



METHOD DEVELOPMENT FOR WIDE SIZE-DISTRIBUTION SAMPLES

Measurement of wide size distribution materials presents a number of challenges to proper analysis. Sampling from the bulk material, sample preparation, and analysis are all affected by the tendency of this type of sample to segregate based on size during transport or storage. The different size classes may also each contain noticeably different composition. Laser diffraction particle size analyzers are a fast, accurate, and useful tool, but care must be taken in developing the analytical method to ensure that the correct results can be generated.

Sizing Soils & Sediments

Accurate particle size analysis is obviously impossible if the appropriate sampling and preparation steps are not taken, but there are additional difficulties with the measurement itself because of wide size range samples.

A good example of these difficulties is the study of soils and sediments. Soils and sediments are the subject of increasing numbers of agricultural and environmental studies. The particle size distribution (PSD) is one of the key characteristics for these materials both as a measure of substrate type and as an important determinant of the benthic community structure.

Soils and sediments differ greatly in their textures, which are determined by the compositions of sand, silt and clay. Clay is consisted of very fine particles that are less than 2 μ m and its texture ranges from powdery (when dry) to very sticky or slippery (when wet). Silt, on the other hand, usually contains particles with size ranging from 2 to 50 μ m and tends to feel smooth and silky like wet talcum powder or flour. Sands are large granular particles with sizes ranging from 50 to 2000 μ m, are gritty in texture and not slippery even when wet. Texture is one of the key characteristics commonly used to characterize and differentiate soil and sediment samples.

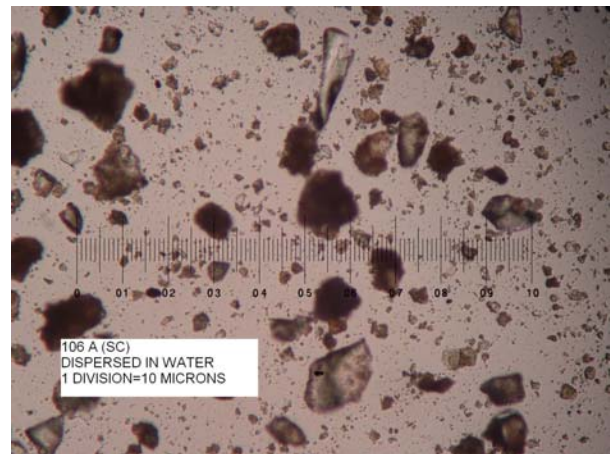


Image of soil sample showing wide range of sizes

Measurement Methods

A range of common methods are being used to measure the particle size distribution of soil and sediment samples. While samples with large particles such as sands and gravels are measured with the popular sieving method, the silt and clay fractions are often measured with pipettes (Plumb 181), hydrometers, or laser diffraction particle size analyzers.

The difficulty in measurement is mainly due to the complex nature of these samples, especially the very broad particle size distribution that can easily range over five orders of magnitude (typically from 0.1 to 1000 μ m). The increasing measurement range of laser diffraction analyzers has led to their greater use,



but success has a number of challenges that must be addressed for proper results.

Instruments such as Horiba's LA-930 offer a wide measurement range of 0.02-2000 μm , fast analysis times allowing a large number of samples to be run, and a high resolution of the sample distribution, particularly when compared to traditional methods such as sieves.



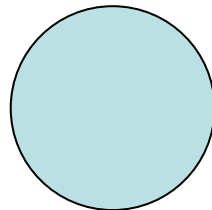
LA-930 Particle Size Analyzer

Importance of Sampling

The first challenge is to obtain and handle representative samples on a consistent basis. While the former is largely dependent on the way samples are collected in the field, the latter requires proper handling of samples in the lab.



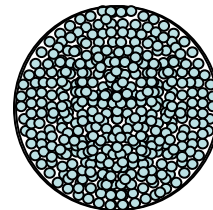
Diameter = 3
Volume = 27



1.0 μm



0.1 μm



The volume of 1.0 μm filled with 1000 0.1 μm particles

Numerous studies have been conducted on this subject (Particle Size Measurement, Terence Allen, Chapman & Hall, 1990) showing that errors as large as 14% can be attributed to this step alone. For example, handling errors during sample transfer from their containers to the particle sizing instruments can cause additional variations and may produce misleading results. Proper sample handling protocol must be established and strictly followed.

Limitations of Laser Diffraction

The second challenge is the intrinsic limitation of laser-diffraction based particle sizing instruments to measure a small number of particles. To reliably determine particle size distribution, a minimum number of particles for each size class are needed inside the measurement cell.

Since volume is proportional to the cube of particle diameter, large particles tend to take up a large volume percent in the mix even when their number is very small.

For example, the volume of one 1000 μm particle is equivalent to one thousand 100 μm particles or a billion 1 μm particles. Thus, to supply the minimum number of large particles needed for proper measurement, when mixed with a range of other sizes, the total sample concentration may have to be increased above the normally recommended range.

**Volume vs. Weight**

The last challenge is the potential confusion between weight percentage and volume percentage and conversion between them. While most people like to use weight percentage in discussing mixed samples, the laser-based particle sizing instruments actually detect and report PSD based on the particle volume. Sieve and sedimentation techniques rely on actually weighing the sample in different size fractions, thus the results are reported as weights.

Furthermore, to convert between weight percentage and volume percentage, one needs to know the densities of the material involved. These wide distribution materials are often mixtures of different types of materials. If all the particles have the same density, volume- and weight-based distributions are equal. However, when the particles have different densities, the conversion is not as straightforward.

For example, clay has a specific gravity around 1.2 and sand between 1.4 and 1.7. With a simple density adjustment, a sample with 70 wt% sand and 30 wt% clay actually contains only 55 vol% of sand. For someone who overlooks the difference, it may seem that the instrument under-reports large particles, when in fact it is reporting the correct result, only in different units.

Developing Methods

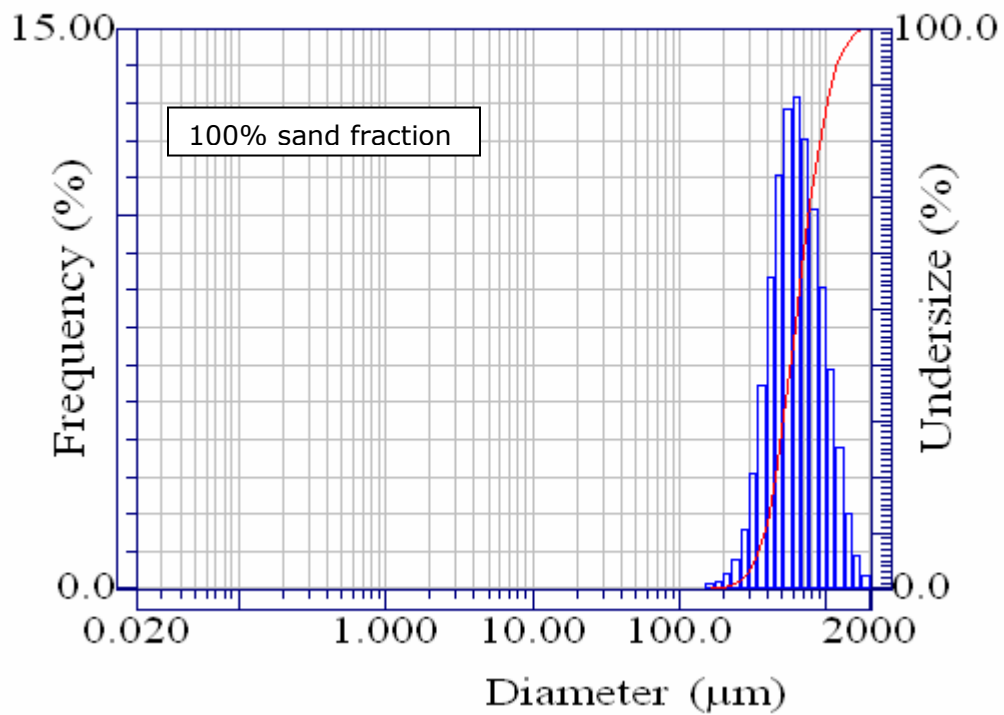
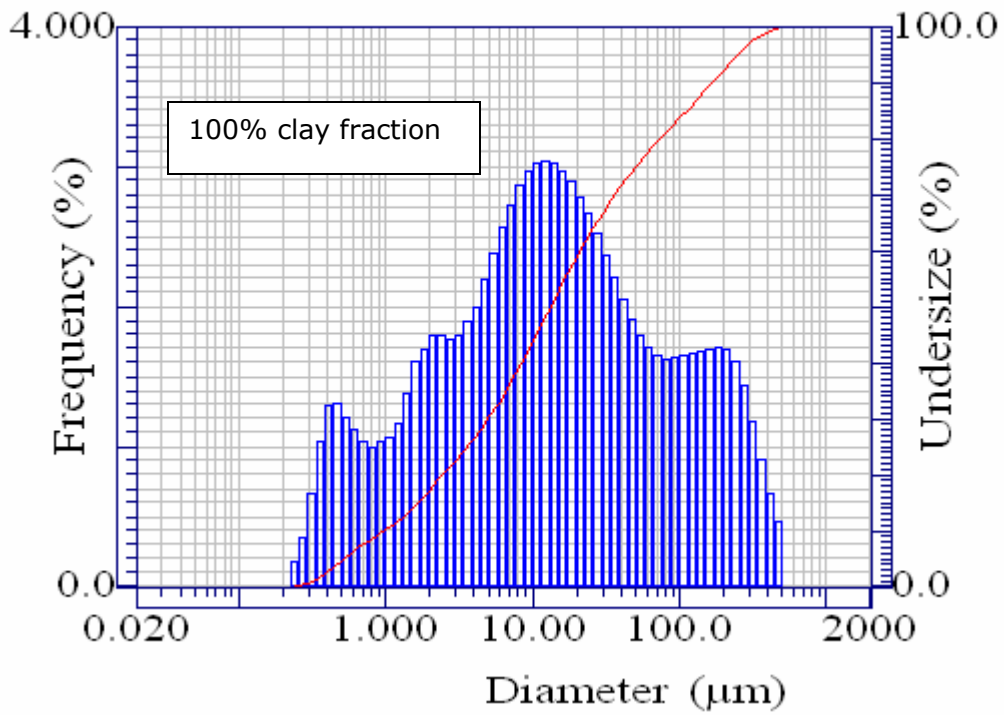
To develop a robust method for routine analyses of this type of wide size range material, tests must be carried out to determine the appropriate sample concentration for

accurate reporting of all the size components of the final mixture. The basic approach for a test to confirm the true distribution is this:

1. Split the sample with a sieve at a size range of interest.
2. Measure the weight and density of the separated samples.
3. Measure the size distribution of the separate samples with the analyzer.
4. Calculate the particle size distribution of the total sample using a simple linear combination model.
5. Compare to measurement results from the mixed sample.

Following is an example of using this method to obtain the actual particle size distribution of a mixed sample with silt, clay and sand. First, the mixed sample was separated by sifting it through a 300 μ m (50 mesh) sieve. The silt and clay passed through the sieve and was collected in the pan. The sands remained on the sieve. The relative weight ratio was 3:7. The separated sand and clay/silt samples as well as the mixed sample were measured with a Horiba LA-930 laser diffraction particle size analyzer.

For large particles like sands, a minimum sample weight is usually required (known as "wt1") to supply enough particles in the flow system for a laser diffraction particle sizing instrument to detect. Here the "wt1" was determined by measuring the separated sand sample with incrementally increased sand concentration until a stable, accurate result was obtained.





When measuring the mixed sample, we first added "wt1" weight of sand into the flow system and then added clay by $wt2 = wt1 \times (30/70)$. By doing so, we not only kept the 3:7 weight ratio between clay and sand but also had enough sand particles in the flow system for the instrument to detect the large particles.

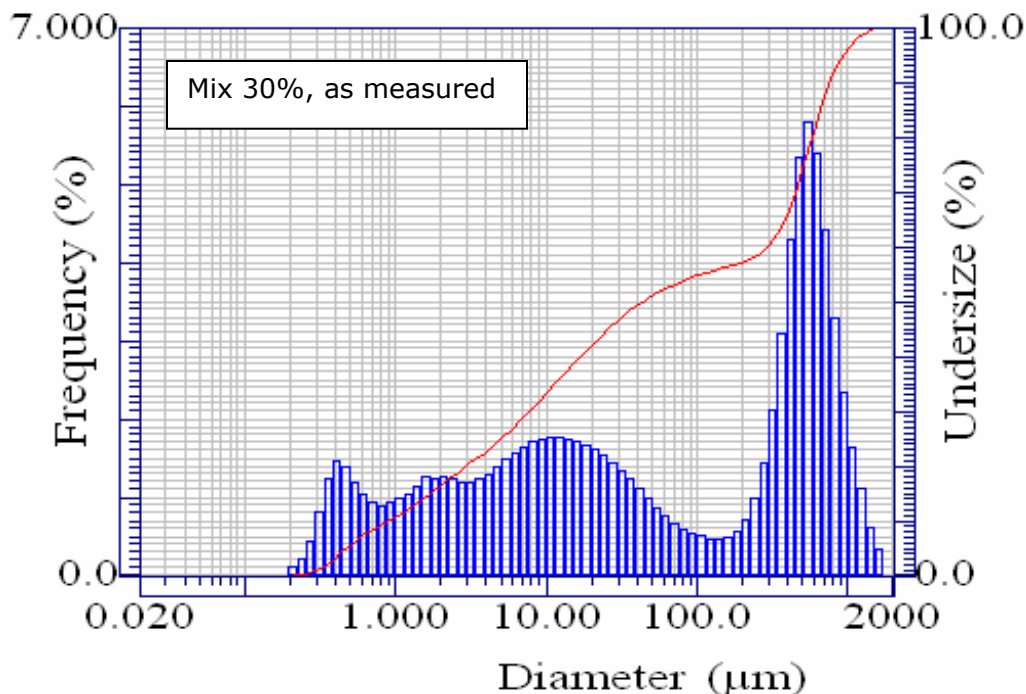
For this example the laser transmission (T%) was reduced to below 30%, well outside the default operating range of 70-95%. The requirements of the sample demand this approach and show that the normal operating procedures do not apply to such an unusual sample.

With such a high particle concentration in the measuring cell, multiple scattering does occur, which tends to over-report the smaller particles. The user must decide if this is an acceptable error, however small, in order to simplify the measurement procedure. Measuring this type of

sample complete, without splitting different fractions and combining results, will cause some error.

For the mixed sample (with 70 wt% sand), Horiba LA-930 reported 40 vol% of the sand particles. This seemingly large discrepancy between the known sand concentration and that reported by the instrument was due to the weight-to-volume conversion. Using the water displacement method, we measured the density of sand as 2.20 g/cc and that of clay as 1.21 g/cc.

With weight-to-volume conversion with these densities, the 70 wt% sand in mixed sample should be 56 vol%, while 40 vol% was reported by the Horiba LA-930. While this is not completely accurate, it shows that correcting for density can get us much closer to the correct answer. Part of the error can also be attributed to over-reporting of the fines due to multiple scattering.





The remaining error is at least partially due to the differing measurement methods. Particle shape has a significant effect on how each technique responds to a given sample. Sieving will allow non-spherical particles to pass on the second-smallest diameter, sedimentation reports on the smallest diameter due to settling on the highest hydrodynamic drag orientation, while laser diffraction measures a random orientation of the particles, averaging out all the different dimensions.

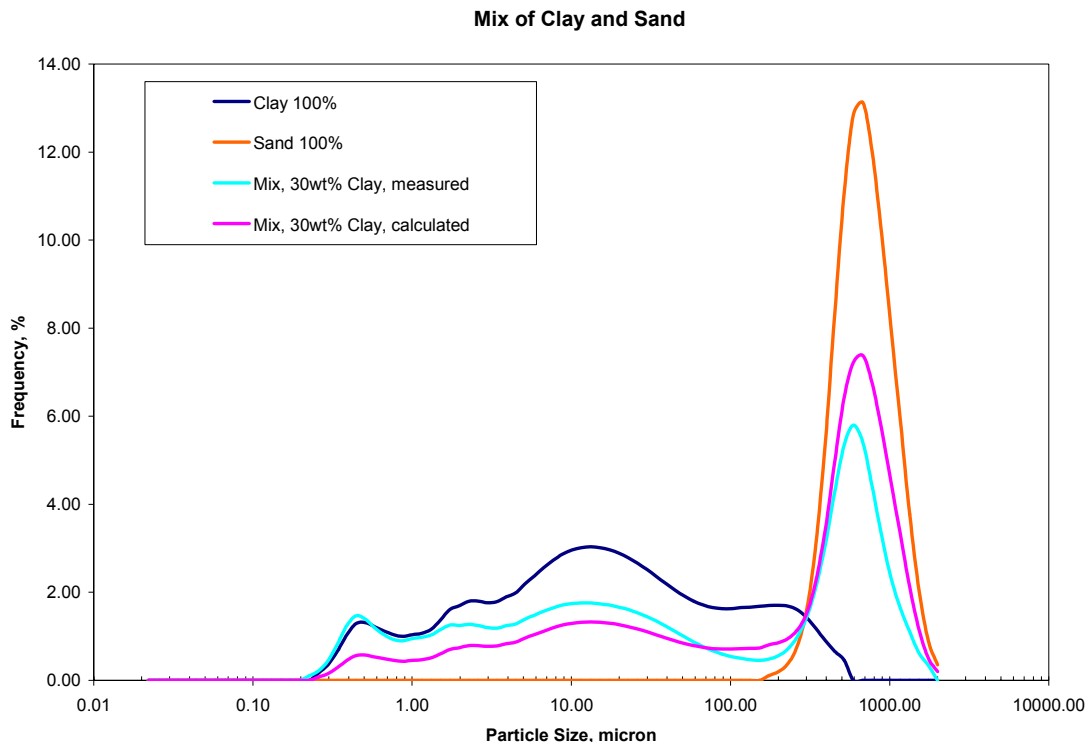
Measure Separately and Combine

An alternative approach is to measure the components separately and combine the data mathematically. The data for the individual components was exported into Excel. Then we used the following equation to calculate the distribution for the total sample.

$$\text{MIX} = (\text{volume\% of clay}) \times (\text{CLAY}) + (\text{volume\% of sand}) \times (\text{SAND}).$$

Where, MIX is the mix's differential percentage at a particular particle size, volume% of clay is the volume percentage of clay in the mix, CLAY is the differential percentage at the particular particle size, volume% of sand is the volume percentage of sand in the mix, and SAND is the sand differential percentage at this particular particle size.

This calculated data is very close to the real distribution of the mix. Because of the measurement difficulties of the high concentration required by this sample, this separated measurement may be more accurate because it allows for more appropriate measurement conditions for each component.





Summary

There is no one specific method that will work for all samples. Care must be taken to evaluate the sample of interest, or the range of samples that are expected to be measured and develop methods that are appropriate for such materials.

Current laser diffraction instruments are a fast, convenient method for size analysis, but their limitations must be kept in mind when looking at these difficult samples. These instruments work well within the standard

recommended optical concentration range (70-95% transmittance for the Horiba LA-930), but this is based on distributions that normally are only one or two orders of magnitude wide.

Samples that are unusually wide distributions must be treated more carefully and a robust method developed for the standard procedure. For the most accurate results, this may mean separating the different components, measuring separately, and combining with the appropriate corrections.

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