

## Critical Evaluation of the Use of Laser Diffraction for Particle-Size Distribution Analysis

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### ABSTRACT

Determination of soil particle-size distribution (PSD) by sieving, hydrometer, and pipette methods as well as by laser diffraction (LD) suffers from inherent flaws, mainly due to the difficulty in defining the size of irregularly shaped particles. Therefore these methods yield only estimates of PSD. The objective of this study was to determine whether a functional relationship exists between the PSDs obtained by the combined sieve-pipette method and those obtained by LD. Samples from 42 California soils were analyzed. For the LD measurements a Beckman-Coulter LS-230 apparatus with a 750-nm laser beam that measures particles in the range of 0.04 to 2000  $\mu\text{m}$  was used, employing the Mie theory for the PSD calculations. Values of 1.5 and 0.2 for the real part and the imaginary term of the refractive index (RI), respectively, gave satisfactory results for the optical model calculations. Volume percentage of the clay-size fraction obtained by LD was generally lower than mass percentage of the clay fraction derived by the pipette method. The opposite trend was noted for the silt-size fraction. Coefficients of determination for the regression equations for the clay, silt, and sand fractions determined by the two methods were 0.702, 0.689, and 0.821, respectively. Good agreement between measured and calculated LD values for one size class was accompanied by poor agreement between measured and calculated values for the other. The LD method provides a continuous PSD curve, which enables a detailed data analysis and a flexible application of different particle-size dependent classification systems.

PARTICLE-SIZE DISTRIBUTION is one of the most fundamental physical properties of a soil, defining, for example, the soil's texture, and strongly affecting many physical and chemical soil properties. It is typically presented as percentage of the total mass of soil occupied by a given size fraction. Determination of the soil PSD is not a trivial task because of the heterogeneity of the shape and density of particles.

### Particle-Size Distribution Determination Methods Employed in Soil Science

The classical techniques for determining PSD in soils include sieving and procedures based on sedimentation, such as the pipette and hydrometer methods (Gee and Bauder, 1986). The pipette and the hydrometer methods give comparable results (Liu et al., 1966; Walter et al., 1978) provided similar pretreatment techniques are used. However, the pipette method, requires the additional step of first separating the sand fraction (50–2000

$\mu\text{m}$ ) by wet sieving from the clay and silt (particles <50  $\mu\text{m}$ ). The sand fraction is determined by drying and weighing the material remaining on the sieve. The silt and clay fractions are then determined by the pipette method (Gee and Bauder, 1986; Janitzky, 1986). These classical methods suffer from numerous disadvantages. For example, the sieving method is useful only in the size range of 2000 to 50  $\mu\text{m}$  (Gee and Bauder, 1986). A major source of error in the hydrometer method is simply the inaccuracy in the hydrometer reading (Gee and Bauder, 1979). The sedimentation-based methods require relatively large samples (10–20 g for the pipette and 40 g for the hydrometer) and their capacity for resolution to subgroups of sizes is limited. In addition, the sedimentation-based procedures are time-consuming, especially for the determination of <2- $\mu\text{m}$  size fraction, while for particles <1  $\mu\text{m}$  the results are increasingly unreliable because of the effect of Brownian motion on the rate of sedimentation (Allen, 1981; Loveland and Whalley, 2001).

Numerous techniques for the determination of PSD, other than these classical ones, were developed, mainly for industrial applications such as the determination of the homogeneity of powders and gels (Buurman et al., 1997). Among those techniques, LD (or laser light scattering) was occasionally applied to soil material (e.g., Cooper et al., 1984; Levy et al., 1993; Buurman et al., 1997; Muggler et al., 1997; Konert and Vandenberghe, 1997; Chappell, 1998; Beuselinck et al., 1998). In this latter method the forward diffraction of a laser beam by the particles is used to determine their size distribution. The angle of diffraction is inversely proportional to particle size, and the intensity of the diffracted beam at any angle is a measure of the number of particles with a specific cross-sectional area in the beam's path. Two optical models are commonly used to calculate PSD, the Fraunhofer diffraction model and the Mie theory. The former is based on the approximation that the laser beam is parallel and the detector is at a distance that is very large compared with the size of the diffracting particle. The Mie theory is a solution of the Maxwell equations (i.e., a set of four fundamental equations governing the behavior of electric and magnetic fields) describing propagation of the electromagnetic wave of light in space. The theory provides a solution for the case of a plane wave (i.e., the wavefronts of which are planes) on a homogeneous sphere of any size (Jonasz, 1991). In addition, it takes into account phenomena other than diffraction (e.g., transmission through the particle) and therefore requires knowledge of the RI of

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**Abbreviations:** LD, laser diffraction; PIDS, polarization intensity differential of scattered light; PSD, particle-size distribution; RI, refractive index.

the material tested. The Mie theory thus offers an exact solution to the scattering of light from a homogeneous sphere (but not from an irregularly shaped particle). The resultant PSD computed by either the Fraunhofer diffraction or the Mie theory is a volume (rather than mass) based size distribution. Early generation LD instruments for PSD determination suffered from a size detection limit  $>0.5 \mu\text{m}$ . In addition, they employed mainly the Fraunhofer diffraction, which is inaccurate for particles smaller than  $d = 10\lambda$  (where  $\lambda$  is wavelength) (Bayvel and Jones, 1981; de Boer et al., 1987); for the Beckman-Coulter LS-230 it would be particles  $<7.5 \mu\text{m}$  in diameter. In newer LD apparatus, the lower detection limit was extended to approximately  $0.04 \mu\text{m}$ .

Main advantages of the LD technique for PSD determinations include: short time of analysis (5–10 min per sample), high repeatability, small size of sample needed ( $\leq 1 \text{ g}$ ), and a wide range of size fractions into which the entire range of particle sizes can be divided. The latter point is of particular importance because the availability of a continuous PSD, rather than an arbitrary division of the particles among a limited number of size fractions (as is obtained by the pipette method), enables a more detailed data analysis and a simultaneous use of the same data sets for classification of the analyzed samples under different national classification systems. Furthermore, PSD is used for the prediction of soil hydraulic properties (e.g., Bloeman, 1980; Arya and Paris, 1981). Because porosity and pore-size distribution in soils or other porous media are key parameters in the calculation of hydraulic properties, direct determination of PSD in terms of volume percentage by LD, rather than in terms of mass percentage as is the case in the pipette method, eliminates the need to adopt the rough approximation of a single value for soil particle density in the prediction process.

Main disadvantages are high cost of the LD instrument and insufficient confidence in the results due to the relatively low number of LD analyses of soils as compared with the enormous number of analyses performed by the classical methods. Entire texture-based classifications of soils are dependent on correlations that were established between soil properties and PSDs derived by classical methods. Correlations between PSDs obtained by LD and soil properties are yet to be established.

### The Effects of Nonsphericity and Uncertainty in Particle Density on Particle-Size Distribution Analysis

Portrayal of irregularly shaped particles, such as those found in soils, requires a complex three-dimensional description. For simplicity and ease of analysis, particles in soils are presented as an equivalent spherical particles, which allows the use of a single length (apparent diameter) as the descriptor. This simplification leads to a dependence of the obtained PSD on the method used for its determination (Mathews, 1991).

In the case of sieving, for example, the likelihood of a nonspherical particle to pass through or be retained

on a sieve of a given mesh size depends on the particle's shape and the probability of the particle to assume, during the time allotted for sieving, an orientation relative to the sieve that will allow it to pass through. Such an orientation exists for particles whose smallest cross-section can clear through the sieve's aperture. The net outcome of the nonsphericity of soil particles is, as a rule, that a coarser population is retained by the sieve than the actual population of particles with apparent diameters corresponding to the sieve size (Mathews, 1991). Exceptions to this rule may occur, for example, when the soil sample contains a significant quantity of very flat disk-shaped particles with a diameter exceeding that of the sieve aperture.

In sedimentation-based techniques the particle shape has the following effect. The most stable position of a settling nonspherical particle is the one in which the maximum cross-sectional area is perpendicular to the direction of motion (Krumbein, 1942). This position increases the expected particle drag, which, in turn, results in a decrease in the settling velocity (Mathews, 1991). Thus, the fine size fraction is overestimated.

When wet sieving through a sieve with a  $53\text{-}\mu\text{m}$  effective opening and sedimentation are combined for the determination of PSD (as is the case in the pipette method), an overestimation of the silt plus clay fraction at the expense of the sand fraction usually occurs during the sieving stage. Thereafter, during the sedimentation stage, the clay fraction is overestimated at the expense of the silt fraction.

An additional source of error in the sedimentation-based techniques is the heterogeneity in the particles' density. For soil and earth materials, particle density is commonly taken as  $2.65 \text{ Mg m}^{-3}$ . Yet, Clifton et al. (1999) found that the density of marsh sediment particles can vary between  $1.66$  and  $2.99 \text{ Mg m}^{-3}$ . The uncertainty regarding the actual density of the particles may strongly bias the size distribution in the sedimentation analysis.

Unlike PSDs derived from sedimentation-based techniques, a PSD measured by the LD method is independent of the density of the particles. On the other hand, LD derived PSD is also affected by the shape of the particles. The projected cross-sectional area of a nonspherical particle averaged over all the particle's possible orientations relative to the direction of the beam is larger than that of a sphere with an equal volume (Jonasz, 1991). This may lead to the assignment of a measured particle to a larger size fraction than it actually belongs to on the basis of its apparent radius; that is, a shift of the PSD toward its coarser fractions. It should be borne in mind however that for particles with an equivalent spherical diameter  $\leq 0.1 \mu\text{m}$ , the projected cross-sectional area becomes nearly the same as that of a sphere of equal volume (Jonasz, 1987).

In the present study, we performed a critical evaluation of the LD method and the combined sieve-pipette method for determining PSD of soils and assessed (i) whether a functional relationship existed between the two types of methods for determining PSD, and (ii)

the suitability of LD as a routine procedure for PSD determination in soil science.

## MATERIALS AND METHODS

Data from both methods were compared for 42 soil samples from California, representing a diversity of parent materials, climate and time of soil formation, and varying considerably in their PSD, particle shape, and mineralogy (Table 1). Samples from the cultivated layer (0–250 mm) of each soil were collected, air dried, and crushed to pass through a 2.0-mm sieve.

### Particle-Size Distribution Determination

Soil samples were not pretreated for removal of organic matter or carbonates before carrying out the PSD analyses.

#### Pipette Method

Forty-gram samples were dispersed overnight in a 50-g L<sup>-1</sup> sodium hexametaphosphate solution. Thereafter, the sand-size particles were separated from the suspensions by wet sieving through a 53- $\mu$ m sieve. The fraction retained by the sieve was dried and then weighed. The clay fraction was determined using the pipette method as described by Janitzky (1986). The silt fraction was determined by difference. Analyses were performed in triplicate.

#### Laser Diffraction

A Beckman-Coulter LS-230 with a 750-nm laser beam was used for PSD analysis; software version 3.01 was used for the calculation of the PSD. The instrument measures particle size over the range of 0.045 to 2000  $\mu$ m. The laser beam accurately measures particles of an apparent cross-sectional diameter >0.4  $\mu$ m (Buurman et al., 1997). For particles with an apparent cross-sectional diameter  $\leq$ 0.4  $\mu$ m, the LS-230 employs the polarization intensity differential of scattered light (PIDS) system, which uses polarized beams of 450-, 600-, and 900-nm wavelength. The PIDS system determines particle sizes between 0.1 to 0.6 times the wavelength of the polarized beam, thereby extending the measurement limit to 0.045  $\mu$ m (Coulter Co., 1994).

The calculation module offers the use of two optical models, the Fraunhofer diffraction model and the Mie theory. Because

the Fraunhofer model is not accurate enough for the determination of the clay-size fraction (Bayvel and Jones, 1981; de Boer et al., 1987), calculations based on the Mie theory were used. It should be borne in mind that the Mie theory applies rigorously to spherical, homogeneous particles and fits less satisfactorily nonspherical or nonhomogeneous particles (Jonasz, 1991) as are commonly found in soil. The Mie theory model requires, as an input parameter, the RI, which is a complex number comprised of (i) a real part ( $n_r$ ) which represents the change in the velocity of light through the tested material compared with the velocity of light in vacuum; and (ii) an imaginary term ( $n_i$ ) which represents the transparency and absorptivity of that material. Different RI values were tested to obtain the most suitable PSD.

For the PSD analysis, 0.1 to 0.5 g of soil was dispersed overnight in a 20-mL scintillation vial containing 10 mL of a 50-g L<sup>-1</sup> hexametaphosphate solution. Thereafter, individual dispersed samples were transferred to the fluid module that contained 1.7 L of deionized water ( $n_r = 1.33$  at 20°C) and subjected to a 1-min ultrasonication at energy level 3. Three to five replicate samples of each soil were then subjected to three consecutive 1 min runs at a pump speed of 8 to 12 L min<sup>-1</sup>.

To further compare the pipette method with the LD technique, we chose surface horizon samples from three representative soils varying considerably in their PSD: Clear Lake Series Fine, smectitic, thermic Xeric Endoaquerts; Yolo Series Fine-silty, mixed, superactive, nonacid, thermic Mollic Xerofluvents; and Auberry Series Fine-loamy, mixed, semiactive, thermic Ultic Haploxeralfs (Table 1: Soils No. 2, 22, and 35, respectively). From these soils we extracted the silt + clay fraction by wet sieving with a 53- $\mu$ m sieve. The <53- $\mu$ m particles were then placed in a 1-L settling cylinder as used in the pipette method. Immediately after homogenizing the suspension in the cylinder a sample was taken with a pipette and placed in the LD analyzer for PSD measurement. Thereafter, we rehomogenized the suspension in the cylinder and allowed for particles >2  $\mu$ m to settle. A sample was then taken with a pipette from a depth of 10 cm (to include only particles <2  $\mu$ m) to the LD analyzer and PSD analysis was performed. All measurements were performed in two replicates for each soil.

**Table 1.** The texture classes and the Great Group classification of the soils used for the study.

Soil no.†	Great group	Texture class‡	Soil no.†	Great group	Texture class‡
1	Endoaquerts	Clay	22	Xerofluvents	Silt loam
2	Endoaquerts	Clay	23	Durixeralfs	Loam
3	Haplodurids	Clay	24	Durixeralfs	Loam
4	Haploxerolls	Clay	25	Endoaqualfs	Loam
5	Haploxerolls	Clay	26	Haploxerolls	Loam
6	Haploxeralfs	Silty clay	27	Palehumults	Loam
7	Natrargids	Silty clay	28	Palexeralfs	Loam
8	Palexeralfs	Silty clay	29	Palexeralfs	Loam
9	Haploxeralfs	Silty clay loam	30	Argixerolls	Loam
10	Haploxeralfs	Silty clay loam	31	Argixerolls	Sandy loam
11	Natrixeralfs	Silty clay loam	32	Argixerolls	Sandy loam
12	Torrifluvents	Silty clay loam	33	Durixeralfs	Sandy loam
13	Torrifluvents	Silty clay loam	34	Haplocalcids	Sandy loam
14	Argixerolls	Clay loam	35	Haploxeralfs	Sandy loam
15	Haploxeralfs	Clay loam	36	Haploxerolls	Sandy loam
16	Haploxerolls	Clay loam	37	Palehumults	Sandy loam
17	Rhodoxeralfs	Clay loam	38	Torrifluvents	Sandy loam
18	Haploxeralfs	Sandy clay loam	39	Xerochrepts	Sandy loam
19	Haplagids	Sandy clay loam	40	Xerorthents	Sandy loam
20	Haploxeralfs	Silt loam	41	Haploxeralfs	Sand
21	Haploxeralfs	Silt loam	42	Torrifluvents	Sand

† Soils No. 2, 22, and 35 are the Clear Lake, Yolo, and Auberry soils respectively.

‡ Based on the pipette analysis.

RESULTS AND DISCUSSION

Refractive Index

When the Mie theory is used, choosing the correct RI for the optical model is important for ensuring the accuracy of the computed-size distributions. For the Auberry, Yolo, and Clear Lake soils, as the real part of the RI ( $n_r$ ) increases, the clay content decreases, and the silt and sand content increase (Fig. 1). The sensitivity of the PSD to  $n_r$  was most pronounced at  $n_r < 1.65$ . Jonasz (1991) showed that the scattering cross-section for a particle of a given size increases with the RI of the particle. The RI values of the minerals commonly found in soil fall between 1.48 and 1.71, but for some minerals like hematite, the RI may reach values of 2.9 to 3.2 (Table 2). Yet, for most minerals an  $n_r$  value of approximately 1.53 is suitable. For instance, Muggler et al. (1997) used  $n_r = 1.56$  for their deeply weathered Oxisol from Brazil. If the effect of coating of clay-size particles by organic matter and oxides on the RI is to be considered, an  $n_r$  value of 1.6 or higher should be employed. However, because LD analysis is likely to overestimate the size of nonspherical particles (e.g., Jonasz, 1991), one should use a somewhat lower  $n_r$  to counteract this effect. Therefore, we chose  $n_r = 1.5$  and  $n_i = 0.2$  for the optical model calculations. The PSD is less sensitive to  $n_i$  than to  $n_r$  but the above chosen values for the RI components resulted in a smooth PSD curve without artificial periodical interferences (Fig. 2).

The LS-230 enables the assignment of a separate  $n_r$

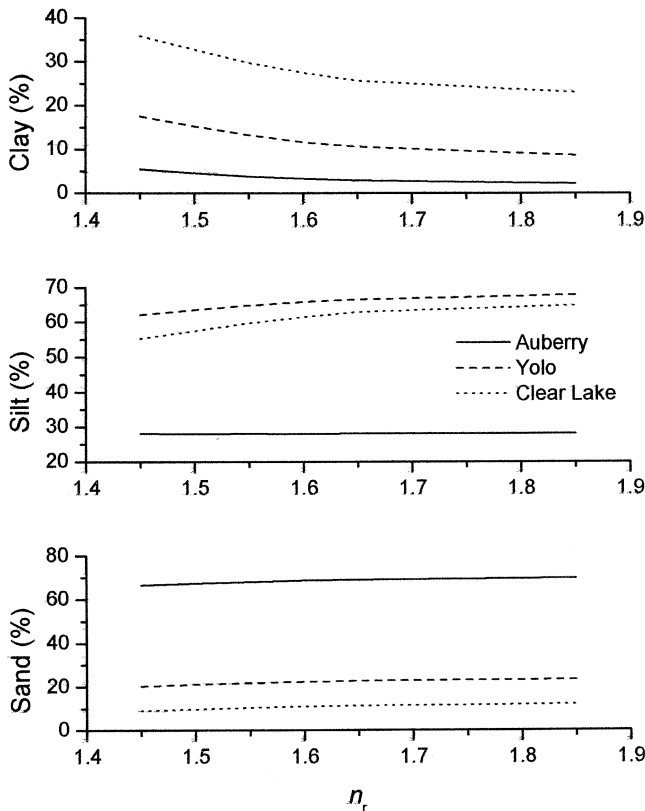


Fig. 1. Effects of the real part of the refractive index ( $n_r$ ) on the volume percent of (a) clay, (b) silt, and (c) sand in the Auberry, Yolo, and Clear Lake soils.

Table 2. The real part of the refractive index ( $n_r$ ) and particle density of common soil minerals. Unless stated otherwise, data were taken from the Handbook of chemistry and physics (CRC Press, 2002).

Minerals	$n_r$		Particle density Mg m <sup>-3</sup>
	589 nm		
Albite (Na feldspar)	1.525–1.536		2.61–2.64
Andesine	1.549–1.556		2.647–2.69
Anorthite	1.575–1.588		2.703–2.763
Apatite	1.65–1.648		3.1–3.3
Calcite	1.48–1.66		2.7
Feldspars	1.525†		2.5–2.8‡
Gibbsite	1.566–1.587		2.3–2.4
Gypsum	1.52–1.53		2.32
Hematite	2.91–3.19		4.9–5.3
Humus			<1.5‡
Illite	1.56–1.59		2.8
Kaolinite	1.549–1.565		2.65
Micas	1.53–1.7†		2.7–3.3‡
Microcline (K feldspar)	1.522–1.53		2.54–2.57
Montmorillonite	1.55–1.57		2.5
Quartz	1.544–1.553		2.65

† Beckman Coulter (1999).

‡ Gee and Bauder (1986).

value for particles  $\leq 0.4 \mu\text{m}$  that are determined by the PIDS procedure. Because the projected cross-sectional area of nonspherical small particles ( $< 0.1 \mu\text{m}$ ) is similar to that of equivalent spherical particles, the aforementioned need to choose a lower  $n_r$  no longer exists. In addition, the clay-size particles are frequently coated with oxides and organic matter whose  $n_r$  is higher than that commonly considered for clay minerals. Therefore, it might be advantageous to increase the  $n_r$  in the  $\leq 0.4$

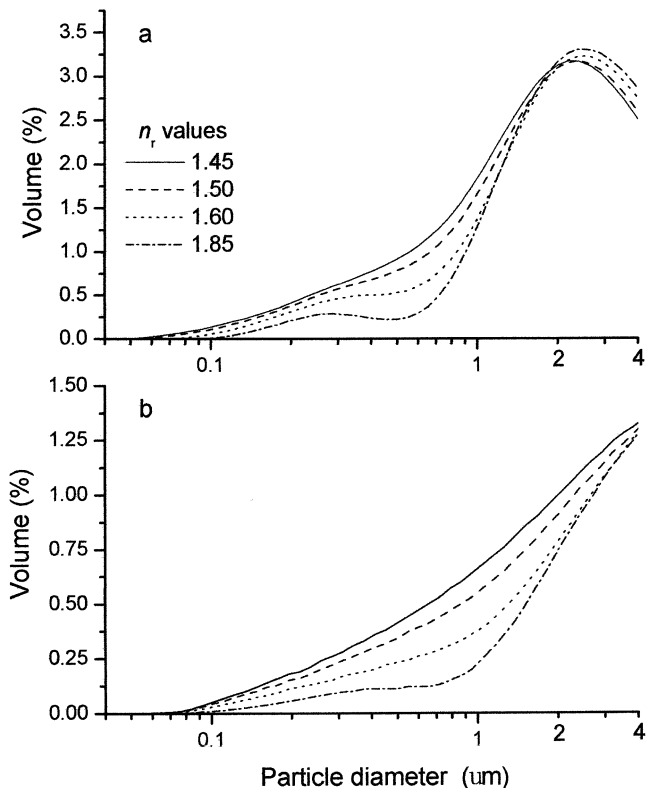


Fig. 2. Laser diffraction derived size frequency distribution (presented as volume percentage) of particles in the clay-size fraction for different values of the real part of the refractive index ( $n_r$ ) in (a) Clear Lake and (b) Yolo soils.

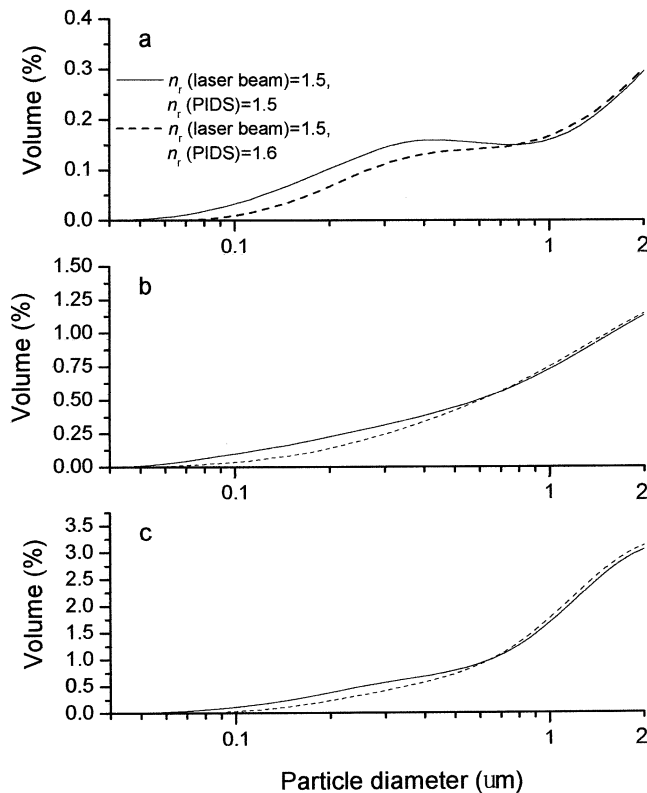


Fig. 3. Effects on the size frequency distribution (presented as volume percentage) of particles in the clay-size fraction of assigning different values for the real part of the refractive index ( $n_r$ ) for the regular laser beam and the polarization intensity differential of scattered light (PIDS) system. (a) Auberry, (b) Yolo, and (c) Clear Lake soils.

μm range to approximately 1.6. For the investigated soils, adopting the larger  $n_r$  value for the lower range of the PSD affected the curve's shape only in the part of the curve corresponding to clay particles <1 μm (Fig. 3). It led to a decrease of 0.7, 1.1, and 1.9% in the total volume of the clay fraction of the Auberry, Yolo, and Clear Lake soils, respectively. Even though the magnitude of the total clay fraction did not change substantially in the present case by the application of a larger  $n_r$  to the size region in which the PIDS procedure is employed, it is suggested that whenever this option exists a higher  $n_r$  value should be adopted in the PIDS as it may have a significant effect on the PSD in some cases (e.g., soils rich in oxides or organic matter).

### Comparison of Laser Diffraction to the Pipette Method in Determining Particle-Size Diffraction

The PSDs obtained by the pipette and the LD methods for the 42 investigated soils are presented in Fig. 4. It was noted that except for 2 of the 42 soils studied, the LD method yielded a smaller clay fraction than the pipette method (measured points are below the 1:1 line) (Fig. 4a). Conversely, the LD method yielded, in general, a higher proportion of silt than did the pipette method (measured points are above the 1:1 line) (Fig. 4b). In the case of the sand fraction, a trend was noted

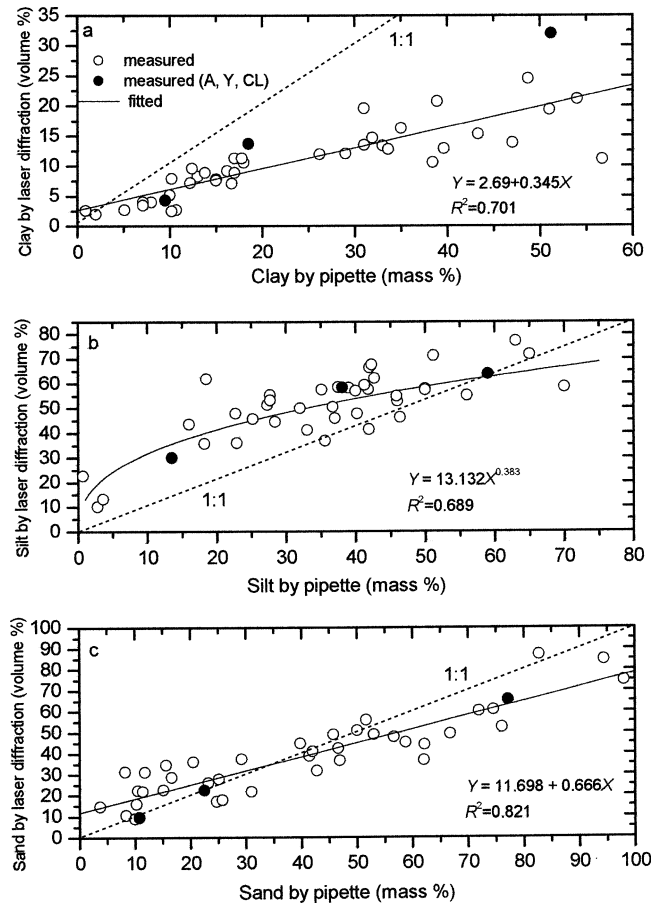


Fig. 4. Comparison of (a) clay, (b) silt, and (c) sand fractions obtained by the pipette (mass percentage) and the laser diffraction (volume percentage) methods for our 42 soils. The three soils marked with a filled symbol are Auberry (A), Yolo (Y), and Clear Lake (CL).

whereby at sand fractions <35% the LD method yielded a higher proportion of sand than the pipette method. The opposite was true for sand fraction >55% (Fig. 4c).

Although relationships were found between the LD derived and the pipette derived data for the three different size fractions, the scatter of the points around the fitted line for each size fraction was fairly wide. This type of scatter clearly implies that attempts to convert LD derived volume percentage of a given size fraction to mass percentage (or vice versa) will not be accurate and thus be of limited value. In addition to the less than satisfactory relation that we obtained between LD derived and the pipette derived data for the clay fraction (Fig. 4a), it differed from previously published data. Konert and Vandenberghe (1997) obtained for 158 samples a coefficient of determination of  $R^2 = 0.91$  for the relation  $y = 0.361x - 0.232$  where  $y$  was the volume percentage of the clay fraction obtained by the LD, and  $x$  was the mass percentage of the clay fraction obtained by the pipette. In another study, Van Dongen (1989, as reported by Konert and Vandenberghe, 1997) studied 68 samples and obtained a coefficient of determination of  $R^2 = 0.7225$  for the relation  $y = 0.185x + 0.662$ .

The above discussion about the relation between pipette and the LD data refers only to the clay-size fraction. To evaluate all three size-classes, we calculated the

**Table 3. Particle-size distribution (PSD) measured by laser diffraction, calculated PSD using the equations in Fig. 4, and the relative error of the calculated values (i.e., the difference between measured and predicted values expressed as the percentage of the measured value).**

Soil no.	Measured			Predicted			Relative error		
	Sand	Silt	Clay	Sand	Silt	Clay	Sand	Silt	Clay
	Volume %						%		
1	36.0	50.1	13.9	25.4	49.5	18.9	29.6	1.2	35.9
2	9.6	58.4	32.0	18.9	52.9	20.3	96.5	9.4	36.4
3	31.3	57.6	11.1	17.2	51.3	22.2	45.2	11.0	100.4
4	22.3	58.3	19.4	18.7	53.4	20.3	16.2	8.4	4.5
5	17.0	61.9	21.1	28.2	40.1	21.3	65.6	35.2	1.0
6	9.0	66.5	24.5	18.4	54.9	19.5	104.0	17.4	20.5
7	22.6	62.1	15.3	21.8	55.3	17.6	3.7	11.0	15.2
8	34.5	54.9	10.6	22.1	56.8	15.9	36.0	3.5	50.3
9	14.7	71.8	13.5	14.2	64.9	13.4	3.6	9.6	0.9
10	10.6	77.3	12.1	17.3	64.2	12.7	63.2	17.0	4.8
11	28.7	57.9	13.4	22.8	58.7	14.1	20.7	1.4	5.0
12	21.9	57.4	20.7	19.3	58.7	16.1	11.9	2.3	22.2
13	15.8	71.3	12.9	18.6	59.3	16.3	17.5	16.9	26.7
14	27.8	59.4	12.8	28.4	54.6	14.3	2.2	8.1	11.5
15	17.7	67.6	14.7	28.9	55.1	13.7	63.2	18.5	6.9
16	26.0	57.7	16.3	27.2	54.8	14.8	4.5	5.0	9.5
17	21.8	58.6	19.6	32.4	52.6	13.4	48.4	10.2	31.8
18	36.6	51.4	12.0	42.9	46.6	11.7	17.3	9.4	2.3
19	49.4	47.9	2.7	56.1	43.4	6.4	13.6	9.4	137.2
20	31.1	58.6	10.6	19.6	66.8	8.9	37.1	14.0	16.1
21	37.3	55.2	7.6	31.2	61.3	7.9	16.5	11.2	4.1
22	22.4	63.9	13.7	26.7	62.6	9.1	19.1	2.1	33.9
23	44.8	46.3	8.9	38.2	57.1	7.4	14.7	23.3	16.4
24	42.4	50.5	7.1	42.7	52.2	8.4	0.8	3.4	18.3
25	38.8	52.9	8.3	39.3	56.9	7.2	1.2	7.6	13.9
26	40.9	47.8	11.3	39.7	54.0	8.8	3.0	13.0	21.9
27	56.0	36.8	7.2	46.1	51.6	6.9	17.7	40.0	3.9
28	31.7	57.0	11.3	40.2	53.9	8.5	26.9	5.4	24.4
29	49.0	41.4	9.6	42.1	54.9	7.0	14.0	32.6	27.6
30	50.9	41.2	8.9	45.0	50.1	8.5	11.6	21.6	4.0
31	36.7	55.4	7.9	53.0	46.8	6.2	44.4	15.5	21.3
32	44.2	53.3	2.5	53.1	46.8	6.2	20.1	12.0	144.2
33	47.8	44.5	7.8	49.4	47.3	7.9	3.4	6.4	1.4
34	87.2	10.1	2.7	66.8	19.5	4.4	23.4	93.4	62.8
35	65.6	30.1	4.3	63.0	35.5	6.0	3.9	18.1	38.1
36	45.2	45.7	9.2	50.8	45.2	8.3	12.4	1.0	9.6
37	60.2	35.9	4.0	59.6	43.6	5.1	1.0	21.5	30.0
38	60.8	35.7	3.5	61.4	39.9	5.1	0.9	11.6	47.3
39	49	45.9	5.2	47.0	52.3	6.1	4.1	14.0	17.7
40	52.4	43.6	4.0	62.3	38.0	5.4	19.0	12.9	35.8
41	84.8	13.2	2.0	74.6	21.4	3.4	12.0	62.7	67.0
42	74.8	22.7	2.6	77.0	11.5	3.0	2.9	49.4	17.4

relative error, that is the absolute value of the difference between measured and calculated LD values (based on the equation presented in Fig. 4 for each size class) expressed as percentage of the measured value, for the sand, silt, and clay fractions (Table 3). The results indicated that even if the relative error was small for one size fraction, as was the case for the clay fraction in soils 5, 10, 15, and 27, the relative error was still high for one or both of the other fractions (Table 3). These observations strongly suggest that in individual studies LD data could at times be satisfactorily correlated with pipette data for a given size fraction, but no universal relation between PSD obtained by LD and that obtained by the pipette method can be formulated for the entire PSD range.

Our observations were also in disagreement with other studies. For instance, Beuselinck et al. (1998) noted that LD measurements do not always underestimate the magnitude of the clay fraction as compared with analyses performed by the pipette procedure. Their study showed that while LD did underestimate the size of the clay fraction in silty soil samples, it overestimated the content of clay-sized particles in milled quartz sam-

ples. These authors suggested that regression equations derived by reduced major axis analysis could be used to define the relation between PSDs derived by the sieve-pipette method and by LD for a given set of soils. They did, however, emphasize that there is no unique relationship between the PSDs derived by LD and by the sieve-pipette method. It was concluded that this relationship appears to be most strongly affected by variations in mineralogy and morphology over the various particle-size classes (Beuselinck et al., 1998). Vitton and Sadler (1997), who compared PSDs obtained by the sedimentation-based hydrometer procedure with PSDs obtained by LD, reported a satisfactory agreement between results derived by the two methods, with the agreement improving as mica content in the soils decreased. Muggler et al. (1997) also reported reasonable agreement in PSDs obtained by the pipette and the LD methods for soils containing <60% clay.

To further examine the differences in PSD between the pipette and the LD methods, we examined the PSD using the LD of the silt + clay (<53  $\mu\text{m}$ ) and clay fractions (<2  $\mu\text{m}$ ) of the Auberry, Yolo, and Clear Lake soils that were isolated by wet sieving and the pipette

**Table 4. Volume percentage determined by laser diffraction of samples from the soils investigated that were initially isolated by sieving (particles <53  $\mu\text{m}$ ) and by the pipette (particles <2  $\mu\text{m}$ ). Number after  $\pm$  represents one standard deviation.**

Soil	Volume percentage	
	<53 $\mu\text{m}$	<2 $\mu\text{m}$
Auberry	91.9 $\pm$ 0.14	52.5 $\pm$ 0.21
Yolo	96.0 $\pm$ 0.07	58.7 $\pm$ 0.42
Clear Lake	98.3 $\pm$ 0.14	60.9 $\pm$ 1.48

method, respectively (Table 4). In all three soils, after sieving, the volume percentage of the <53- $\mu\text{m}$  fraction was <100%, thus indicating, as discussed above, that during the sieving part of the combined sieving–pipette method, particles >53  $\mu\text{m}$  passed through the sieve and were thus no longer considered as sand particles. Concerning the clay-size fraction (<2  $\mu\text{m}$ ), only approximately 57% by volume of the samples were determined to be within the clay-size fraction, while approximately 43% of the particles were in the size fraction >2  $\mu\text{m}$  (Table 4). These results are similar to those of Clifton et al. (1999), who analyzed marine sediments and found that approximately 35% of the clay-size particles obtained by settling were considered coarser than 2  $\mu\text{m}$  by a LD determination. These results further highlight the consistent lack of agreement regarding the clay fraction between the sedimentation-based pipette method and the LD technique.

The varying and at time conflicting results appearing in the literature with regard to the comparability of PSDs obtained by the pipette–hydrometer method to those obtained by LD, may originate from two sources. First, there are biases, or sources of error, inherent in the sedimentation-based and in the LD methods. These sources of error, which were discussed earlier, dictate predictable differences between the PSDs measured by the two methods. Second, the LD analysis is independent of the particles' density and accordingly produces PSDs expressed in volume percentage. In the sedimentation-based methods the determined particle size is dependent on the particle's density, which in the case of soil is approximated by an assumed mean value. The PSD is expressed in this case in terms of mass percentage. The deviation of the true density of the particle from the assumed mean density is a source of error that is specific to the analyzed soil. The mere assumption of a single value for particle density in soils, as is presumed for the purpose of size determinations by the pipette method, is an obvious source of error. Particle density of soil components may vary between soils and among the different size fractions in a given soil (e.g., Table 2).

The difference between a PSD obtained by LD and the one obtained by sedimentation for a given soil is dependent in a complex fashion on the properties of the soil and especially on its mineralogy (that determines, e.g., the RI and the density) and morphology (that affects the shape, or deviation from sphericity) of the soil particles. The overall consequence of the predictable, procedure-dependent sources of error inherent in the PSD determinations by the two methods and the harder to estimate soil-dependent sources of

error is that no consistent relationship between PSDs derived by LD and PSDs derived by sedimentation methods can be formulated. This is so, despite some recurring features of the differences in the derived PSDs (e.g., a lower clay fraction produced by the LD method).

Finally, it should also be born in mind that disparities between measured PSDs may also occur when different LD apparatus are used. Loizeau et al. (1994), for example, reported a discrepancy between PSDs obtained by two LD instruments for particles <10  $\mu\text{m}$ . This discrepancy was probably the result of using two instruments with different detection limits. Not only may LD instruments differ in their detection systems, but the optical model employed for PSD determination may also be different. In some studies the Fraunhofer diffraction model was used (e.g., Loizeau et al., 1994; Konert and Vandenberghe, 1997; Beuselinck et al., 1998), while in others the Mie theory was used (e.g., Buurman et al., 1997; Muggler et al., 1997). Differences inherent in the two optical models, and especially the fact that the Fraunhofer model has difficulties in calculating the magnitude of particles in the size range of the laser beam wavelength or smaller, may affect the outcome of the PSD determination for a given sample.

## CONCLUSIONS

We compared the PSD between the pipette and the LD method for 42 soils. For the purpose of conversion of data from one type of measurement to the other, the relationship between the pipette data and the LD data for the different size fractions was less than satisfactory. In addition, the relationship between the pipette- and the LD-derived clay fraction in our study differed from those appearing in the literature. Furthermore, in many of the soils that exhibited good agreement between measured and calculated LD values for the clay fraction, poor agreement between measured and calculated values existed for the silt or sand fraction.

It should be realized that there is no method for PSD determination of soil materials that can serve as a universal yardstick, because all available methods, whether classic (e.g., pipette) or new (e.g., LD), suffer from some inherent flaws. The choice between methods depends, therefore, on the balance between the pros and cons of each. Advantages of the LD procedure over the pipette method include (i) need for only a small sample, (ii) short time of analysis, and (iii) a continuous PSD curve.

Compared with the pipette method, the LD procedure suffers from two main disadvantages. One is the high cost of the instrumentation. However, with the increase in cost of labor and the constant pressure for greater reliability, reproducibility, and speed of analysis, the attractiveness of LD apparatus is expected to grow. The second disadvantage is the lack of a database that correlates LD-derived PSDs with soil properties, similar to the very extensive database existing for pipette-derived PSDs. Nonetheless, should the LD method become more accepted in the soil science community, the well needed database will gradually be established.

We believe that the LD method and the resultant expression of PSD based on volume is a valid method even though it does not provide data that are fully comparable with data derived by classical methods. Because of the speed, small sample size, and range of output options available with laser diffraction, we foresee the method becoming more widely used for PSD.

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