Precision of Soil Particle Size Analysis using Laser Diffractometry

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Precision, particularly in terms of repeatability in particle size analysis (PSA), has recently resurfaced as an issue due to the increased use of laser PSA for PSA. Because laser diffractometry produces much more detailed data than does traditional pipette analysis, and because a much smaller sample is used in the analysis, precision or repeatability of laser-produced PSA data is a legitimate concern. For example, of the 1485 soil samples analyzed in our study, most of which are silty or loamy and each of which was analyzed at least twice, 11.5% changed texture class, from the first to the second PSA measurement. Statistical analysis of these paired, subsample data was therefore used to establish a standard for normal variance among the subsamples, as a test of precision. Subsample pairs with an absolute cumulative bin difference (CBD) of <1 SD above the population's mean CBD were determined to have acceptable precision. This approach provides both a simple method for assessing the variation in PSA data sets and establishes a comparable standard for determining when additional measurements are needed to find a more precise result. Researchers may be tempted to simply use mean PSA data for samples that have been run multiple times. However, we found that using the per bin mean for only the two best matching subsamples was the optimal approach. Our analysis also indicates that, as particle size gets coarser (at least within silty/loamy samples), the precision of laser-generated particle size data generally decreases.

Abbreviations: CBD, cumulative bin difference; MBD, mean bin difference; PSA, particle size analysis; USDA, United States Department of Agriculture.

Particle size is a fundamental analysis procedure for soils, and pedological and paleopedological analysis (e.g., Langohr et al., 1976; Rawls, 1983; Miller et al., 1988; Mason and Jacobs, 1999; Konen et al., 2003; Lindbo et al., 2008). Soil particle size distributions influence most pedogenic processes, and therefore, allow for inferences about the pathways of soil development. For example, soil particle size is an important factor in the formation of subsoil structure (White, 1967), the depth of clay accumulation (Muckenhirn et al., 1955; Putman et al., 1988), the degree of podzolization (Schaetzl, 1991; 1996), and thickening of the solum (Protz et al., 1985; Harden, 1988; Schaetzl, 1992). The particle size characteristics of sediments are also very important criteria in determining their geologic origins (Fehrenbacher et al., 1965; Frazee et al., 1970; Tsoar and Pye, 1987; Ding et al., 1999; Mason, 2001; Stanley and Schaetzl, 2011). And lastly, soil particle size influences many soil quality factors, for example, permeability, water retention, ease of tillage, seed germination, aggregate structure, erosion potential, C storage, and nutrient storage (Ben-Hur et al., 1985; Rawls et al., 1982; Imhoff et al., 2002; Keddy and Constabel, 1986; Chepil, 1953; Silver et al., 2000). Therefore, few question the need for accurate and precise particle size data in soils and Earth

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science applications. As a result, advances in the accuracy and efficiency of particle size characterization have accrued for as long as scientists have been analyzing mineral sediment.

During the last three decades, laser diffractometry has been increasingly used as a means of characterizing particle size distributions of a variety of sediments. Laser diffractometry provides both quick and highly detailed PSA results (e.g., Loizeau et al., 1994; Manalo et al., 2003; Goossens and Buck, 2009; Stanley and Schaetzl, 2011). With some conversion, these types of data have also been found to provide comparable results to previously accepted methods of soil particle size characterization, for example, pipette analysis (Beuselinck et al., 1998; Campbell, 2003; Eshel et al., 2004; Arriaga et al., 2006, Duc et al., 2007).

Laser particle size analyzers report results as the percent of the sample's particle volume, within categories of small, discrete particle size ranges. Like a relative frequency histogram, the ranges of the categories (bins) can be adjusted to increase or decrease the resolution of the results, for example, the traditional five sand category "splits" (cf., Scull and Schaetzl, 2011). The laser particle analyzer used in this research–a Malvern Mastersizer 2000–uses a 105 bin distribution. Because particle size data are, in theory, continuous, more (and smaller) bins provide a better and more detailed picture of the particle size distribution. Indeed, with the higher level of detail, minute textural differences between samples can be determined. However, the higher level of detail provided by laser particle size analysis also has drawbacks; it increases the sensitivity of the results to different measurement values. For example, if a second (different) measurement value for a particle's size places that particle in the same bin as the first measurement, then the difference has no effect on the resulting data. But if the difference is enough to change the bin that the particle is categorized in, which happens more frequently with smaller bin sizes, the resulting data are affected.

When considering smaller differences between samples, the precision (or repeatability) of the result increases in importance because differences could potentially be only due to measurement variability. Measurement variability, that is, lack of precision, can result from different sample preparation methods, varying strength or optical properties of the sediment being analyzed, and/or machine parameters (Sperazza et al., 2004; Storti and Balsamo, 2010). Sperazza et al. (2004) found that the precision of the Malvern Mastersizer 2000 for samples measured 15 consecutive times, that is, without being removed from the machine, was within 1% for medium grain sizes. However, additional measurement variability could be introduced from different subsamples of the original sample. Separate subsamples can have differences in concentrations in the test solution (i.e., obscuration), human error, as well as other factors that may change between the measurement of one subsample and the next. Despite efforts to avoid issues associated with the natural heterogeneity of soil samples, soil scientists often measure particle size distributions on samples that are not completely homogeneous within the sample container, usually a bag. This is especially a potential problem for laser PSA, because such a small subsample (usually 0.3–0.5 g) is used for analysis. By way of comparison, in traditional pipette analysis, samples of 10 to 40 g are commonly used (Day, 1965; Soil Survey Staff, 1996, 2009). Use of small subsamples does have some advantages, as well, for example, when only minute amounts of sample are available for analysis. Nonetheless, when small samples are analyzed, repeatability can be an issue, and that is the focus of our research.

The solution to the issue of measurement variability among subsamples, which could also be called random error, rests in understanding the frequency distribution of possible results. Comparisons of data from multiple subsamples can provide a reference for the normal variation that can be expected within a sample proper. These data, plotted as frequency distributions, allow researchers to have a basis for evaluating if differences in particle size distributions between samples are attributable to measurement variability. In this study we use simple descriptive statistics to examine the precision of laser PSA results for multiple subsamples. Our results will be useful for soil scientists who need to eliminate the possibility of measurement variability as an explanation for inter- or intra-sample differences.

When multiple measurements from the same sample produce markedly different results, a dilemma is created: How should one derive a single, best, representative result from multiple data? Where measurement variability is an issue for analyses with single value results, that is, concentration, a common approach is to use the mean of multiple measurements. Assuming a normal distribution of measurement results, the mean of an infinite number of measurements would be at the middle of the frequency distribution curve. Because particle size analysis provides results that consist of a set of interdependent values, that is, a decrease in one bin's value causes an increase in other bins' values, calculating a single representative result is more complicated. Therefore, in this study we also propose and compare two methods for deriving a single set of values from data derived from PSA measurements of multiple subsamples.

MATERIALS AND METHODS

Using a standard bucket auger, we collected 1485 samples from loess and similar deposits across Wisconsin and the western Upper Peninsula of Michigan. In the field, we recovered samples of approximately 500 g and air-dried them. Next, the samples were lightly crushed and passed through a 2-mm sieve to remove coarse fragments. Because laser PSA uses a very small subsample from the larger soil sample (<0.001% of a 500-g sample), it was important to make the sample as homogeneous as possible, to minimize the potential variability of results from different subsamples. Therefore, each fine-earth sample was passed through a sample splitter– and recombined–four times. It is important to note that loess is quite homogeneous naturally, because of the sorting nature of the eolian depositional system. Therefore, our data should be viewed as representative of samples that have a minimum amount of inherent particle size variability.

Particle size was measured using a Malvern Mastersizer 2000E laser particle size analyzer with a Hydro 2000MU pump

accessory (Malvern Instruments Ltd., Worcestershire, UK). As different materials and dispersants have different optical properties, this device requires these parameters to be set by the user. For our analysis, we assumed the refractive index and absorption values of the soil materials to be 1.549 and 0.01, respectively. Distilled water was used as the dispersant; it was assumed to have a refractive index of 1.33.

A subsample of 0.3 to 0.5 g was removed from a sample bag, using a spoon, and placed into a 25-mL glass vial. Five milliliters of a sodium hexametaphosphate dispersing solution was then added to the vial. The vial was filled with »15 mL of distilled water, bringing it to ≈90% capacity, and shaken for 1 to 2 h, to disperse any aggregates. The soil water suspension was then added to a 500 mL beaker of distilled water that was continuously stirred by a turbine, spinning at 3000 rpm, on the laser diffraction unit. The Malvern Mastersizer continuously pumps a portion of this suspension through a gap between two glass lenses, or windows. The size of the particles passing between the lenses is measured by the scattering pattern of the laser, as it diffracts off of the particles, by application of the Fraunhofer model, and using Mie theory (Malvern Instruments, 2004). Since laser diffractometry often underestimates the amount of <2 μm particles due to their plate-like shape (Loizeau et al., 1994; Beuselinck et al., 1998). Konert and Vandenberghe (1997) suggested the clay–silt break be set at 8 μm for comparison with traditional, that is, pipette, PSA. In house data have suggested better correlations between laser diffractometry and the pipette method when the clay–silt break is set at 6 μm. Therefore, we used a 6 μm clay–silt break in this study. For further discussion on the principles and application of laser diffractometry for particle size characterization see McCave et al. (1986), Singer et al. (1988), Agrawal et al. (1991), Loizeau et al. (1994), and Wen et al. (2002).

Laser PSA was conducted on 1485 samples, and then repeated by extracting and analyzing an additional subsample from each of these samples. Data from the first two subsamples were used as a starting point to ascertain the normal amount of intrasample variation. The results of the multiple measurements were compared by reporting in the traditional three bin (clay, silt, sand) and seven bin (clay, silt, and the division of sand into five subcategories) formats, as well as the more detailed 105 bin format of our laser diffraction setup. The distribution of differences was analyzed on a per bin basis for the three and seven bin reporting formats. However, analyzing per bin differences was not practical for 105 bins. Therefore, two summary statistics were calculated and implemented for all three reporting formats. The cumulative bin difference (CBD) was calculated by summing the absolute differences between two subsamples for all of the bins, as follows:

$$CBD = \sum_{k=1}^{n} | a_k - b_k | \quad [1]$$

a = first subsample value, b = second subsample value, n = number of bins.

The mean bin difference (MBD) was calculated by dividing the CBD by the number of bins:

$$MBD = \frac{CBD}{n} \quad [2]$$

Because the bin values are in volume percent, the units for CBD and MBD are also in percentages.

Both the CBD and MBD for the population of 1485 samples were plotted and analyzed in histograms, to better understand their frequency distributions. The analysis of histograms is a common strategy for quality control (Tague, 2005). As a means of identifying outliers in this dataset, subsample pairs with CBDs exceeding 1 SD more than the mean of all samples' CBDs were considered to be unacceptable, that is, their precision or repeatability was not high enough for this type of analysis. In other words, CBD values in the far right-handed tail of a positively skewed distribution were selected for MBD was also investigated. The CBD was chosen to identify outliers because the CBD will be larger when many bins have small differences, while the MBD would remain small.

When necessary, additional subsamples were measured, using the same procedure as the previous subsamples, until every sample had a pair of subsamples with a CBD less than the precision threshold. The samples chosen for additional measurements were only those that continued to exceed the precision threshold. This precision threshold was the mean of all samples' CBDs plus 1 SD, as set by the analysis of the first two subsamples.

RESULTS AND DISCUSSION

Precision of Particle Size Analysis in Sandy/Loamy Soils using Laser Diffractometry

Sample textures analyzed in this study ranged from loamy sand, to loam, to silt (Fig. 1). Clay contents of the samples ranged from 0.7 to 33.5%. Silt and sand contents had wider ranges of volume percent contents; silt contents ranged from 9.4 to 79.0%; sand contents ranged from 0.0 to 89.9% (Table 1; Fig. 1). For this reason, extrapolating our results (below) to fine-textured or sandy soils should be done with caution

It is important to note that because the PSA results are reported as volume percentages, bin values as well as their differences, means, and SDs all are reported in "percent" units. To avoid confusion, we will not report any of the variances as percentages of a result, but only as differences in content volume percentages.

The mean of all the samples' CBDs between the first two subsamples, for 3, 7, and 105 bins, were 7.3, 9.5, and 10.6%, respectively (Table 2). The increase in the CBDs with a higher quantity of bins was expected because of the lower impact that measurement differences would have on results that used wider bin ranges. It is this effect that makes the analysis of precision more important when reporting particle size distributions with higher levels of detail, for example, data from laser PSA.

The three bin reporting format allows for the analysis of the precision of each bin individually. The mean (and SD) of the differences between all of the samples' first two subsample measurements for the clay, silt, and sand bins were 1.1 ± 1.3%, 2.8 ± 3.1%, and 3.5 ± 4.0%, respectively. Therefore, this analysis suggests that 84.1% of repeated measurements can be expected to return dif-

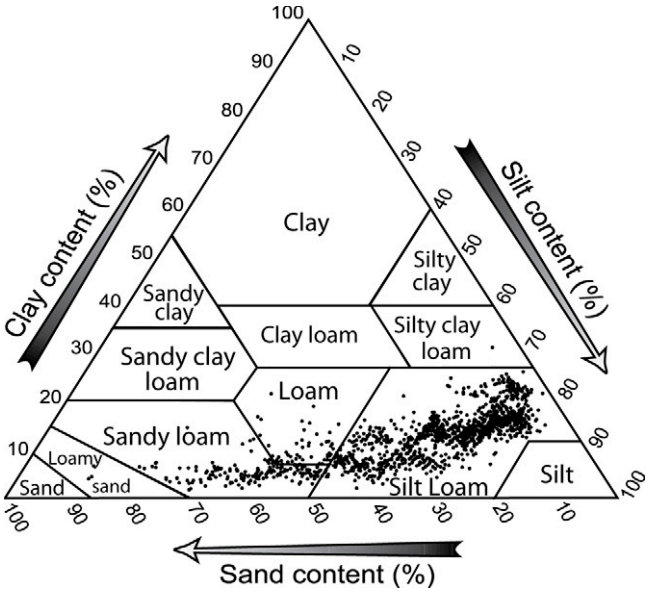

Fig. 1. Distribution of all sample results from the first two subsamples, plotted on a standard USDA texture triangle.

ferences of <2.3% for clay, <5.8% for silt, and <7.5% for sand. For comparing volume percentages quantitatively, the precision values provide a range of likely repeated measurement results. In this way, these precision ranges are describing the random error, with the large sample set on which they are based thereby providing a high level of confidence for the range of samples. These values could be used instead of standard error for identifying differences between samples that could be due to random error.

In addition to determining the sand–silt–clay absolute differences that can be expected from multiple PSA runs (see above), we also calculated the USDA texture classification of the subsamples (Soil Survey Division Staff, 1993), based on the same data. Our data show that 171 of the 1485 (11.5%) samples actually changed texture class, from the first to the second PSA measurement. Of the samples that changed texture class, the most common shifts were between silt loam and loam, and between silt loam and sandy loam. From the 171 samples that changed texture class, only one sample shifted to a nonadjacent texture class. The sample that changed more than a single texture class, shifted from silt loam to loamy fine sand. These shifts likely show the impact that a few grains of sand can have on volumetric percentages. If a texture class shift is acceptable »12% of the time, then one PSA measurement by laser diffractometry should be sufficient. However, because particle size data are, in theory, continuous, more bins do provide a better and more detailed picture of the particle size distribution. Indeed, with the higher level of

Table 1. Summary of particle size results for the first two subsamples from entire sample population, separated into three particle size bins, and with the sand category divided into five bins.

Percentage of volume	Clay	Silt	Sand	VFS†	FS	MS	CS	VCS
Minimum	0.74%	9.39%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
Maximum	33.52%	79.0%	89.9%	34.7%	54.7%	44.3%	26.2%	2.0%

† VFS, very fine sand; FS, fine sand; MS, medium sand; CS, coarse sand; VCS, very coarse sand.

Table 2. Particle size differences between first two subsamples, by three particle size bins and with the sand category divided into five bins.

Difference	Clay	Silt	Sand	VFS†	FS	MS	CS	VCS
Minimum	0.00%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
Maximum	18.77%	38.3%	54.4%	13.9%	30.0%	17.2%	15.1%	1.7%
Mean	1.07%	2.8%	3.5%	1.1%	1.5%	1.6%	1.4%	0.1%
Different SD	1.27%	3.1%	4.0%	1.3%	1.9%	1.9%	1.6%	0.2%

† VFS, very fine sand; FS, fine sand; MS, medium sand; CS, coarse sand; VCS, very coarse sand.

detail, small textural differences between samples can be determined. We point out, however, that the repeatability of texture class determination may be considerably greater than 88% for finer-textured samples (see below).

The decrease in precision, that is, the increase in variability that occurs with the increase in particle size, as shown above in the three bin reporting format (Table 2), is also observed when the reporting formats include more bins. The mean bin difference, as well as the SD of bin differences, increased with increasing particle size for the 105 bin reporting format (Fig. 2). The seven bin reporting format followed this trend through the fine sand fraction. However, mean bin differences and their SDs decreased as fewer samples had particles in the coarser size ranges. Also, our laser particle size analyzer does not evaluate particles within the 1100 to 2000 μm range, and therefore, the mean differences and SDs were 0 for bins within this range. The seven bin reporting format expands the number of bins in the sand fraction from one to five, placing a greater emphasis on the sand fraction. The trend of an increase in measurement variability with larger particle sizes was less clear for the seven bin reporting format because of its emphasis on the sand fraction and the limitations of our analyzer to detect particles >1100 μm. However, given the clarity of the trend in the 3 and 105 bin reporting formats, it is

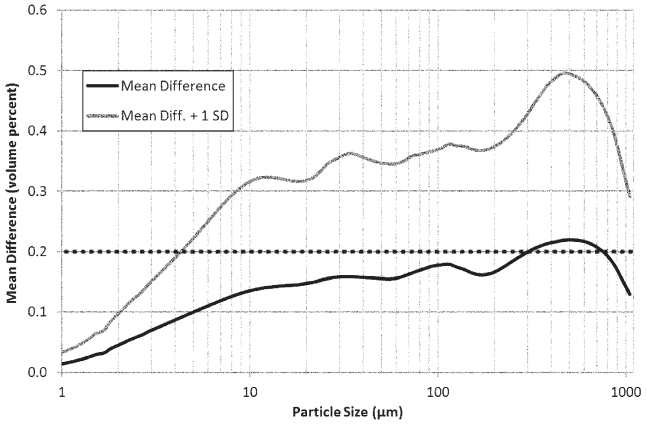

Fig. 2. Mean difference and mean difference plus 1 SD for the 105 particle size bins of all 1485 samples. The mean bin difference (MBD) precision threshold, which identified the same samples as the cumulative bin difference (CBD) precision threshold, was 0.2% (dashed line). Individual bins with mean differences greater than the MBD precision threshold indicate the types of samples that were most likely to have unacceptable precision. The decline in mean difference and SD for particle sizes >500 μm is likely due to the paucity of samples in our population containing those particle sizes.

likely that the trend would hold if samples with particle sizes in the 1100 to 2000 μm range had also been analyzed.

The increase in measurement variability in samples with larger particle sizes is likely due to sample homogeneity issues. A small number of sand particles–either included in a given subsample or not included in another–can have a large effect on the volume percent measurements of the sand fraction. Despite efforts to homogenize our samples, larger particles (which comprise a proportionately smaller quantity of the particles in a sample) are difficult to make equally represented in every subsample.

The per bin variance ranges shown earlier for the three bin reporting format are not as practical for describing the precision for the more detailed 105 bin format. Keeping in mind that precision decreases with increased particles size, the precision of the 105 bin format can be summarized with the MBD of all samples plus the SD. In our analysis, we found that 86.1% of repeated (second) results had a MBD of 0.2% or less. However, the repeated measurements also indicated that subsample results can have a MBD as high as 1.0% or an individual bin difference as high as 3.8% in the 105 bin format.

After analyzing the precision characteristics of the first two subsamples (Table 3), we used the mean and SD of the CBD to (i) establish a threshold for determining which samples are statistical outliers and (ii) determine the number of repeated measurements required to provide two subsample results whose differences are below that threshold. The CBD and MBD histograms of all three reporting formats showed positively skewed distributions (Fig. 3). This finding supports the classification of subsample differences in the right-handed tail of the CBD or MBD histograms as outliers. Therefore, we set the CBD precision threshold as the mean CBD plus 1 SD that was established by the first two subsample measurements of all 1485 samples to identify outliers that needed additional analysis. As the mean CBD and SD differed, depending on the number of bins, the precision threshold was necessarily different for the three reporting formats. The CBD precision threshold for the 3, 7, and 105 bin reporting formats was set at 15.1, 18.0, and 19.8%, respectively.

We chose to use CBD as our precision standard because the CBD is likely to be more sensitive than the MBD to small differences across many bins. However, we did calculate the MBD and evaluated subsample measurement results in the same manner as with the CBD precision threshold. After performing this comparison, we found that the MBD precision threshold identified the same sample measurements in our data set as outliers as did

Table 3. Mean and standard deviation data for measures of difference between the first two subsamples.

Bin	Mean	1 SD	Precision thresholds†
Cumulative 3 bin (-)	7.3%	7.8%	15.1%
Mean 3 bin (-)	2.4%	2.6%	5.0%
Cumulative 7 bin (-)	9.5%	8.5%	18.0%
Mean 7 bin (-)	1.4%	1.2%	2.6%
Cumulative 105 bin (-)	10.6%	9.2%	19.8%
Mean 105 bin (-)	0.1%	0.1%	0.2%

† Set by calculating the mean plus 1 SD.

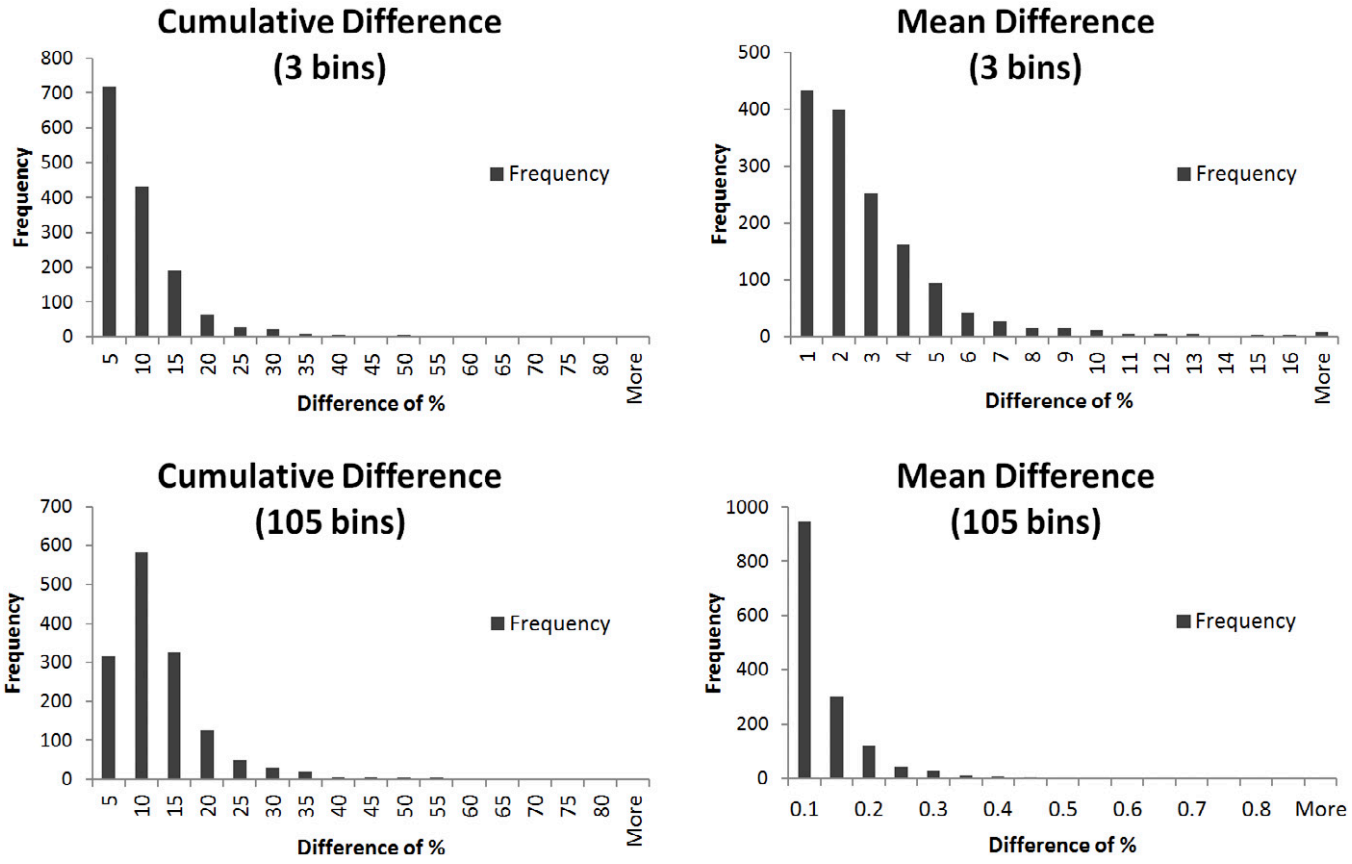

Fig. 3. Histograms of cumulative and mean bin differences, by 3 and 105 bins.

Table 4. Summary statistics for each subsequent subsample measurement dataset.

Range/Mean percentage	First set†	Second set†	Third set‡	Fourth set§
Clay range	1–34%	2–25%	1–24%	2–19%
Silt range	9–79%	11–78%	11–76%	24–70%
Sand range	0–90%	5–85%	5–86%	14–73%
Mean percent clay	11%	11%	9%	8%
Mean percent silt	58%	59%	52%	49%
Mean percent sand	30%	30%	39%	43%

† Includes all samples.

‡ Includes only samples that exceeded the acceptable difference threshold from the first two sets.

§ Includes only samples that continued to not have an acceptable pair after three subsamples.

the CBD precision threshold. Therefore, the special case where CBD might be more sensitive than MBD did not occur in any of our samples. This result suggests that the MBD precision threshold identifies the same outliers as the CBD precision threshold, in most circumstances.

In cases where repeatability is a concern, the precision threshold can be used to identify samples requiring additional subsample measurements; the repeated measurements can assist in filtering out random, intrasample variability. From a quality control point of view, using a threshold of the mean difference plus 1 SD identifies outliers from the expected, normal variation. However, the level of precision needed for different types of research may differ from this standard. Using the assumption that measured values will have a Gaussian distribution around the population mean, the probability of two sample measurements being similar increases closer to the population mean. Therefore, the more similar two runs are to each other, the greater the likelihood that they are similar to the population mean. This assumption does not eliminate the possibility of two similar sample measurements being greatly different than the population mean, but reduces the risk with practical methods (i.e., the quantity of repeated measurements is minimized).

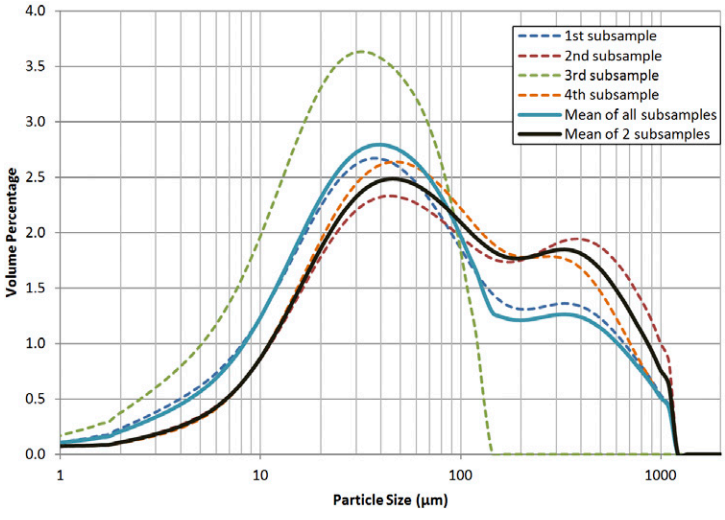

Fig. 4. Example of continuous particle size curves generated from the 105 bin results of a single, silty, loess sample. In this example, the mean of the two best matching subsamples has been used for the best possible representative curve for repeatable results. This method avoids the influence of data from extremely different, that is, anomalous, subsamples (i.e., the 3rd subsample). In this case, including the third subsample increases the percentages in the silt fraction while decreasing the percentages in the sand fraction.

From our 1485 samples, the 105 bin standard found 134 samples (9.0%) to be outliers, that is, they had a CBD above the threshold. The three bin standard identified 140 such samples, and the seven bin standard identified 143 outliers from the original two subsamples of all the samples. The higher number of outliers identified by the three and five bin formats is likely due to the higher level of precision set as the precision threshold for these reporting formats. Using fewer bins made the results less sensitive to measurement differences, and therefore the CBD distribution was not as wide.

Each of the 134 samples identified as outliers by the 105 bin standard were run a third time. Results from this third analysis show that the 134 subsamples had a generally similar soil texture *range* as the first two subsample sets. Interestingly, many of these "third run" subsamples had higher sand contents than did the larger sample population. The mean percentage of sand was 30.1% for the full sample set, but 38.9% for the set of 134 samples that warranted a third analysis run (Table 4). This finding supports our conclusion that greater measurement variability appears to correlate with sandier samples. The per bin precision analysis conducted on the 105 bin reporting format for the entire data set predicted that over 50% of samples with mean weighted particle sizes >302 µm would likely have unacceptable precision after two runs (Fig. 1).

From the 134 samples that had a third subsample tested, 39 still did not have a pair of subsamples that were similar enough to be considered acceptable. As would be expected, the smaller population of this fourth set of subsamples had a narrower range of percentages in the soil separates categories. The continued increase in the mean percent sand for this fourth subsample set (Table 4) again supports our conclusion that samples with coarser particle sizes, especially those with more sand, yield more variable, intrasample, particle size results by laser diffractometry. This finding again demonstrates the impact that a few grains of sand can have, in a small sample, on PSA data, due to their exponentially larger volume.

After a fourth subsample was analyzed on the remaining 39 outlier samples, only one sample did not have a pair of subsamples similar enough to be below the 105 bin precision threshold. This sample had total sand content results that ranged from 48 to 68%, showing that it was one of the sandier samples of the entire population. A fifth measurement on this sample did produce results similar enough to a prior measurement to be below the precision threshold. The particle size distribution for this sample was bimodal, with concentrations of particles in the 63 to 69 µm and 302 to 331 µm ranges. Depending on the subsample, different relative amounts of these particle size groups were included in each of the analysis runs. All five subsamples from this sample would have classified within (or very close to) the sandy loam textural class.

The CBD and MBD statistics were useful tools for analyzing the precision of PSA results. They were also useful for quantitatively identifying repeated PSA results that differed from each other, beyond a specified standard. Whereas our study used this method to analyze the precision of PSA results from laser diffractometry, they could be used in a similar way for assessing the precision of other PSA methods, or conceivably for any other measurement that consists of a series of interdependent values.

Obtaining Representative Values from Multiple Measurements

Because each PSA run returns unique data, researchers will need to develop strategies that return a single, representative (but optimal) set of particle size values for that sample. In other words, after all the extra work of running subsamples twice, or more often, what should be done to these data to yield an optimal suite of data for the 105 (or 7 or 3) particle size bins? To answer this question, we calculated and graphed the continuous particle size curves for each sample, using two approaches: (i) using the mean for each particle size bin, based on all subsample results, and (ii) using the mean values for only the two best matching subsample results (Fig. 4). The best matching subsamples were considered to be the subsamples with the lowest CBD. For samples whose first two subsamples were similar enough to not warrant additional subsample analysis, there is no difference in these two approaches. The approach using the mean of the two best matching subsamples avoided influence from subsamples' measurements that were unacceptably different than the others. The two best matching subsample approach, that is, no. (ii) above, was also more likely to ignore anomalous particle size peaks, which usually occurred in the sand fraction (Fig. 5).

Nonetheless, the two best matching subsample approach did not always remove the influence of irregular particle size peaks. If a single subsample with an "anomalous" peak closely matches another subsample in most of the other particle size bins, that subsample could be one of the two best matching subsamples, in which case, the resulting mean curve would still reflect some of that peak. Figure 6 shows an example where a subsample with an anomalous peak is one of the best matching pairs. In this situation, there would again be little difference between the mean of all subsamples and the mean of only the best two matching subsamples. Therefore we recommend using the mean of the two subsample values with the least CBD in combination with the CBD precision threshold used in this paper. The CBD precision threshold will identify which samples require more measurements to increase the confidence that the two best matching subsamples are representative of the sample. Although the mean of the two best matching subsamples will not always eliminate anomalous particle size peaks, it will minimize their influence.

CONCLUSIONS

Although laser diffractometry has many advantages over previous PSA methods, the increased level of detail it provides, and the smaller sample sizes used in the analysis method, both introduce concerns of repeatability. Our research addresses these issues by characterizing the precision of repeated subsample measurements and evaluating methods for producing sample results with a high confidence of repeatability. Use of either an absolute CBD or an absolute MBD will prove helpful for researchers examining PSA data precision.

We caution that our results were based on research performed on silty–loamy soils; sandy or clayey soils may have different variances. Although our results showed trends that could be extrapolated to samples with other textures, only additional work–on samples of those kinds–can provide the higher level of

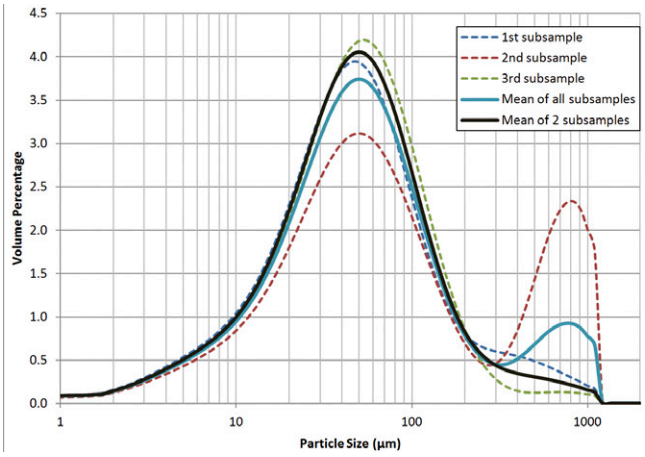

Fig. 5. An example of continuous particle size curves generated from the 105 bin results of a single sample. In this example, the mean of the two best matching subsamples filter out the "anomalous" sand peak in the particle size distribution. Although deemphasized, using the mean of all four subsamples allowed for the sand peak to remain a part of the particle size curve. Use of the mean of the two best matching subsamples removed the peak that was only measured in one subsample.

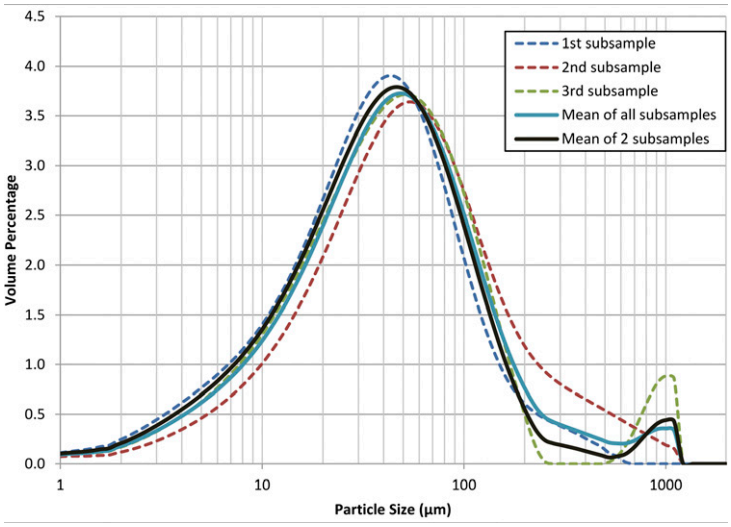

Fig. 6. Example of continuous particle size curves generated from the 105 bin results of a single sample. In this example, the mean of the two best matching subsamples does not filter out the "anomalous" sand peak, because the subsample with the anomalous peak closely matches another subsample in the other particle size bins. Schaetzl and Luehmann (2012) examined the origins of these "anomalous" sand peaks in loess samples.

confidence in the expected variability of laser particle size measurements for clayey and sandy samples. Also, even though this study specifically assessed the precision of PSA results from a Malvern Mastersizer 2000E, the variation is largely due to the measurement of different subsamples. Therefore, we anticipate this method to be useful for assessing and comparing the precision for similar data generated using other PSA methods.

In this analysis, performed on silty/loamy soils, result variability, that is, random error that was most likely due to intrasubsample heterogeneity, increased with coarser textured samples, especially those that were slightly sandier. Results from 1485 samples, mostly within the sandy– loam, loam–silt part of the texture triangle (Fig. 1), suggest that the normal variance of measurements had only minor impacts on the soil texture classification. Within a three bin reporting format, the mean difference between two subsamples was 1.1% (SD = 1.3%) for clay, 2.8% (SD = 3.1%) for silt, and 3.5% (SD = 4.0%) for sand. Because these values are based on a large sample set, they could be used as a type of confidence interval for assessing differences due to random error. Variability of results in a 105 bin format is better described with a summary statistic. For the samples in our study, the MBD was 0.1% with a SD of 0.1%.

To screen for outliers, we recommend measuring a second subsample and conducting further analysis on samples exceeding a precision threshold. In our study, samples with two subsamples that had a CBD greater than the population's mean CBD plus 1 SD were selected for additional analysis. For the 105 bin reporting format, our precision threshold was set at a CBD of 19.8%. Within five subsample measurements, all samples had a pair of subsample results with CBDs below this threshold for acceptable differences.

We reiterate that finer-textured samples are least likely to require additional PSA measurements; coarser-textured samples are best run at least twice, because of higher measurement variability. Our data suggest that, if the analyst wishes only to categorize samples to texture class, only one measurement is usually necessary, especially on finer-textured samples. After all samples have subsamples meeting the CBD precision threshold, we recommend using the per bin mean of the two subsamples with the least CBD to derive the best representative values for repeatable results.

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