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TECHNICAL ARTICLES

A FAST METHOD FOR DETERMINING SOIL PARTICLE SIZE DISTRIBUTION USING A LASER INSTRUMENT

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The sieve-pipette is the standard method for determining soil particle size distribution (PSD) because it is precise and reproducible. However, this method requires considerable time. Light diffraction methods for determining PSD are fast, but there is no standard procedure and often, results do not agree precisely with the pipette. The objective of this study was to develop a simple and fast procedure for sample handling and treatment of light diffraction method. A commercially available laser-light diffraction instrument was used. Soil samples were loaded dry into the instrument for ease and speed. A combination of chemical and physical dispersion within the instrument was found to be convenient and effective. Time required to analyze a sample was at most 15 min. Reproducibility between different operators was good, with S.E. ranging from 0.2% to 3.6%. Furthermore, we attempted to identify optimal values for the real refractive index and imaginary refractive index used in the optical model for light diffraction. Values of 1.42 and 0.001 for real refractive index and imaginary refractive index, respectively, were found to give acceptable results when compared with the pipette method. The light diffraction method was not significantly different from the pipette method for sand ($P = 0.084$), silt ($P = 0.743$), and clay ($P = 0.052$). Correlation between the light diffraction and pipette method for sand, silt, and clay was acceptable ($R^2 = 0.88, 0.80, \text{ and } 0.69$, respectively). The light diffraction technique does not have a perfect agreement with the pipette method, but it provides data rapidly and was reproducible. This method can be very valuable when a large number of samples need to be analyzed for relative comparisons between different sites. (Soil Science 2006;171:663-674)

Key words: Soil particle size distribution, pipette method, light diffraction, soil texture.

THE sieve-pipette method is often used in soil science as a standard method from which to compare other particle size distribution (PSD) methods (Gee and Or, 2002). Results obtained with the sieve-pipette method are precise and reproducible. The sieve-pipette

method is based on Stokes law (Gee and Or, 2002). The most important assumption in the application of Stokes law is that soil particles behave like spheres. However, sand particles have sharp angular edges (Wilding et al., 1977), and clay particles resemble stacked paper sheets rather than marbles (Hurlbut, 1971). Furthermore, Stokes law is valid for a single particle that is settling slowly in a fluid without the interference of other forces or motions. Therefore, laminar flow must be maintained, which presents a problem for larger particles because they create turbulence as they settle and for smaller particles because they can be disturbed by Brownian

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motions (Allen, 1981; Vitton and Sadler, 1997; Xu, 2002). In addition, at high particle concentrations, there are interactions and interferences between particles (Allen, 1981; Vitton and Sadler, 1997; Xu, 2002). On the practical side, the application of Stokes law to the sieve-pipette method works well for analysis of soil-particle size distribution (Indorante et al., 1990; Gee and Or, 2002). However, settling times for small particles, such as clay, are long (~8 h).

In the sieve-pipette method, the sand fraction is determined by sieving, increasing the time needed for analysis. Because the orifices of most sieves are square, the geometry and orientation of the particles play an important role (Allen, 1981; Matthews, 1991; Xu, 2002). The main drawbacks of the sieve-pipette method include the difficulty of setting up the equipment properly, time of analysis, and differences in operators' skills (Indorante et al., 1990). Laboratory technique and operator error can have a significant impact on results obtained with the sieve-pipette method (Syvitski et al., 1991). Advantages include relative low cost of the equipment, high precision, and reproducibility.

An alternative method for PSD determination is laser-light diffraction. Laser-light diffraction technology may fulfill the need for a fast and reproducible method for PSD determination. One of the initial problems associated with light diffraction techniques was that the detection ranges of available instruments were not adequate to cover the complete range of particle-size limit classification established by the United States Department of Agriculture (USDA). New instruments and technologies have reduced this problem. Another drawback is cost, but the expense of purchasing the equipment could be offset in the long run by the large number of samples that can be analyzed and reductions in labor per sample.

Light diffraction methods for measuring soil and sediment PSD have been investigated by many scientists with mixed results (Loizeau et al., 1994; Buurman et al., 1997; Konert and Vandenberghe, 1997; Muggler et al., 1997; Beuselinck et al., 1998; Arriaga et al., 2000; Eshel et al., 2004; Zobeck, 2004). The main problems with light diffraction techniques at the moment are the lack of a simple, fast, standard sample treatment procedure and the low correlation with the sieve-pipette method. The principal advantage of laser methods is that sample analysis can be performed fast, almost 50 times faster than the sieve-pipette, increasing

the number of samples that can be analyzed. In addition, the amount of sample needed for analysis is greatly reduced. Laser methods can be useful in situations where PSD needs to be determined for a large number of samples from one location, such as in landscape studies.

The main advantage of laser-light methods is speed of analysis; therefore, the objective of this work was to develop a quick and simplified protocol for preparing soil samples and performing PSD measurements with a laser-light scattering device. In addition, optimal values for the refractive indices used in the optical model were determined to match PSD to the sieve-pipette method as close as possible.

MATERIALS AND METHODS

Soil samples from seven different states in the United States were used to test the proposed protocol (Table 1). These soils covered a large range of soil orders and various PSD. In addition, a set of six constructed samples was tested. These samples were created by decanting different soil fractions (i.e., silt and clay) from a Dubuque silt loam soil (fine-silty, mixed, mesic, Typic Hapludalfs) (Starr et al., 2000). The sand was removed by sieving and set aside. The pure sand, silt, and clay were mixed in different proportions to obtain the set of six samples (Table 2). The sieve-pipette method as described by Gee and Or (2002) was performed for silt and clay determination, whereas the sand fraction was determined by sieving. The sieve-pipette analysis was conducted for all soil samples at the USDA-NRCS Soil Survey Laboratory located in Lincoln, Nebraska.

When developing the analytical procedure for PSD using the light diffraction instrument, soil pretreatment and handling was kept as simple as possible to stay within the intent of the principal objective of this work. Sample preparation, including carbonate and organic matter removal, is the same as for the sieve-pipette method (Gee and Or, 2002). Soil samples were stored dry in Whirl-Pak® bags that measured 7.6 by 12.7 cm (NASCO, Fort Atkinson, WI), or similar bags, after preparation.

Laser-light Diffraction Instrument

A Coulter LS230 (Beckman-Coulter Inc., Miami, FL)¹ with a fluid sampler module and

¹ Mention of company or product name does not constitute endorsement by the USDA-ARS, NRCS or the University of Wisconsin-Madison to the exclusion of others.

TABLE 1
Location, taxonomic, and textural classification of soils analyzed by light scattering and sieve-pipette methods

ID*	Taxonomic classification	State	Textural classification
1	Fine-silty, mixed, mesic, Typic Hapludalfs [†]	Wisconsin	Silt loam
2	Fine-silty, mixed, mesic, Typic Hapludalfs	Wisconsin	Silt loam
3	Fine-silty, mixed, mesic, Typic Hapludalfs	Wisconsin	Silt loam
4	Fine-silty, mixed, mesic, Typic Hapludalfs	Wisconsin	Silt loam
5	Fine-silty, mixed, mesic, Typic Hapludalfs	Wisconsin	Silt loam
6	Fine-silty, mixed, mesic, Typic Hapludalfs	Wisconsin	Silt loam
7	Fine-silty, mixed, mesic Typic Haplaquolls	Wisconsin	Silt loam
8	Fine-silty, mixed, mesic Typic Haplaquolls	Wisconsin	Silt loam
9	Fine-silty, mixed, mesic Typic Haplaquolls	Wisconsin	Silty clay loam
10	Fine-silty, mixed, mesic Typic Argiudolls	Wisconsin	Silt loam
11	Fine-silty, mixed, mesic Typic Argiudolls	Wisconsin	Silty clay loam
12	N/A	Iowa	Clay loam
13	Fine-loamy, mixed, mesic Aridic Haplustalfs	New Mexico	Loam
14	Fine-silty, mixed, superactive, mesic Oxyaquic Argiudolls	Illinois	Sandy clay loam
15	Mixed, mesic Aquic Udipsamments	Indiana	Sand
16	Fine-loamy, mixed, mesic Aridic Haplustalfs	New Mexico	Sandy loam
17	Fine-loamy, mixed, superactive, thermic Typic Haploxeralfs	California	Silt
18	Fine-loamy, mixed, superactive, thermic Typic Haploxeralfs	California	Silty clay loam

*Sample identification number.

[†]Taxonomic and textural classifications of Samples 1 to 6 represent the original soil used to obtain the separate soil fractions to make the constructed soil samples.

N/A indicates information not available.

connected to a Windows-based computer was used for this work. This instrument uses a laser-light diffraction technique for measuring the diameter of particles between 2000 and 0.4 μm in size. To extend its range to 0.04 μm and improve the resolution from 2000 to 0.8 μm , the LS230 relies on a polarization intensity differential scattering (PIDS) measurement technique. These two readings are combined to produce a continuous particle size distribution curve for particles from 2000 to 0.04 μm . Samples in the LS230 are suspended in a liquid, in this case water, and circulated via a centrifugal pump during sample loading and analysis.

Tap water was used in the LS230 instead of distilled water because this instrument has sensors that rely on electrical conductivity to control the level of water in the sample vessel and prevent overflow. The tap water was softened to aid soil dispersion and avoid flocculation that can be caused by Ca and Mg in the water and to avoid calcification of internal parts of the instrument. For this purpose, a residential style water conditioner system set to 2500 grains of hardness and a disposable filter assembly rated at 0.2 μm were used (Pall Corporation, East Hills, NY). The sample vessel has a capacity of about 1.7 L.

TABLE 2
Particle size distribution determined by mass, sieve-pipette, and light scattering for the constructed soil samples

ID*	Mass			Pipette			Light scatter			S.E. [†]		
	Sand	Silt	Clay	Sand	Silt	Clay	Sand	Silt	Clay	Sand	Silt	Clay
1	35.1	59.9	5.0	37.3	57.8	4.9	51.1	43.6	5.6	5.0	5.1	0.1
2	60.3	34.7	5.0	62.4	33.1	4.5	61.5	28.3	10.2	0.6	1.9	1.8
3	25.1	59.9	15.0	29.0	56.0	15.0	32.0	50.7	17.3	2.0	2.7	0.8
4	59.9	25.0	15.1	61.6	24.5	13.9	52.2	27.5	20.3	2.9	0.9	2.0
5	10.3	59.8	29.9	11.8	58.4	29.8	17.6	48.4	34.0	2.2	3.6	1.4
6	60.0	10.0	30.0	65.5	7.6	26.9	46.8	19.8	33.4	5.5	3.7	1.9

*Sample identification number.

[†]Standard errors between the mass, sieve-pipette, and light scattering particle size determinations.

TABLE 3

Amount of soil sample needed for the light diffraction instrument to reach a proper obscuration level to make a reading varied with the particle size distribution of the soil

Sample	Size fraction (%)			Amount of soil needed (g)
	Sand	Silt	Clay	
Fine sand	100	0	0	8.3
Silt	0	100	0	0.87
Clay	0	0	100	0.31
Sample A*	55.4	37.1	7.5	1.0
Sample B	37.2	37.1	25.7	0.69

*Particle size distribution for Samples A and B determined by the sieve-pipette method; USDA-NRCS Laboratory, Lincoln, Nebraska. Sample A is listed as ID 17 in Table 1, whereas sample B is not listed in Table 1 (taxonomic classification not available).

Sample Analysis

The following steps were followed when analyzing samples with the light diffraction device. (i) With the sample vessel filled with about 1.5 L of water and the pump running at 62% ($\sim 10 \text{ L min}^{-1}$), 100 mL of a solution of 50 g of sodium hexametaphosphate per liter of water was added. (ii) Once the dispersant was mixed with the water, a background reading was taken. This background reading was compared with a standard background reading that was saved in the computer. (iii) Dry soil sample was added in small increments, usually about 10 increments, until the obscuration and PIDS readings were

from 5% to 10% and 45% to 48%, respectively. The amount of sample needed for the instrument to take a reading varied as a function of the PSD of the soil to be analyzed (Table 3). The dry sample was mixed in the Whirl-Pak[®] bag before sample loading by rotating the bag about 10 times along its short and then long axis. The bag was then opened carefully, and a small spatula was used to carefully mix the soil sample to assure that a representative portion of the soil sample was added into the instrument. (iv) The sonicator was turned on for 480 sec. (v) Data from the detectors were collected for 90 sec. (vi) An optical model was applied to the data, and a

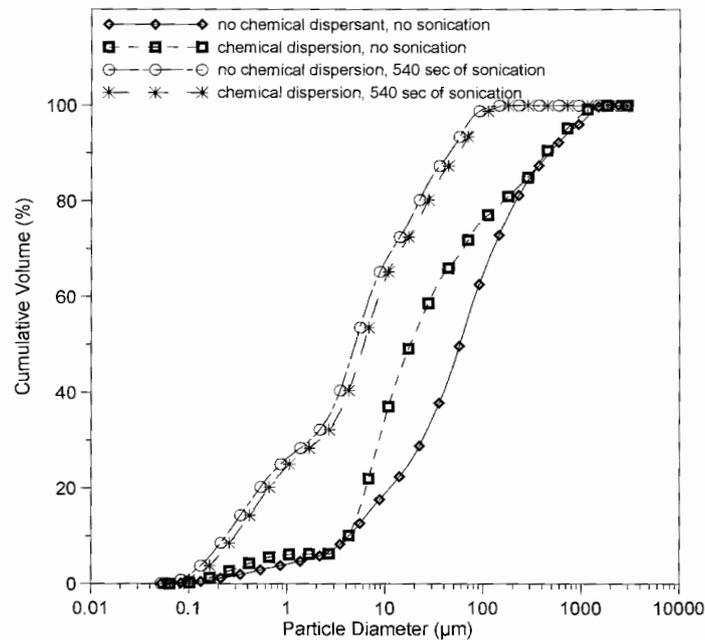


Fig. 1. Effect of sodium hexametaphosphate and sonication on dispersion of a soil sample. The symbols represent every fifth data point.

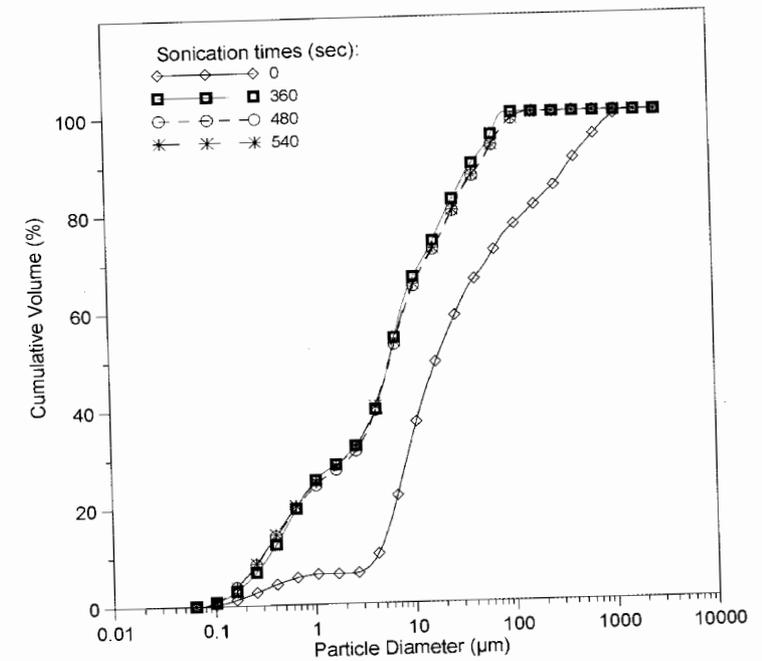


Fig. 2. Sonicating a soil sample for 360 sec yielded similar results as sonicating for 540 sec. No chemical dispersion used. The symbols represent every fifth data point.

continuous curve of PSD was created. Following these steps, a sample can be analyzed every 12 to 15 min including the time needed to clean the instrument between samples.

Refractive Indices and the Optical Model

Data collected by the different detectors in the instrument were in raw format and needed

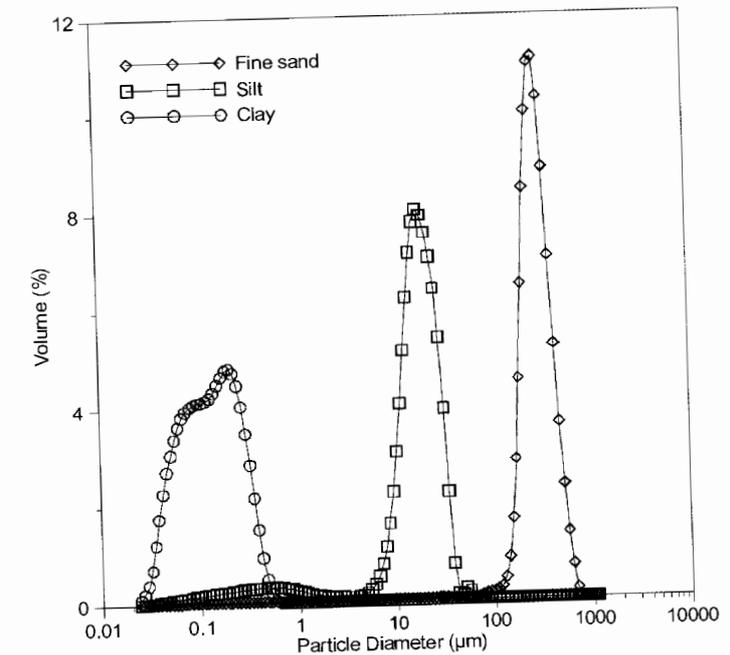


Fig. 3. The components (sand, silt, and clay) for the constructed soil samples were readily detected with the light diffraction instrument when analyzed separately.

to be converted mathematically using an optical model into particle sizing. There are two optical models commonly used, the Fraunhofer and Mie theories. The Mie theory is based on electromagnetic theory of scattering developed by Gustav Mie and published in 1908 (Williams, 1968). The mathematics involved are complex, and not until computers were available was the Mie theory used and applied with success. For this reason, the Fraunhofer diffraction approximation has been widely used (Agrawal et al., 1991). However, the Fraunhofer theory is a limiting case of the Mie theory which is only applicable when particles are large relative to the light wavelength (de Boer et al., 1987). Furthermore, de Boer et al. (1987) concluded that for laser diffraction instruments, the Mie theory better describes the size of particles that are suspended in a liquid. Therefore, the Mie theory was used as the optical model for this study.

The optical model using the Mie theory requires the input of three refractive indices. A refractive index for the liquid used to suspend the particles, in this case water, which has a refractive index of 1.33. The other two indices needed are the real refractive index (RRI) and imaginary refractive index (IRI). These values are mainly affected by mineralogy and color. Values for the RRI and IRI directly affect the determination of the different soil fractions with the light diffraction method and their correlation to the sieve-pipette method. For this reason, values for the RRI and IRI to match as close as possible the results from the sieve-pipette method were used. Data collected with the light diffraction instrument were analyzed using several values for the RRI and IRI to determine optimal values for both parameters. Further description of this process will be covered in the Results and Discussion.

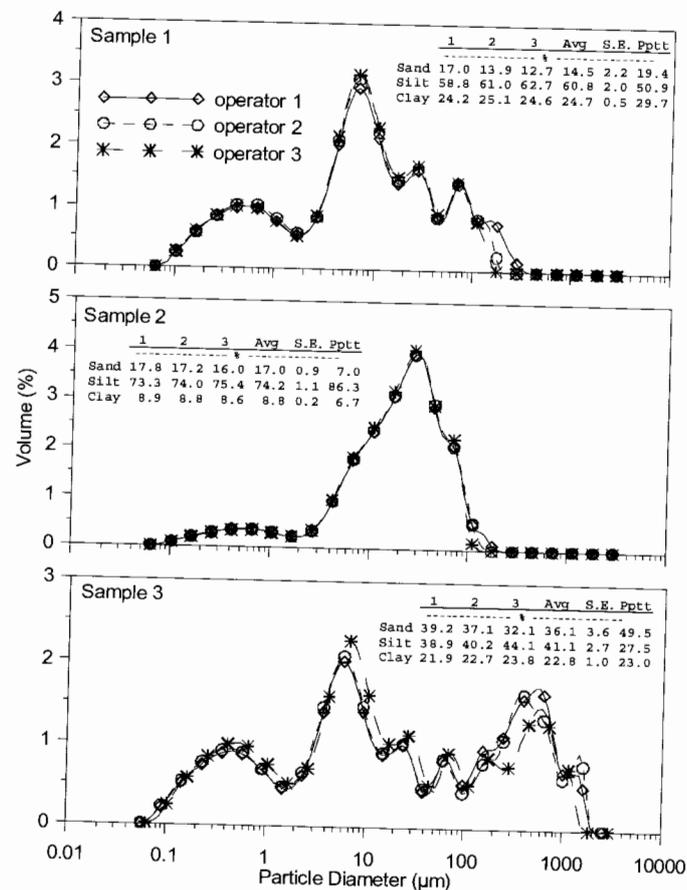


Fig. 4. Reproducibility between operators of the proposed procedure for PSD by light diffraction was acceptable. Avg indicates average particle content from the operators; Ppnt; particle content from sieve-pipette method listed as a reference. The symbols represent every fifth data point.

Statistical Analysis

The laser-light method proposed here and the sieve-pipette method were compared using a paired-sample *t* test (Zar, 1984). Data were tested for normality before any statistical analysis was conducted using the D'Agostino-Pearson test (Zar, 1984). A lack of significant difference ($P > 0.05$) denotes no statistical difference between the two methods. Statistical analysis was performed using CoStat statistical software (CoHort Software, Monterey, CA).

RESULTS AND DISCUSSION

Analysis Procedure

As stated in the Materials and Methods, the soil samples were introduced into the instrument dry. Because the amount of soil needed for the instrument to reach adequate obscuration levels varied from sample to sample, the soil samples were introduced dry to maintain the simplicity and speed of the procedure, which is in accordance with the main objective of this work. Another option explored was to introduce the soil sample in solution after shaking overnight in sodium hexametaphosphate (Eshel et al., 2004; Zobeck, 2004), but this presented a

problem because sand particles settled out quickly before the sample was introduced in the machine, making it difficult to obtain a representative subsample. Thus, dry samples were placed into the sample vessel and dispersed in the instrument before taking the reading. The volume of water in the system was not very critical, but using a near to maximum amount of water in the sample vessel allowed for a larger subsample of soil to be used for each analysis. The proper pump speed to keep all particles in suspension, including sand, was determined by observing changes in the obscuration values while changing the pump speed. The centrifugal pump was operated at 62% of its capacity, about 10 L min^{-1} .

Sample dispersion of the soil was achieved by a combination of chemical and physical means. Chemical dispersion was done by adding 100 mL of 50 g L^{-1} of sodium hexametaphosphate solution to the sample vessel before obtaining a background reading. Physical dispersion was performed with the built-in sonicator of the instrument as well as the action of the pump. Chemical dispersion with no sonication was almost as effective as sonicating for 540 sec with no chemical dispersant added (Fig. 1). Several

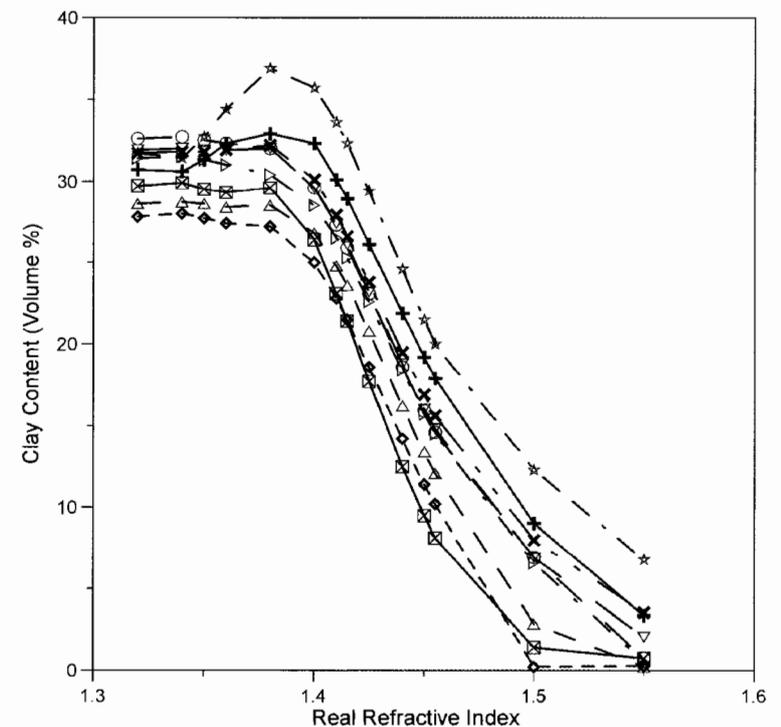


Fig. 5. Clay content prediction by the optical model reached a maximum at an RRI of approximately 1.4. Each line represents a different soil.

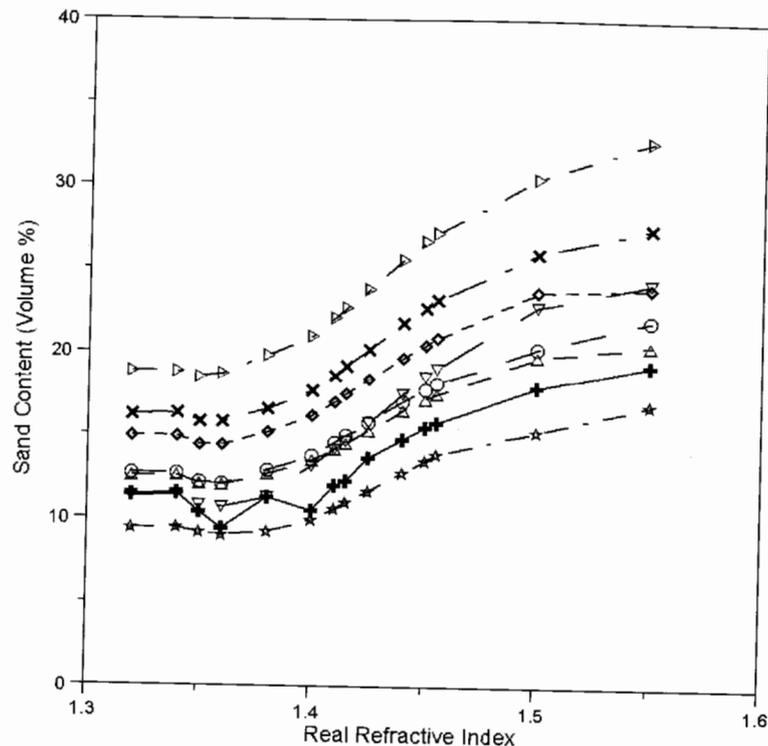


Fig. 6. Sand content as predicted by the optical model increased with increasing RRI values. Each line represents a different soil.

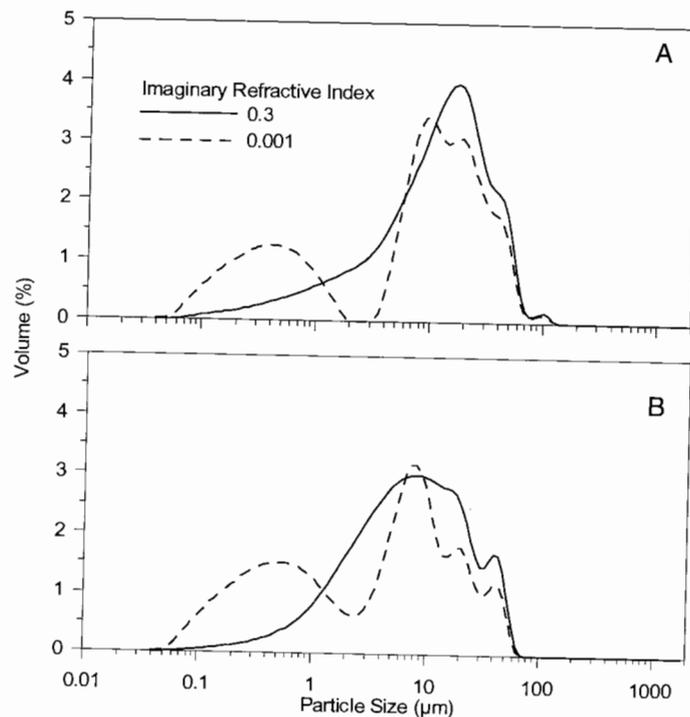


Fig. 7. A small value for the IRI helps identify the particles in the clay size range more readily. Two different soil samples (graphs A and B) with a high silt content were used to evaluate the effect of IRI.

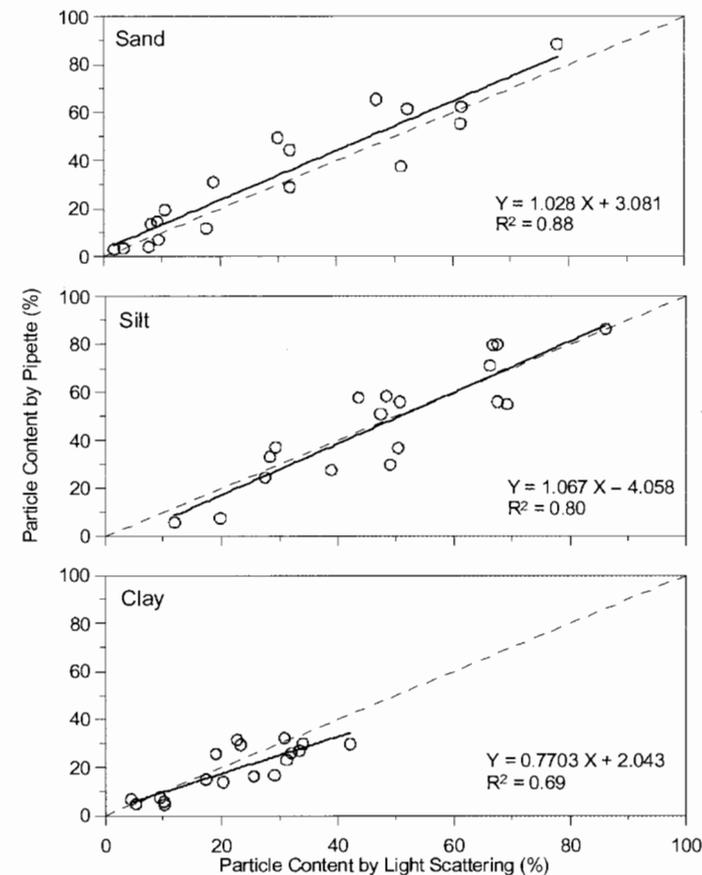


Fig. 8. The proposed light scattering procedure predicted soil fractions as measured with the sieve-pipette method with some confidence. Each symbol represents one sample.

sonication times were evaluated, with 360 sec of sonication being adequate for most soils tested; however, we recommend sonication for 480 sec to assure proper dispersion (Fig. 2). After sample dispersion, the detectors were set to take readings continuously for 90 sec. Total time required to analyze a sample, including cleaning the sample vessel after analysis, was approximately 12 min.

The detection range of the instrument tested falls within the range of PSD of most soils. Sand, silt, and clay fractions used to construct soil samples (see Table 2) were readily identified when analyzed separately (Fig. 3). Soil samples were evaluated several times by different operators to establish reproducibility of the method. Reproducibility of the proposed procedure was good, with S.E. ranging from 0.2% to 3.6%. Figure 4 shows differences between replicate runs of same soils, including dry sample loading, conducted by different operators.

Determination of the Real and Imaginary Refractive Indices

While trying to find the best possible RRI and IRI values to compare the PSD obtained with light diffraction to the sieve-pipette method, it was noted that the instrument generally overestimated the silt fraction and underestimated the clay fraction. This is in agreement with the findings of Arriaga et al. (2000) and Eshel et al. (2004). Therefore, different values for RRI and IRI were tested for several samples to determine which values provided the greatest clay prediction possible. A value of 1.40 for the RRI typically gave the greatest clay content prediction by the optical model (Fig. 5). Similarly, sand content as predicted by the optical model peaked around an RRI value of 1.54 (Fig. 6). With these two limits established for RRI, several values of RRI

TABLE 4

Particle size analysis as determined by the sieve-pipette and light diffraction methods and the difference between measurements for sand, silt, and clay

Sample	Pipette			Light diffraction			Difference*		
	Sand	Silt	Clay	Sand	Silt	Clay	Sand	Silt	Clay
1 [†]	37.3	57.8	4.9	51.1	43.6	5.3	13.8	-14.2	0.4
2	62.4	33.1	4.5	61.5	28.3	10.2	-0.9	-4.8	5.7
3	29.0	56.0	15.0	32.0	50.7	17.3	3.0	-5.3	2.3
4	61.6	24.5	13.9	52.2	27.5	20.3	-9.4	3.0	6.4
5	11.8	58.4	29.8	17.6	48.4	34.0	5.8	-10.0	4.2
6	65.5	7.6	26.9	46.8	19.8	33.4	-18.7	12.2	6.5
7	3.0	71.0	26.0	1.8	66.2	32.0	-1.2	-4.8	6.0
8	3.4	79.9	16.7	3.4	67.5	29.1	0	-12.4	12.4
9	13.7	54.9	31.5	8.1	69.2	22.7	-5.7	14.3	-8.7
10	4.0	79.7	16.3	7.7	66.7	25.6	3.7	-13.0	9.3
11	14.7	56.0	29.4	9.2	67.5	23.3	-5.5	11.5	-6.0
12	19.4	50.9	29.7	10.5	47.4	42.1	-8.9	-3.5	12.4
13	7.0	86.3	6.7	9.4	86.1	4.5	2.4	-0.2	-2.2
14	44.4	29.9	25.7	32.0	49.0	19.0	-12.4	19.1	-6.7
15	88.6	5.8	5.6	78.0	11.9	10.1	-10.6	6.1	4.5
16	49.5	27.5	23.0	29.9	38.9	31.2	-19.6	11.4	8.2
17	55.4	37.1	7.5	61.3	29.3	9.4	5.9	-7.8	1.9
18	31.0	36.8	32.2	18.8	50.4	30.8	-12.2	13.6	-1.4
				Mean [‡]			-3.9 (8.8) [§]	0.8 (10.5)	3.1 (6.0)

*A positive difference value indicates that the light diffraction method overestimated the percent particle size for that soil fraction, whereas a negative value indicates an underestimation.

[†]Samples 1 to 6 are the constructed samples listed in Table 2.

[‡]Mean of the differences for each soil fraction.

[§]Numbers between parentheses indicate the S.D.

between 1.40 and 1.54 were tested. An RRI of 1.42 seemed to produce good results and was compared with the sieve-pipette method. This discussion follows below in the next section.

The IRI component of the optical model had an effect on the PSD curve and the boundary between the silt and clay size classes. Samples with a relatively large silt content were selected to determine an adequate value for the IRI that would enhance the boundary between silt and clay. Although IRI values around 0.1 are commonly used, it was noted that a value of 0.001 for the IRI made it easier to distinguish between silt and clay (Fig. 7) and improved model prediction. Values greater than 0.01 had little effect on the optical model output (data not shown). Parameters for the optical model using the Mie theory were set at 1.42 and 0.001 for the RRI and IRI, respectively.

Relationship to the Sieve-Pipette Method

Particle size distribution data obtained with the light diffraction method was compared with sieve-pipette measurements of the same soil samples. A paired-sample *t* test was used to determine

separately if there was statistical difference between sand, silt, and clay contents measured by the two methods. Several combinations of RRI and IRI were used for the optical model and tested against the sieve-pipette results. With the RRI and IRI values at 1.42 and 0.001, respectively, there were no significant differences between the two methods for sand ($P = 0.084$), silt ($P = 0.743$), and clay ($P = 0.052$) at the 95% level.

Relationship between the light diffraction and sieve-pipette methods varied for the different soil fractions (Fig. 8). Correlation between the sieve-pipette and light diffraction measurements for sand was acceptable ($R^2 = 0.88$). However, for the silt and clay fractions agreement between methods was lower, with R^2 values of 0.80 and 0.69, respectively (Fig. 8). This was in accordance with the findings of Konert and Vandenberghe (1997). Furthermore, the slope of the lines were close to one for sand and silt (1.028 and 1.067, respectively), whereas for clay, it was 0.7703 (Fig. 8).

Algebraic differences between the two methods were varied (Table 4). Overall differences ranged between 0% and 19.6%, with

typically all three fractions varying for a specific soil. Mean differences for sand, silt, and clay were -3.9, 0.8, and 3.1, respectively. In general, the light diffraction method as presented here tended to underestimate the sand fraction.

The constructed soil samples created from the fractionated soil served as a check for the sieve-pipette technique, as well as the laser-light diffraction. As expected, there was excellent agreement between the sieve-pipette method and the artificial soil samples (Table 2). Agreement with the light diffraction procedure was acceptable, with S.E. between the three measurements ranging from 0.2% to 9.6%. Although the laser-light scattering technique for PSD determination does not have perfect agreement with the sieve-pipette, it provides a means of rapidly obtaining soil PSD data.

CONCLUSIONS

The procedure described in this paper for analysis of soil PSD with a light diffraction method was robust and reproducible. Soil sample analysis was achieved in a fraction of the time when compared with the traditional sieve-pipette method. Operator error and differences seem to be minimal with the proposed procedure. Sonication alone seemed to work as well as chemical dispersion. In addition, modifications to the RRI and IRI of the optical model improved results and comparison with the sieve-pipette method. Discrepancies between the light diffraction and the sieve-pipette method are a concern, but with the advent of new instrumentation and data analysis software, these differences could be reduced further.

The light diffraction method can be of value when used to make relative comparisons of a large number of soil samples of similar origin, within the same soil profile, or across a given landscape, such as for detailed soil mapping and spatial variability work. If a large number of samples from the same landscape need to be analyzed, it may be feasible to develop a soil specific calibration. In this situation, a subset of samples could be analyzed by the sieve-pipette method and used to calibrate the light diffraction method. This process would speed the total analysis time and allow for intensive soil sampling in the field. The laser-light diffraction technique does not have a perfect agreement with the sieve-pipette method, but it is reproducible and provides data rapidly. This type of measurement can be of value, but for an exact

and accepted determination of soil PSD, the sieve-pipette method is still recommended.

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REFERENCES

- Allen, T. 1981. Particle Size Measurement. 3rd ed. Chapman and Hall, New York, NY.
- Agrawal, Y. C., I. N. McCave, and J. B. Bailey. 1991. Laser diffraction size analysis. In: Principles, Methods, and Application of Particle Size Analysis. J. P. M. Syvitski (ed.). Cambridge University Press, New York, NY, pp. 119-128.
- Arriaga, F. J., B. Lowery, S. Park, and D. W. Mays. 2000. Determining particle size using a laser light diffraction technique. In: Agron. Abst., Soil Sci. Soc. Am., Madison, WI, p. 213.
- Beuselinck, L., G. Govers, J. Poesen, G. Degraer, and L. Froyen. 1998. Grain-size analysis by laser diffractometry: comparison with the sieve-pipette method. *Catena* 32:193-208.
- Buurman, P., Th. Pape, and C. C. Muggler. 1997. Laser grain-size determination in soil genetic studies: 1. Practical problems. *Soil Sci.* 162:211-218.
- de Boer, G. B. J., C. De Weerd, D. Thoennes, and H. W. J. Goossens. 1987. Laser diffraction spectrometry: Fraunhofer diffraction versus Mie scattering. *Particle Characterisation* 4:14-19.
- Eshel, G., G. J. Levy, U. Mingelgrin, and M. J. Singer. 2004. Critical evaluation of the use of laser diffraction for particle-size distribution analysis. *Soil Sci. Soc. Am. J.* 68:736-743.
- Gee, G. W., and D. Or. 2002. Particle-size analysis. In: Methods of soil analysis, Part 4. Physical Methods. J. Dane and G. C. Topp (eds.). Soil Sci. Soc. Am. Madison, WI, pp. 255-294.
- Hurlbut C. S. 1971. Dana's Manual of Mineralogy. 18th ed. John Wiley and Sons Inc., New York, NY.
- Indorante, S. J., L. R. Follmer, R. D. Hammer, and P. G. Koenig. 1990. Particle-size analysis by a modified pipette procedure. *Soil Sci. Soc. Am. J.* 54:560-563.
- Konert, M., and J. Vandenberghe. 1997. Comparison of laser grain size analysis with pipette and sieve analysis: a solution for the underestimation of the clay fraction. *Sedimentology* 44:523-535.
- Loizeau, J. L., D. Arbouille, S. Santiago, and J. P. Vernet. 1994. Evaluation of a wide range laser diffraction grain size analyser for use with sediments. *Sedimentology* 41:353-361.
- Matthews, M. D. 1991. The effect of grain shape and density on size measurement. In: Principles,

- Methods, and Application of Particle Size Analysis. J. P. M. Syvitski (ed.). Cambridge University Press, New York, NY, pp. 22–33.
- Muggler, C. C., Th. Pape, and P. Buuman. 1997. Laser grain-size determination in soil genetic studies: 2. Clay content, clay formation, and aggregation in some Brazilian Oxisols. *Soil Sci.* 162: 219–228.
- Starr, G. C., P. Barak, B. Lowery, and M. Avila-Segura. 2000. Soil particle concentrations and size analysis using a dielectric method. *Soil Sci. Soc. Am. J.* 64:858–866.
- Syvitski, J. P. M., K. W. G. LeBlanc, and K. W. Asprey. 1991. Interlaboratory, interinstrument calibration experiment. *In: Principles, Methods, and Application of Particle Size Analysis.* J. P. M. Syvitski (ed.). Cambridge University Press, New York, NY, pp. 174–193.
- Vitton, S. J., and L. Y. Sadler. 1997. Particle-size analysis of soils using laser light scattering and X-ray absorption technology. *Geotech. Test. J.* 20:63–73.
- Wilding, L. P., N. E. Smeck, and L. R. Drees. 1977. Silica in soils: quartz, cristobalite, tridymite, and opal. *In: Minerals in Soil Environments.* J. B. Dixon and S. B. Weed (eds.). Soil Sci. Soc. Am., Madison, WI, pp. 471–552.
- Williams, J. 1968. Problems of ambiguity involved with the utilization of Mie theory in particle size determination. Technical Report 49, reference 69–19, Chesapeake Bay Institute, The John Hopkins University, Baltimore, MD.
- Xu, R. 2002. *Particle Characterization: Light Scattering Methods.* Kluwer Academic Publishers, New York, NY.
- Zar, J. H. 1984. *Biostatistical Analysis.* 2nd edition, Prentice Hall, Englewood Cliffs, NJ.
- Zobeck, T. M. 2004. Rapid soil particle size analyses using laser diffraction. *Applied Eng. Agric.* 20:633–639.

DETERMINATION OF POROSITY AND PORE CONNECTIVITY IN FELDSPARS FROM SOILS OF GRANITE AND SAPROLITE

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Pores in feldspars, the most common silicates, are of great interest for the estimation of internal weathering reactions. Here, we compare three different methods to characterize the porosity of feldspars from four soils of granite and saprolite developed on granites in Germany (Black Forest, Harz mountains). Besides the classical mercury intrusion porosimetry (MIP), we used a technique based on Wood's metal intrusion. The liquid metal was pressed at 100 to 150 °C with approximately 500 bar argon pressure into the pores, and the distribution of the solidified metal in feldspar grains was analyzed using electron microscopy. In addition, the temperature-controlled water release from water-presaturated feldspars was analyzed using Karl-Fischer titration (KFT).

Volume fractions of connective pores determined by the three methods were qualitatively in good agreement, considering that single grains are analyzed using Wood's metal intrusion and KFT but larger batches of feldspar were analyzed using MIP. With MIP, relatively high porosities between 7.8 and 22.3 vol.% were determined in feldspars, much larger than usually found in granite rocks. These high values were confirmed by KFT analyses. Back-scattered electron images of feldspars impregnated with Wood's metal demonstrated that, at the initial stage of alteration, pores propagated from cracks and outer surfaces into the interior of feldspars. Highly altered feldspar species show a patchy distribution of pores, and pores are preferentially formed in the Na-rich phase of alkali feldspars. Enhanced topographical images indicated that some of the connective small pores were not intruded by liquid Wood's metal at the applied pressure of 500 bar, probably because of the filling of pores with secondary minerals and the ink-bottle effect. The ink-bottle effect also leads to an overestimation of small pores by MIP. As a consequence, the specific surface area calculated from MIP data is significantly lower than the value determined by nitrogen adsorption. The pore size distribution obtained by MIP was found to be unimodal with maxima in pore radii between 80 and 390 nm for most of the samples (an exception was the St. Blasien Cv-horizon with two broad maxima at 20 and 540 nm). Organic carbon of up to 2.2 g kg⁻¹ was found in all studied feldspars. This implies that pores within feldspars are, or at least have been, accessible for dissolved organic matter. The volume fraction of pores that are disconnected to the surface (inclusions) was estimated to be 1.2 to 2.7 vol.% based on the water release of above 700 °C, as analyzed using KFT. Secondary minerals, which play an important role in weathering reactions by sealing pores and locally buffering chemical reactions, were identified by X-ray diffraction, thermal analysis, and infrared spectroscopy. Kaolinite, gibbsite, and sericite were found with an overall portion of up to 4.6 vol.% but were strongly varying in abundance. (*Soil Science* 2006;171:675–694)

Key words: Porous feldspars, pore connectivity, Wood's metal, image analysis, secondary minerals, Karl-Fischer titration.