

of oxalates. Both soil types will be centrifuged at different velocities and time durations until a clear supernatant can be consistently produced. The spinning will begin at the current procedure recommendation of 2000 rpms for 5 minutes, then increased in time in five minute intervals to a maximum of 15 minutes and increase rpms in 1000 rpm intervals to a maximum of 10,000 rpms with each time duration or until we can consistently get a clear supernatant indicating there are no particles in suspension (Table 2-3 and Appendix).

Once the spin time and velocity have been found for the samples, run the procedure for PSDA, including treatment with sodium hypochlorite, shaking with calgon, etc. Then compare the percent silt and clay, as well as the silt to clay ratios of the subaqueous soils with differing parent materials, calcium carbonate and quartz. A total of 30 treatments will be analyzed for silt and clay fractions.

TABLES

Table 1: USDA Particle Size Separates. The experiment will focus on the clay fraction (<0.002 mm). (*USDA NRCS Soil Survey Laboratory Methods Manual, 2004*)

USDA Particle Size Separates	Size (mm)
Clay, total	<0.002
Silt, total	0.002-0.05
Silt, fine	0.002-0.02
Silt, coarse	0.02-0.05
Sand, total	0.05-2.00
Very fine sand	0.05-0.10
Fine sand	0.10-0.25
Medium sand	0.25-0.50
Coarse sand	0.50-1.00
Very coarse sand	1.00-2.00

Table 2: Outline of treatments

1. A control, where no pretreatments or spinning will be done; one of the calcium carbonate soil and one of the quartz soil done in triplicates.
2. Samples of quartz parent material at each rpm speed and time interval, done in triplicates.
3. Samples of calcium carbonate parent material at each rpm speed and time interval, done in triplicates.
4. All samples except for controls will be treated with calgon and NaOCl.

Table 3. Summary of spin velocities

	5 minutes			10 minutes			15 minutes		
2000 rpm	Q	Q	Q	Q	Q	Q	Q	Q	Q
	C	C	C	C	C	C	C	C	C
3000 rpm	Q	Q	Q	Q	Q	Q	Q	Q	Q
	C	C	C	C	C	C	C	C	C
4000 rpm	Q	Q	Q	Q	Q	Q	Q	Q	Q
	C	C	C	C	C	C	C	C	C
5000 rpm	Q	Q	Q	Q	Q	Q	Q	Q	Q
	C	C	C	C	C	C	C	C	C
6000 rpm	Q	Q	Q	Q	Q	Q	Q	Q	Q
	C	C	C	C	C	C	C	C	C
7000 rpm	Q	Q	Q	Q	Q	Q	Q	Q	Q
	C	C	C	C	C	C	C	C	C
8000 rpm	Q	Q	Q	Q	Q	Q	Q	Q	Q
	C	C	C	C	C	C	C	C	C
9000 rpm	Q	Q	Q	Q	Q	Q	Q	Q	Q
	C	C	C	C	C	C	C	C	C
10000 rpm	Q	Q	Q	Q	Q	Q	Q	Q	Q
	C	C	C	C	C	C	C	C	C
Control (No Treatments)	Q	Q	Q	Q	Q	Q	Q	Q	Q
	C	C	C	C	C	C	C	C	C
Q	Quartz Soil								
C	Calcium Carbonate Soil								

FIGURES

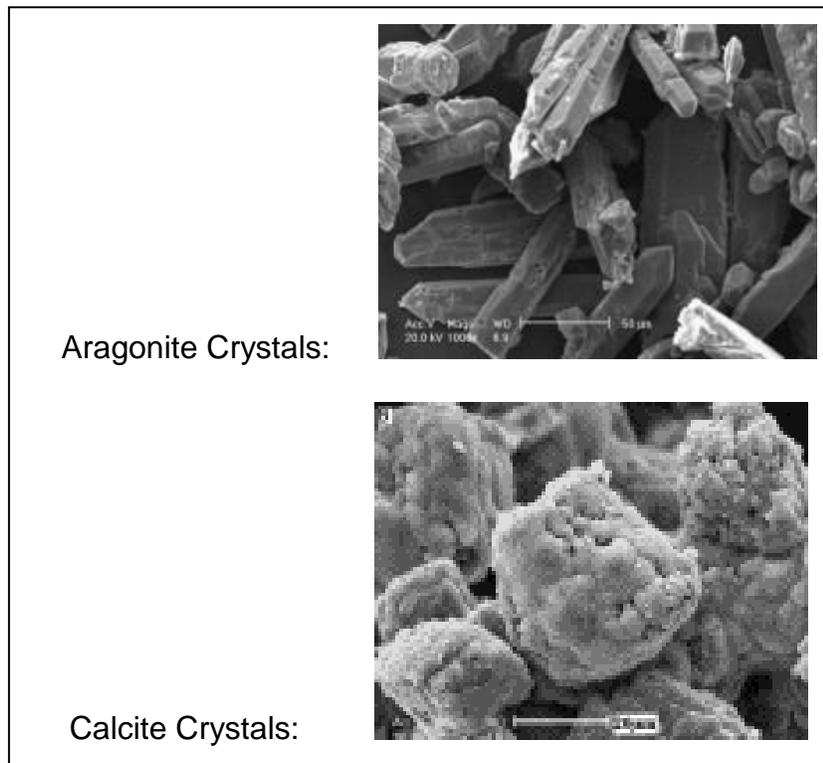


Figure 1: SEM photograph of Aragonite and Calcite Crystals (*Unknown Source*)

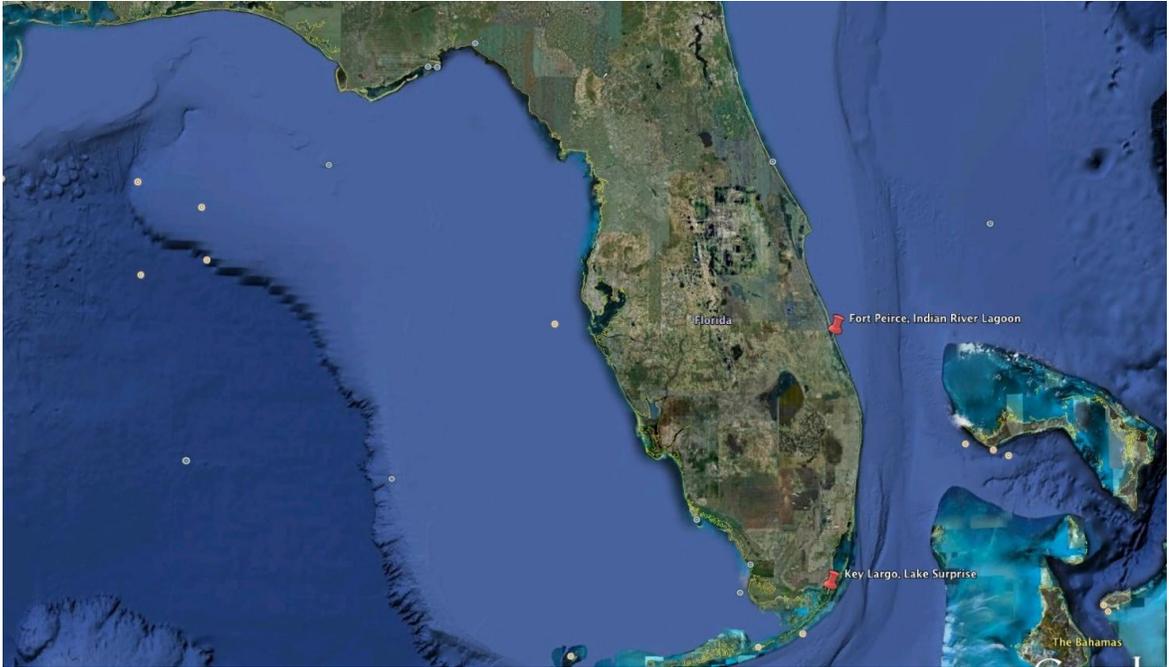


Figure 2: Locations of sample sites in South Florida. The two red pushpins indicate where the samples will be taken. (from *Google Earth 2009*).

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APPENDIX

Particles < 2mm (Pipet Method)

Reagents

Hydrogen peroxide (H₂O₂), 30 to 35 percent.

Sodium hexametaphosphate (NaPO₃)₆. Dissolve 35.7 grams of (NaPO₃)₆ and 7.94 grams of Na₂CO₃ per liter of water.

Demineralized water.

Procedure

Removing organic matter.--Place about 10 air-dry soil containing no particles larger than 2 mm in a tared Fleaker. Add about 50-ml of demineralized water (referred to subsequently as water) and then add 5 ml of H₂O₂. Cover the fleaker with a watchglass. If a violent reaction occurs, repeat the cold H₂O₂ treatment periodically until no more frothing occurs. Heat the Fleaker to about 90°C on an electric hot plate. Add H₂O₂ in 5-ml quantities at 45-min intervals until the organic matter is destroyed, as determined visually. Continue heating for about 30 min to remove any excess H₂O₂.

Removing cementing agents (optional).--Treat the sample with about 200 ml of 1 N sodium acetate buffered at pH 5 to remove carbonates. When CO₂ bubbles are no

longer evident, wash free of salts with a filter candle system. Highly calcareous samples may need a second treatment. Remove siliceous cementing agents by soaking the sample overnight in 0.1 N NaOH. Iron oxide cementing agents are removed by shaking overnight in sodium dithionite (6C2). Wash free of salts with filter candle system before proceeding.

Removing dissolved mineral and organic components.--After the H₂O₂ treatment, place the Fleaker in a rack and add about 150 ml of water in a jet strong enough a short Pasteur-Chamberlain filter of "F" fineness. Five such washings and filterings are usually enough except for soils containing much coarse gypsum. Remove soil adhering to the filter by gentle back pressure; use finger as policeman. Dry the sample overnight in an oven at 105°C, cool in a desiccator, and weigh to the nearest milligram. Use the weight of the oven-dry, H₂O₂-treated sample as the base weight for calculating percentages of the various fractions.

Dispersing the sample.--Add 10 ml of sodium hexametaphosphate dispersing agent to the Fleaker containing oven-dry treated sample. Make the volume to approximately 200 ml. Stopper and shake overnight on a horizontal reciprocating shaker at 120 oscillations per minute.

Separating sands from silt and clay.--Wash the dispersed sample with water on a 300-mesh sieve. Silt and clay pass through the sieve into a 1-L cylinder. Use a clamp and stand to hold the sieve above the cylinder. Avoid using jets of water in washing the

sample. Gently tap the sieve clamp with the side of the hand to facilitate sieving. Continue washing until the suspension volume in the cylinder is about 800 ml. Sand and some coarse silt remain on the sieve. It is important to wash all particles of less than 20μ diameter through the sieve. Remove the sieve from the holder, wash the sands into an evaporating dish with water, and dry at 105 to 110°C . Bring the silt and clay suspension in the cylinder to 1 L with water and cover with a watchglass.

Pipeting.--First pipet the $<20\mu$ fraction at a 10-cm depth. Vary sedimentation times according to temperature. Next, pipet the $<2\mu$ fraction after a predetermined setting time (usually $4\frac{1}{2}$ to $6\frac{1}{2}$ hr). Vary depth according to time and temperature. Use a Lowy 25-ml automatic pipet and regulate filling time to about 12 s. Before each pipeting, stir material in the sedimentation cylinder, and stir the suspension for 30 s with a hand stirrer, using an up-and-down motion. Note the time at completion of stirring. About 1 min before sedimentation is complete, lower the tip of the pipet slowly into the suspension to the proper depth with a Shaw pipet rack. At the appropriate time, fill the pipet and empty into a 90-ml, wide-mouth bottle. Rinse the pipet into the bottle once. Dry in an oven overnight at 105°C . Cool in a desiccator containing phosphorus pentoxide (P_2O_5). Weigh.

Sieving and weighing the sand fractions.--Transfer the dried sands to a nest of sieves. Shake for 3 min on a shaker that has $\frac{1}{2}$ -in vertical and lateral movements and oscillates at 500 strokes per minute. Record the weights of the individual sand fractions.

Calculations

Pipetted fractions:

Percentage of pipetted fractions = $(A - B)KD$

where

A = Weight (g) of pipetted fraction

B = Weight correction for dispersing agent (g)

K = $1000/(\text{ml in pipet})$

D = $100/(\text{g of H}_2\text{O}_2\text{-treated oven-dry total sample})$

The $<20\text{-}\mu$ fraction minus the $<2\text{-}\mu$ fraction equals fine silt.

Sand fractions: Percentage of sieved fractions = weight (g) of fraction on sieve times D.

Coarse silt fraction: Obtain by difference. Subtract the sum of the percentages of sand plus the $<20\text{-}\mu$ fraction from 100.

References

Kilmer and Alexander (1949), Kilmer and Mullins (1954), Tyner (1939), and Grossman and Millet (1961).

Moist Samples (3A2)

If drying affects dispersion of treated sample, over-drying may be avoided by removal of a pipet sample to estimate the total weight of the sample. Pipet 50 ml at a depth of 20 cm at time zero while the suspension is still turbulent. Use the over-dry weight of the aliquot to calculate the total weight of the <0.05-mm fraction. Add this weight to the total weight of the sands to obtain the total weight of the sample.

An optional procedure is to carefully weigh out two identical samples and pretreat to remove organic matter and dissolved mineral matter. The first sample is continued through the standard procedure, excluding over-drying. The second sample is over-dried, weighed, and discarded. The over-dry weight of the second sample is substituted in the calculations for the first sample.