IOWA STATE UNIVERSITY

Department of Agronomy

MICHIGAN STATE UNIVERSITY

Department of Geography, Environment, & Spatial Sciences

Bradley A. Miller and Randall J. Schaetzl

Refining precision and quality control standards for analysis of loess by laser diffractometry

Introduction

Laser diffractometry (LD) is becoming increasingly utilized for determining the particle size characteristics of loess. A potential source of uncertainty and reduced replicability exists in the data due to the small amount of sample used in the analysis, such as with the Malvern Mastersizers we used in this study. Thus, users are challenged to thoroughly homogenize the sample, so that the small subsample analyzed contains a complete representation of the particle sizes in the larger sample. To investigate this issue, each sample in our library was analyzed a minimum of two times and the results statistically compared. Miller and Schaetzl (2012) used a similar approach to propose a method for measuring the precision of laser diffractometry. As a means of quality control, they used data for 1,485 loess samples to establish expected thresholds for the variability in the particle size measurements. The quality control protocol proposed by Miller and Schaetzl (2012) requires two runs to have a cumulative bin difference (CBD) less than the established threshold. We present additional conclusions on the variability of particle size analysis by LD, based on a larger library of replicate samples, so as to reassess expected sample variability. Further, we examine the expanded data set for a more specific assessment of precision.

Methods

Samples in our library, totaling 2,928, were all measured twice using Malvern Mastersizers (figure 1). To examine patterns in measurement variability by different types of particle size distributions, samples were grouped into soil texture class families as shown in table 1. The differences between the two measurements for each of the samples were summarized statistically to establish expected levels of measurement variability. In addition, select samples were analyzed by the pipette method for comparison.

Family	Texture Class
Sandy	sand, loamy sand
Silty	silt loam, silt
Loamy	loam, sandy loam, sandy clay loam, clay loam, silty clay loam
Clayey	clay, silty clay, sandy clay

Table 1. Grouping of soil texture classes into families.

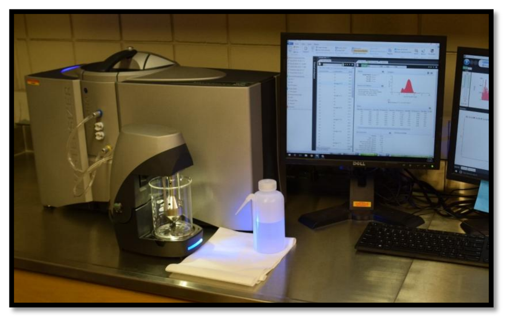


Figure 1. A Malvern Mastersizer 3000.

Results

The mean of differences across particle size ranges was used to summarize disparities between measurements. This metric is the easiest to interpret because it directly relates to how much measurement uncertainty can be expected. For determining tolerable levels of differences between runs, we recommend using the mean variability plus one standard deviation. In theory, measurements with differences greater than 84.1% of observed differences would then be flagged as outliers and in need of additional measurement to determine the most representative result. Grouping samples by texture class family and analyzing separately for measurement variability indicated a trend of finer textures having greater variability in results (figure 2).

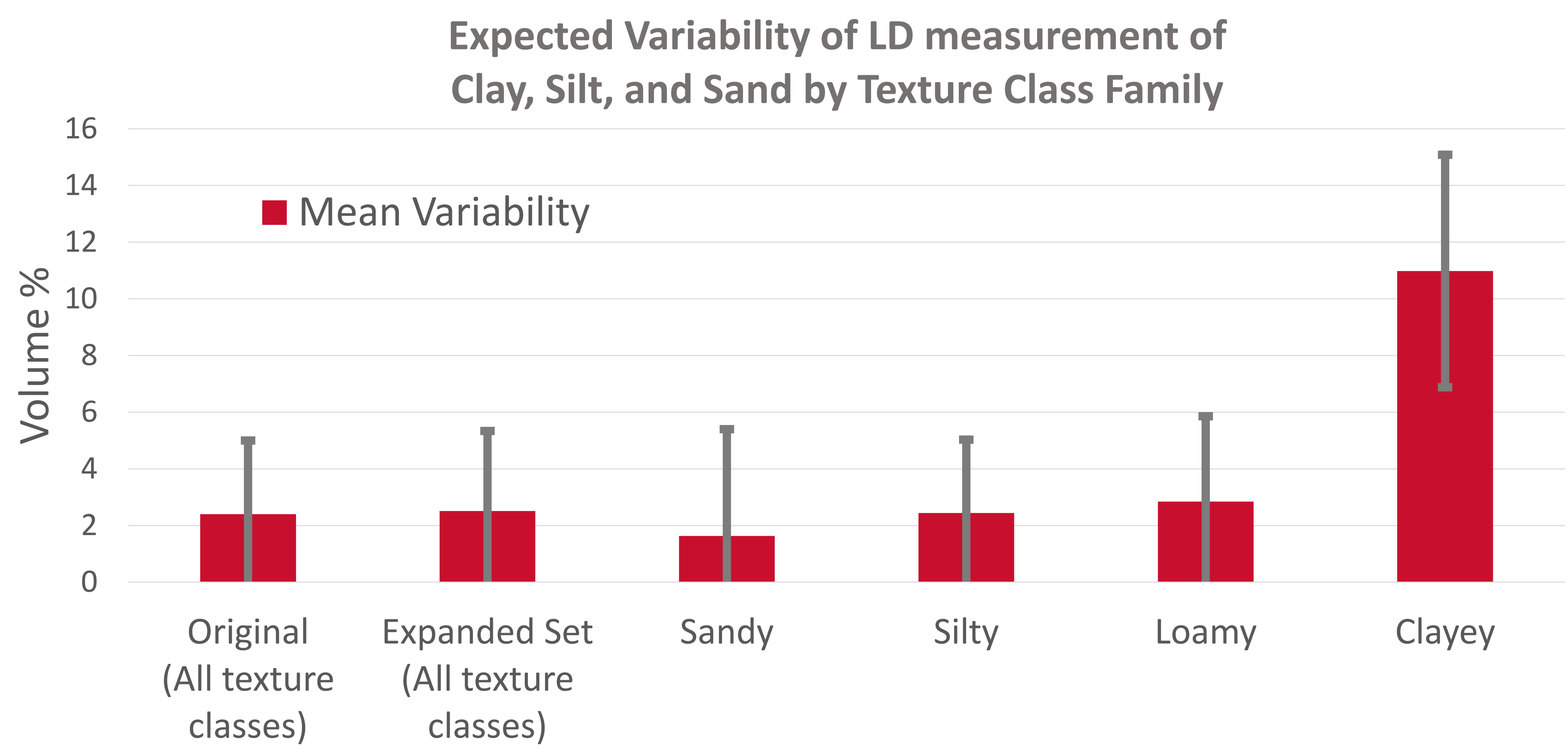


Figure 2. The mean of the mean differences across the clay, silt, and sand separates (3 bins) is the mean or expected variability. To describe the variability of the variability, the grey 'error' bars represent one standard deviation around the mean variability.

CBD		101 bin			7 bin			3 bin		
	n	Mean	SD	Outlier Threshold	Mean	SD	Outlier Threshold	Mean	SD	Outlier Threshold
Original	1485	10.60	9.20	19.80	9.50	8.50	18.00	7.30	7.80	15.10
Expanded Set	2928	11.06	9.76	20.82	10.07	9.17	19.24	7.54	8.47	16.01
Sandy	170	12.88	14.23	27.11	11.63	13.79	25.42	4.88	11.32	16.20
Silty	1955	9.87	8.47	18.34	8.96	8.07	17.02	7.31	7.77	15.09
Loamy	796	13.50	10.75	24.24	12.42	9.84	22.26	8.53	9.03	17.56
Clayey	7	52.88	24.84	77.72	34.23	13.18	47.41	32.94	12.31	45.25

Table 2. The cumulative bin difference (CBD) sums the differences between measurements across the particle size ranges. The 101-bin metric is for all the ranges measured by the Malvern Mastersizer 2000. The 7-bin and 3-bin metrics are the aggregation of those 101 bins into standard ranges of soil separates. CBD is more sensitive to differences in particle size distribution curves and thus better suited for quality control. Similar to figure 2, a trend of greater variability with finer textures is observed. However, sandy samples have a wider range of variability than silty and loamy samples. This deviation from the trend is likely due to the effect that a few sand grains can have on volume percentages, particularly for bimodal samples.

Comparison with Pipette Method

The pipette method is generally considered to be the standard for measuring particle size distributions, especially for soil. To test the repeatability of soil texture class determination, we selected a set of 71 samples covering a spectrum across the texture triangle. <u>Two separate labs proficient in using the pipette method agreed on the texture class of these 71 samples 59% of the time.</u>

One of the major concerns in comparing LD with the pipette method is the underestimation of clay content. The general solution applied has been to shift the clay-silt threshold upwards to make LD results more compatible with pipette method results. Using our systematic distribution of samples, we compared the effect of shifting that threshold on the LD results towards making the texture class determination match the results of the two independent pipette method laboratories. Results indicate that shifting that threshold from the usual 6 or 8 µm to 10 µm increased the agreement with the soil texture classifications made by the pipette method laboratories (table 3).

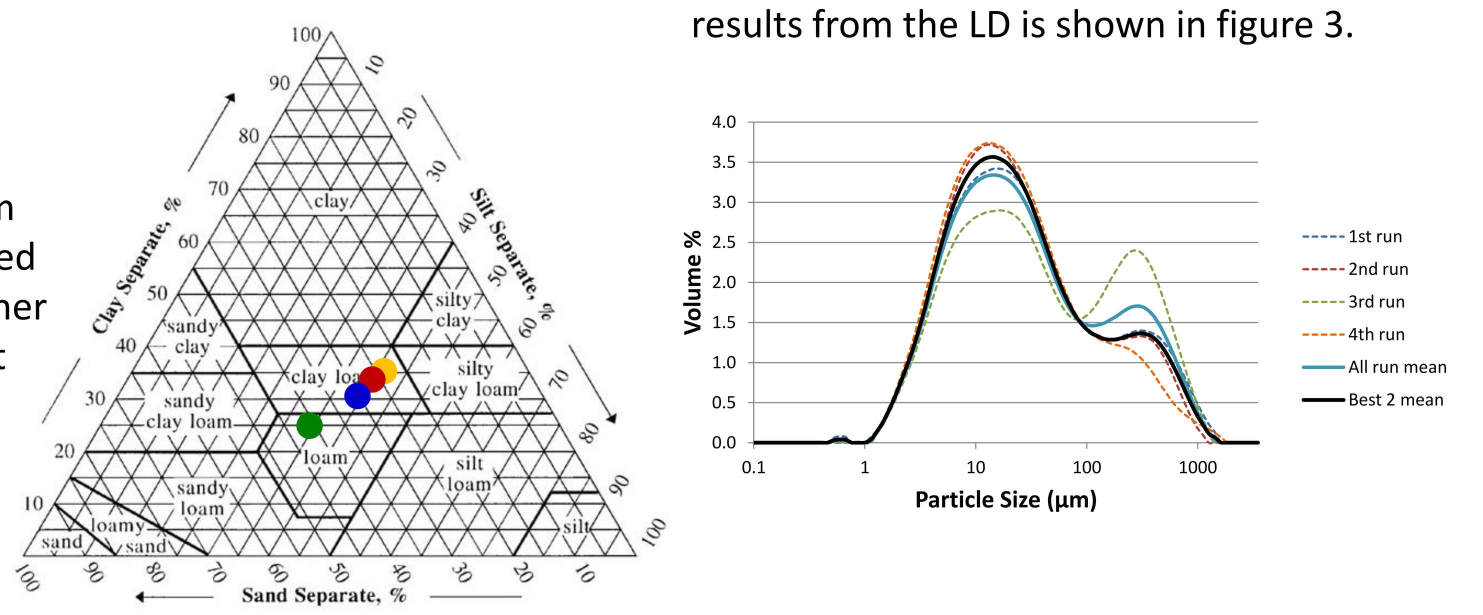
LD clay-silt threshold	Pipette 1 - LD	Pipette 2 - LD
6 µm	38%	33%
8 µm	51%	36%
10 µm	51%	38%

Table 3. Percent agreement between two pipette method labs and LD using different particle size breaks for clay-silt.

Method	Texture Class
Pipette Lab 1	Loam
Pipette Lab 2	Clay
LD (10µm threshold)	Clay Loam

Table 4. Example of texture class determinations for one soil sample measured by three different labs. The variability of results from the LD is shown in figure 3.

Figure 3. Particle size distribution curves (far right) and texture triangle plot (near right) from four runs using LD on the single sample classified in table 4. The third run is a loam, while the other runs are all clay loams when the 10 µm clay-silt threshold is used.



Conclusions

Volume percentages of particle size distributions can vary considerably between measurements of the same sample by both the pipette and laser diffractometry methods, which frequently can cause a change in the determined texture class. However, variability in measured percent volume does not translate to variability in particle size mode determined by laser diffractometry. Spatial analysis of trends in particle size mode will be more reliable than abundance ratios between particle size fractions.

Although pure silt samples, e.g., loess, tend to have the least amount of measurement variability, researchers utilizing laser diffractometry should be aware of two potential issues:

1) Samples with some sand content can have larger differences in subsample measurements due to the inclusion/exclusion of a few grains can have large impact on the volume percentages.
2) Determining clay contents with laser diffraction has proven problematic because the LD software assumes that all particles are round. The size of the plate-shaped clay particles is then either under- or over-estimated depending upon the clay particle's orientation when the laser measures it. This problem results in more random variability in measurement of clayey samples, as compared to other soil texture classes.

References

Miller, B.A., Schaetzl, R.J. 2012. Precision of Soil Particle Size Analysis Using Laser Diffractometry. Soil Science Society of America Journal 76:1719-1727.