Received: 24 October 2023 | Accepted: 24 June 2024

DOI: 10.1002/saj2.20738

Soil Science Society of America Journal

ORIGINAL ARTICLE

Fundamental Soil Science

Establishing a standard protocol for soil texture analysis using the laser diffraction technique

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Assigned to Associate Editor Jingyi Huang.

Abstract

Optical methods including laser diffraction have been increasingly used to measure soil texture and particle size distribution. However, they have not been adopted yet as a routine methodology mainly due to the difficulties in comparing their results to more commonly used techniques (i.e., sedimentation methods). Many attempts exist in the literature to find an agreement between methodologies with relative success. In this work, we aim to improve the agreement between methodologies by adjusting parameters of the laser diffraction analysis, including sample treatment (chemical dispersion, carbonate removal, and sand separation), mode of sample addition (subsampling vs. transmittance matching), and analysis parameters (time of sonication and refractive index). Soil texture class determined by laser diffraction agreed with the sieve–hydrometer method in 78% of the runs when the following parameters were used: (1) Refractive index of 1.44 - 0.100i, (2) 180 s of sonication, (3) sand sieving prior to analysis, and (4) sample dispersion by shaking the sample for 1 h with 5% sodium hexametaphosphate. We observed that adding the entire sample to the analyzer (1 g of soil in 100 mL of dispersant) while keeping the appropriate levels of transmittance through dilution (transmittance matching) is a better way of sample addition in comparison to subsampling, especially for coarser soil samples. This work proposes a standard operation procedure that may broaden the adoption of laser diffraction analysis as a routine soil texture methodology.

1 | INTRODUCTION

Routine analyses of soil texture and particle size distribution are typically conducted using sedimentation methods (Gee & Or, 2002). More recently, optical methods such as laser diffraction (LD) gained popularity due to the easiness of measurements. The adoption of a new methodology requires establishing a relationship with current and commonly used methods to evaluate performance and reliability. The literature provides many quantitative relationships between LD and sedimentation methods with mixed outcomes (Arriaga et al., 2006; Coates & Hulse, 1985; Di Stefano et al., 2010; Eshel et al., 2004; Faé et al., 2019), and a common methodology for using LD for soil texture is yet to be adopted. In this work, we modify parameters of the LD analysis and compare it to sedimentation methods in terms of soil texture classification. We also test reported mathematical relationships between the hydrometer and

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Abbreviations: LD, laser diffraction; Na-HMP, sodium hexametaphosphate; NAPT, North American Proficiency Testing; OM, organic matter; RI, refractive indices.

LD to evaluate their performance across different datasets. We then propose a standard operating procedure for LD analysis to allow direct comparison and interpretation of soil texture.

Laser light scattering from diffraction measurement, that is, LD, is based on the principle that the angle of light diffraction increases with decreasing particle size (Dane & Topp, 2002). The intensity of the diffracted light is measured as a function of the angle of diffraction, and is used to infer the particle size based on Mie's theory, a relationship that transforms the particle–light interaction patterns into a volumetric diameter distribution (Dane & Topp, 2002; Pachon et al., 2019). Fraunhofer diffraction is also a diffraction theory used in laser diffraction applications (Dane & Topp, 2002). Besides being easy to conduct, LD only requires a small amount of sample, and quickly provides a continuous and detailed particle size distribution curve. Furthermore, because LD provides a volume-based size distribution as opposed to a mass-based size distribution, the measurements are independent of soil particle density, a potential source of error in sedimentation-based techniques (Eshel et al., 2004). However, efficacy of the LD method is affected by its inherent difficulty to characterize irregularly shaped particles (Eshel et al., 2004). Similarly to the traditional sedimentation methods, LD assumes spherical particles, but it measures the nominal light scatter diameter as opposed to the Stoke's diameter (Faé et al., 2019). These are different particle measurements, which complicates the direct comparison between methodologies. A critical step to compare LD and sedimentation methods is identifying equivalent clay and silt threshold diameters.

The volume-based versus mass-based distributions and the differences in the measured diameter are two reasons for the difficulty in comparing these methods and why there are many attempts in the literature to create a functional relationship between methodologies. Despite the inherent differences between methods and the difficulty comparing and creating quantitative relationships between them, linking LD results to traditionally used methods is necessary to increase reliance on the new technology and to certify replicability of results. The attempts to improve the agreement between methodologies do not preclude the need to build a database mapping soil properties to LD-derived particle size distributions for a stand-alone method advancement. To accomplish this, a common methodology must be adopted for LD analysis to facilitate comparison and interpretation of results.

Many studies have reported discrepancies between LD and sedimentation methods. Eshel et al. (2004) found that the relationship between LD and the sieve–pipette method depended on the particle size class, with greater agreement for the sand fraction and an underestimation of the clay portion. The authors state that no consistent relationship between sedimentation methods and laser diffraction methods can be formulated, mainly because of variations in soil properties, mineralogy and morphology, and how these affect each method. Clay underestimation by LD is a consistent observation across studies (Di Stefano et al., 2010; Loizeau et al., 1994; Taubner et al., 2009), with varying detection efficiency depending on the amount of clay in the sample (Loizeau et al., 1994; Thomas et al., 2021). This is an inherent characteristic of the LD technique, because the platy shape of the clay particles is described by a cross-sectional area larger than that of a sphere of equal volume (Taubner et al., 2009). Conventionally, the cutoff between clay and silt is 2 μm and between silt and sand is 50 μm, but past studies have proposed a higher clay threshold (4 to 8 μm) for LD analysis to correct for the non-sphericity of the particles (Konert & Vandenberghe, 1997; Makó et al., 2017; Thomas et al., 2021). This adaptation was proven to produce comparable LD and sedimentation results, correcting the underestimation of platy particles with random orientations by the LD method. Svensson et al. (2022) observed that 36 out of 44 samples were assigned to the same texture class in both laser diffraction and sieve–pipette methods when the clay–silt cutoff was 3.905 μm, and silt–sand was 63 μm.

Core Ideas

- We propose a protocol for laser diffraction (LD) analysis of soil texture and particle size distribution.
- We tested parameters of LD analysis and compared the LD-defined soil texture classes to the hydrometer-defined classes.
- The best match was found using 1 h of shaking with 5% sodium hexametaphosphate, followed by sand separation and 180 s of sonication.
- The effect of carbonate removal was minor and did not impact soil texture classification.
- We fitted reported relationships between sedimentation and LD methods to our data and evaluate their efficacy.

Another adaptation suggested in past works refers to reducing the range of particle sizes by sieving out the sand prior to analysis (Faé et al., 2019; Miller & Schaetzl, 2012; Svensson et al., 2022). These studies suggest that reducing the particle size range of the sample could increase precision because the laser-generated particle size data for coarser samples show lower precision. Sample treatment has also been suggested as a strategy to improve the agreement between methodologies, for example, organic matter and carbonate removal (Konert & Vandenberghe, 1997), and sample dispersion through different methods (Faé et al., 2019; Miller & Schaetzl, 2012).

Despite the variations in the methodology and the inherent difficulties of comparison with sedimentation methods,

TABLE 1 Soil properties: median of clay, silt, and sand fractions, and in parenthesis, the median absolute deviation. The USDA textural class is shown.

	Hydrometer (%)						
Soil	**Clay**	**Silt**	**Sand**	**Textural class**	**pH**[a]	**Soil organic matter**[b] **(g kg^{-1})**	**Calcium carbonate (g kg^{-1})**
Bahem	21.7	62	16.3	Silt Loam	8.1	16	BDL[c]
Declo	13.3	45.4	41.2	Loam	8.1	18	74.3
Arbuckle	20.7 (3.25)	24 (3.5)	55.4 (3.28)	Sandy Clay Loam/ Sandy Loam	7.38	1.58	8.5 (2.8)
Bakey	19 (2.1)	19.3 (2.72)	59.5 (2.5)	Sandy Loam	6.1	3.9	5.2 (0.7)
Bauder	35 (3)	28.7 (2.87)	35.8 (3.83)	Clay Loam	7.6	2.7	54.6 (7.7)
Bearden	18.0 (1.9)	20.8 (2.0)	61.0 (1.7)	Sandy Loam	6.5	39 (1.6)	3.4 (2.6)
Belen	45 (4.38)	30.8 (3.45)	25.5 (4.1)	Clay	7.8	2.2	71.0 (5.6)
Cecil	18.1 (2.51)	16 (2)	64.7 (2.75)	Sandy Loam	5.5	3.8	2.5
Cora	49.5 (3.0)	41.4 (5.0)	9.0 (2.0)	Silty Clay	8.0	27.2 (2.0)	122.0 (8.4)
Deerfield	5.02 (1)	8.4 (1.65)	86 (1.8)	Sand/Loamy Sand	6.9	2.7	9.0 (1.8)
Felton	4.8 (0.6)	7.3 (1.5)	88 (2)	Sand	6.4	4.4	2.5 (0.4)
Fielding	23.2 (2.0)	40.0 (5.1)	38.0 (3.4)	Loam	8.1	31.2 (2.1)	46.0 (8.0)
Garrett	25 (1.8)	16 (1.6)	59 (2)	Sandy Clay Loam	7.8	1.7	44.0 (3.7)
Lutz	17.5 (3.2)	41.6 (3.6)	40.2 (5.2)	Loam	7.5	61.0 (2.1)	28.0 (1.9)
Maumee	7.5 (2.5)	8.9 (2.3)	84 (2.6)	Loamy Sand	7.6	3.2	17 (8.5)
Mendon	28.7 (3.2)	44 (3)	29.4 (4.2)	Clay Loam	7.2	5.1	13.0 (2.5)
Paso	52.5 (2.5)	30.3 (3.8)	16 (3.9)	Clay	7.4	4.4	172.0 (29.0)
Watermelon	9 (1.38)	14 (2.4)	76 (1.8)	Loamy Sand/Sandy Loam	7.0	1	4.8 (0.5)

[a] 1:1 soil to water ratio.

[b] Soil organic matter measured via loss on ignition.

[c] Below detection limit.

many studies report a significant linear correlation between the particle size fractions measured by LD and sedimentation methods (Faé et al., 2019; Konert & Vandenberghe, 1997; Svensson et al., 2022). For instance, Svensson et al. (2022) reported a correlation coefficient between pipette and LD of 97.7% for clay, 98.3% for silt when optimized class thresholds were used in the LD analysis. Similarly, Faé et al. (2019) reported that a regression of LD and hydrometer data yielded R^2 values of 0.92, 0.92, and 0.99 for clay, silt, and sand, respectively. Eshel et al. (2013) argues that these relationships are restricted to the dataset used and cannot be generalized for other soils and applications. But the relationships reported in the literature demonstrate that sedimentation and LD methods are comparable, and standardizing the procedure for LD is critical for a wider adoption of LD for soil texture analysis.

The main purpose of this study was to (I) evaluate whether the agreement between LD and the sieve–hydrometer method can be improved by adjusting selected parameters of the LD analysis, (II) evaluate whether existing relationships between hydrometer and LD can be generalized to different soils and method variations, and (III) devise a standard operating procedure for LD analysis compatible with sedimentation methods to be adopted as routine soil texture analysis.

2 | MATERIALS AND METHODS

2.1 | Textural characterization of soils

Eighteen soils were analyzed in this study. Two of them, Bahem and Declo, represent major soil textural classes in agricultural areas of Southern Idaho, the other 16 are standard soils from the North American Proficiency Testing (NAPT) Program. The NAPT soils used in this study represent 16 different soil series and are described in Table 1. The soil texture shown in Table 1 was determined by the combined sieve–hydrometer method (Dane & Topp, 2002). The soil textural classification defined by the USDA was used throughout this paper. For three NAPT soils (Arbuckle, Deerfield, and Watermelon), the classification falls on the line between two textural classes, and both classes are then used to describe these soils. The locally collected soil samples were obtained at 0–300 mm of depth and were air-dried and sieved through a 2-mm sieve prior to analysis. The NAPT soil samples were air-dried and pulverized to pass a 2-mm sieve prior to analysis.

The soils were analyzed using the LD analyzer HORIBA Partica LA-960. The analyzer works with two light sources: (1) a red solid state 5 mW laser diode (650 nm), and (2) a blue solid state 3 mW LED (405 nm). The analyzer uses Mie

TABLE 2 Particle size classes cutoffs in HORIBA LA-960 particle size analyzer.

Class	Diameter (µm)	Class	Diameter (µm)
Fine pebble	≥4000	Coarse silt	31.25–62.5
Very pine pebble	2000–4000	Medium silt	15.63–31.25
Very coarse sand	1000–2000	Fine silt	7.8115.63
Coarse sand	500–1000	Very fine silt	3.91–7.81
Medium sand	250–500	Clay	1–3.91
Fine sand	125–250	Colloid	0.01–1
Very fine sand	62.5–125		

scattering as the measurement principle, which requires RI as an input parameter. This parameter is a complex number, the real part representing the velocity of light through the sample relative to the velocity of light in vacuum, and the imaginary term representing the transparency and absorptivity of the sample (Eshel et al., 2004). The measurement range is 10 nm to 5000 µm. For all measurements, transmittance was maintained within the manufacturer's suggested range of approximately 85% to 95%.

The clay–silt cutoff used is defined by the Udden–Wentworth scale (Wentworth, 1922), it is a higher cutoff than the one traditionally used in pipette and hydrometer analysis. This is intentional, aiming to account for the underestimation of clay in LD analysis (Di Stefano et al., 2010; Eshel et al., 2004; Loizeau et al., 1994; Taubner et al., 2009). This threshold is within the observed size of clay particles, as scanning electron microscopy (SEM) photographs show platy particles with diameters of up to 10 µm (Konert & Vandenberghe, 1997). The cutoffs used in this analysis are described in Table 2. For the total silt and sand fractions, the coarse, medium, and fine portions of each class were summed; the total clay fraction is the sum of clay and colloid.

We tested different parameters of the LD method, aiming to evaluate adjustments to the technique that would result in (1) a significant agreement with sedimentation methods and (2) a standard protocol for soil texture analysis. These parameters are: sample addition, RI, sonication time, and sample pre-analysis treatments (dispersion, sand separation, and carbonate removal). The levels of the parameters tested are described in Figure 1 and are further discussed below.

2.2 | Pre-analysis sample treatments

We tested different pre-analysis treatments by (1) allowing different interaction times with sodium hexametaphosphate (Na-HMP), (2) removing carbonates, and (3) sieving out sand particles.

Na-HMP treatment (p.1 in Figure 1) is the standard dispersion technique in traditional soil texture analysis (Dane & Topp, 2002). We tested three dispersion treatments: (1) 100 mL of 5% Na-HMP added to 1 g of soil and shaken overnight, (2) 100 mL of 5% Na-HMP added to 1 g of soil and shaken for 1 h, and (3) 2 mL of 5% Na-HMP added to the LD sample bath immediately before sample addition (1 g of soil in 100 mL of deionized [DI] water) as suggested by Thomas et al. (2021). In the latter case, the soil samples were mixed with 100 mL of DI water to maintain the same solid to solution ratio as the other treatments. For comparison, we also tested a paste, the mode of addition recommended by the International Organization of Standardization (ISO) for powders. The pastes were formed by adding the 5% Na-HMP solution to 1 g of soil until it reached a consistency typical of toothpaste (International Organization for Standardization, 2020).

For the carbonate removal treatment (p. 2 in Figure 1), we weighed 1 g of soil into a 50-mL centrifuge tube and added 10 mL of DI water and 1 mL of 1 M sodium acetate (NaOAc, adjusted to pH = 5). The mixture was centrifuged for 10 min at 1500 rpm and the clear supernatant was discarded. The soil was then washed twice by shaking with 10 mL of DI water, centrifuging for 10 min at 1500 rpm and discarding the supernatant (Dane & Topp, 2002). We opted for not conducting OM (organic matter) pre-treatment. Previous works evaluated hydrogen peroxide (H_2O_2) treatment for OM removal and observed no major effect on the LD-derived particle size distribution (Callesen et al., 2018; Di Stefano et al., 2010).

Sand separation was tested as a strategy to decrease occlusion and produce a narrower range of particle sizes, ensuring that smaller size fractions were more accurately represented. For removing the sand, after the Na-HMP treatment, the 1-g soil sample in dispersant or water was wet sieved through a No. 300 mesh sieve (50 µm). The wet sand was then oven dried at 60°C overnight and weighted for the calculation of the sand fraction. The remaining sample was analyzed in the LD analyzer. For samples that had sand removed, we used the following equations to recalculate the particle size fractions (Faé et al., 2019; Svensson et al., 2022):

$$f_{\mathrm{cl}} = \frac{(M_{\mathrm{t}} - M_{\mathrm{sa}}) \times f_{\mathrm{cl\text{-}LD}}}{M_t} \tag{1}$$

$$f_{\mathrm{si}} = \frac{(M_{\mathrm{t}} - M_{\mathrm{sa}}) \times f_{\mathrm{si\text{-}LD}}}{M_{\mathrm{t}}} \tag{2}$$

where M_{t} is the sample total mass, M_{sa} is the mass of sand (weight of oven-dry the sieved-out portion), f_{cl} and $f_{\mathrm{cl\text{-}LD}}$ are the recalculated and measured fractions of clay, and f_{si} and $f_{\mathrm{si\text{-}LD}}$, the recalculated and measured fractions of silt, respectively.

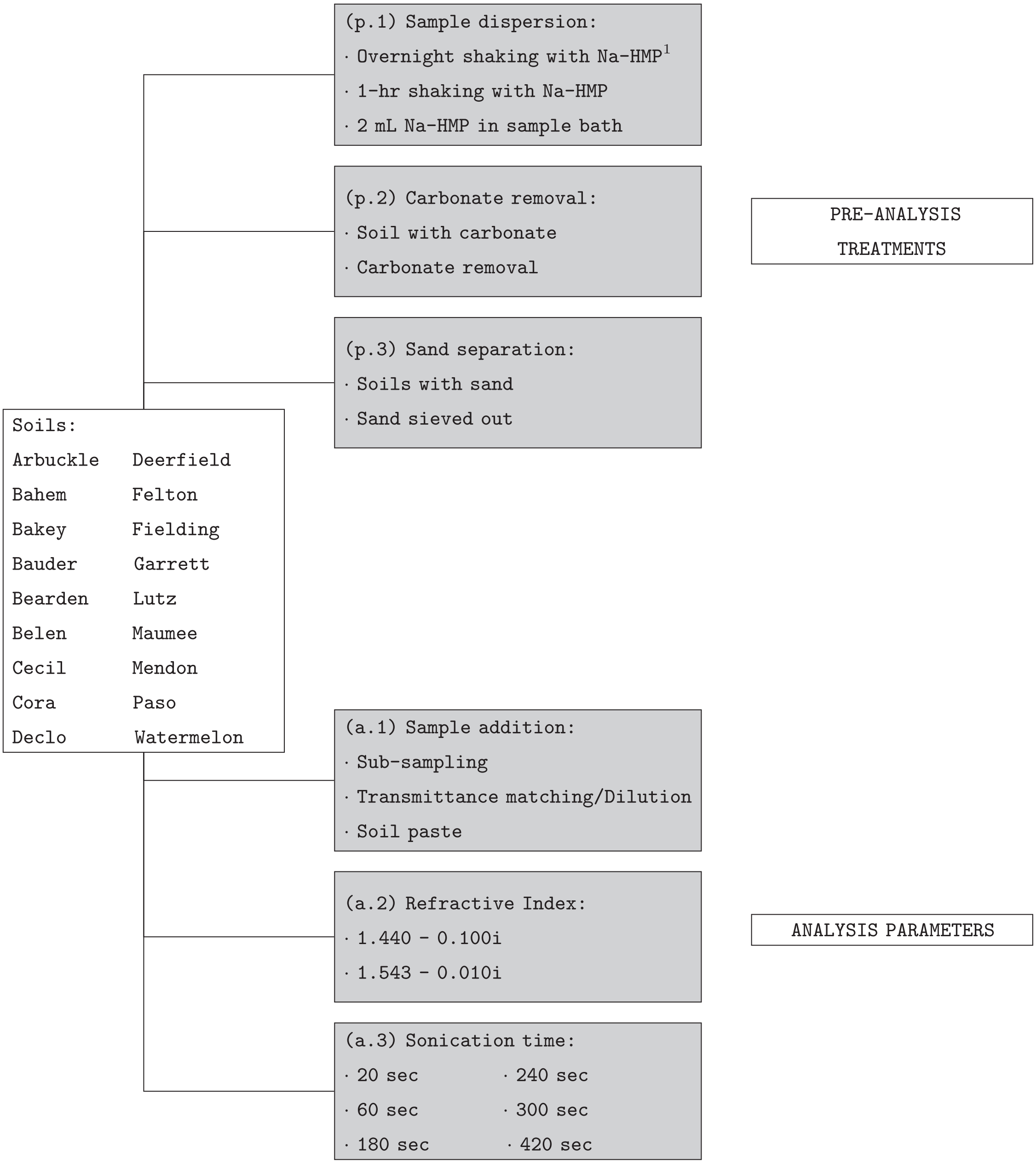


FIGURE 1 Evaluating the impact of pre-analysis (p) and analysis (a) parameters on the laser diffraction technique: different pre-analysis treatments (sieving of sand prior to analysis, sample dispersion, and carbonate removal) and analysis parameters (sample addition, refractive index, and sonication) were tested. Na-HMP, sodium hexametaphosphate.

2.3 | Analysis parameters

After pre-analysis treatments, samples were added to the analyzer by either (1) pipetting 1 to 2.5 mL aliquot directly into the bath containing 200 mL of reverse osmosis degassed water, (2) adding the entire sample (100 mL) to the bath (transmittance matching), or (3) adding the entire sample as paste (International Organization for Standardization, 2020). In the two latter cases, we used the analyzer function “auto-dilute” to bring the transmittance back to appropriate levels upon sample addition. This is not an exclusive function of this analyzer and the technique can be easily replicated by manually adding water to the sample bath for a similar dilution effect. The bath was constantly agitated at speed levels of 5–12 (2000 to approximately 4000 rpm), higher speeds were used in the dilution mode as more water was added to the system. The suspension circulated through the machine at speed 3 (maximum circulation speed is 15 at 10 L min^{-1}), as recommended by HORIBA for soil analysis. After each analysis, the bath was drained and the machine was rinsed and refilled with clean degassed reverse osmosis water.

The RIs selected reflect the index recommended by HORIBA (1.44 - 0.100i) as well as an index commonly reported in the literature (1.543 - 0.010i) (Faé et al., 2019; Svensson et al., 2022). The RI of 1.543 - 0.010i is generally accepted for soils because it is the reported RI for quartz, a common soil mineral.

For the sonication, we used an in-line ultrasonic probe (30 W, 20 kHz) and tested the sonication times: 20, 60, 180, 240, 300, and 420 s to examine whether longer sonication periods would affect particle disaggregation. The objective was to find the optimal time of sonication, which will disaggregate particles without fracturing the primary particles.

The analysis was divided into two steps: (1) establishing the laser diffraction protocol and (2) testing and validating the protocol. In the first set of experiments, we selected the optimal chemical dispersion treatment, carbonate and sand presence, mode of addition, RI, and sonication time to use in the subsequent analysis. The basis for our decision was to choose the levels of treatment that showed the lowest root mean square error (RMSE) when comparing the percentages of clay, silt, and sand determined by the hydrometer and laser diffraction methods. The Chi-square and R parameters were also used to validate the choice of the RI kernel and are shown in Equations (3) and (4).

$$\chi^2 = \sum \frac{1}{\sigma_i^2}[y_i - y(x_i)]^2 \tag{3}$$

$$R = \frac{1}{N}\sum_{i=i}^{N} \frac{1}{y_{(x_i)}}|y_i - y(x_i)| \tag{4}$$

where y_i is the measured scattered light and $y(x_i)$ is the calculated scattered light at each channel (i) of the detector. The standard deviation of the scattered light intensity is σ_i and N is the number of detectors. A lower Chi-square or R parameter indicates a better fit of the raw data to the reported particle size distribution.

The hydrometer and pipette results were obtained from the NAPT reports. For the local soils, the hydrometer soil texture was previously determined (Dari et al., 2019). Average sand, silt, and clay fractions were calculated from the LD analyses conducted with the optimal levels of the parameters tested. We then attributed the LD-defined USDA textural class and compared to the sieve–hydrometer classes.

2.4 | Using reported relationships between sedimentation and LD methods

Many dataset-specific regression equations exist in the literature to compare sedimentation and LD methods. Using two of these quantitative relationships, we evaluated whether a dataset-specific equation could be applied to other datasets to predict hydrometer values based on LD data. The objective was to examine whether a universal set of equations would work in any LD particle size classification to predict their correspondent hydrometer-derived class.

We used the equations developed by Faé et al. (2019) and Di Stefano et al. (2010) to predict hydrometer clay, silt, and sand fractions based on the LD-derived classes. Di Stefano et al. (2010) proposed sets of equations to refer LD measurement to sieve–hydrometer results according to homogeneous zones of the USDA triangle in terms of sand content (i.e., different sets of equations for soils with sand content greater than 50%, quasi equal to 50%, and less than 50%). Faé et al. (2019) developed regression equations comparing their LD data and the reported hydrometer results for 54 standard soil samples. We used the proposed equations on our LD-derived data and evaluated whether a common model can be used to relate the LD and sieve–hydrometer methods for different datasets.

3 | RESULTS AND DISCUSSION

We report the results in terms of fractions of clay, silt, and sand. Although the LD analysis provides more detailed information for characterizing particle sizes (e.g., particle size distribution, or the 10th, 50th, or 90th percentile of the particles diameters), reporting textural classes allows for direct comparison with the results obtained from the sieve–hydrometer method. Colloids (particles $\leq$1 μm) were identified in a few samples in the LD analysis, in values ranging from 0% to 9%. In those cases, the colloid percentage was added to the clay fraction.

Across all soils and analyses with different parameters (488 datapoints), LD-assigned textural class was the same as the hydrometer method in 55% of the cases. Using only the analyses with the optimal parameters (68 datapoints), 78% of the samples were correctly assigned to their hydrometer-defined classes. Out of the 18 soils tested, LD assigned the hydrometer-defined class to 14 (78%) soils. In comparison, the hydrometer-defined classes for NAPT soils agree with their pipette-defined classes in 81% of the cases. This means that even when comparing conventional and broadly used soil texture methods, a variation in the soil texture obtained is expected. The variation observed between the LD and the hydrometer is similar to the variation between the pipette and the hydrometer for the tested soils. The four soils that were misclassified in all replicates of the LD analysis were Declo, Garrett, Maumee, and Mendon. Figure 2 shows how the underestimation of clay is a significant factor in the misclassification of these soils.

Below, we detail the effects of the tested parameters on the efficacy of the method and the steps to determine the optimal LD methodology.

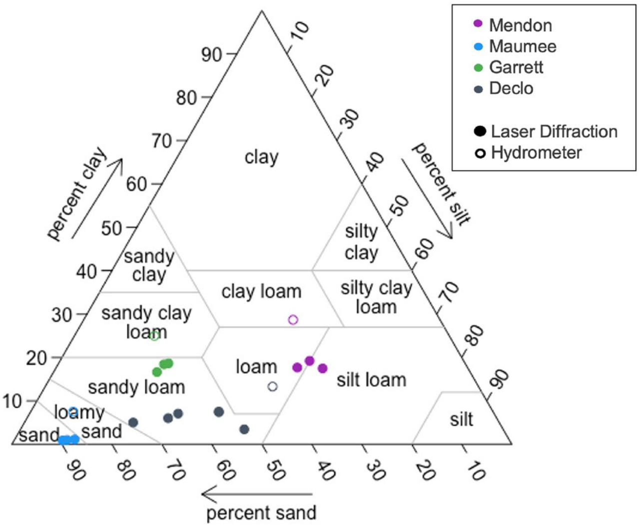


FIGURE 2 Soil texture triangle showing the soils for which the laser diffraction (LD) textural classification differed from the hydrometer classes in all replicates. Same color open and filled circles show the hydrometer and LD classes, respectively, for each of the soils.

TABLE 3 Root mean square error across all chemical dispersion treatments. The results indicate the difference (%) between the laser diffraction-derived classes compared to the values measured by the sieve–hydrometer method. The sodium hexametaphosphate in bath treatment was tested only on samples with sand.

Pre-analysis treatment: Chemical dispersion			
	In bath	1-h prior	Overnight
Bahem			
Clay	9.40	7.91	12.20
Silt	20.56	15.22	12.00
Sand	15.20	12.35	11.80
Declo			
Clay	7.22	6.94	6.46
Silt	24.92	16.71	16.74
Sand	18.57	15.16	16.86

3.1 | Establishing the LD protocol

3.1.1 | Pre-analysis treatment: Chemical dispersion

The 1-h shaking with Na-HMP yielded similar results compared to the overnight Na-HMP treatment, more commonly used in conventional soil texture analysis (Table 3). By using both chemical and physical dispersion techniques, the time of interaction with the chemical dispersant can be decreased to 1 h without loss of disaggregation efficacy. The shortest time of interaction between Na-HMP and soil sample was tested in the in-bath treatment. Adding Na-HMP directly to the bath results in an insufficient Na-HMP concentration in solution that summed to the short time of interaction between soil and Na-HMP results in an ineffective dispersion treatment. Figure 3 shows the silt fractions of the tested soils, in which the inefficiency of the in-bath treatment was most evident.

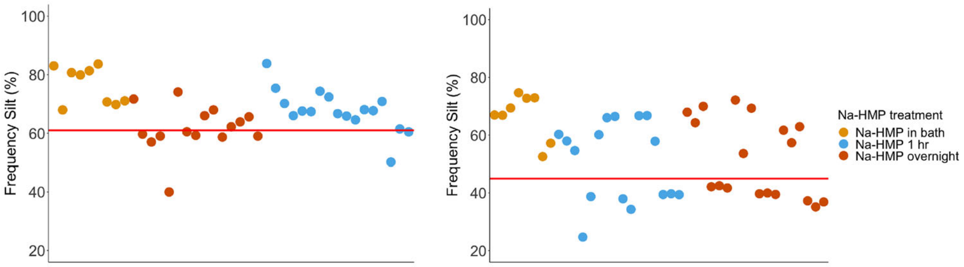
Additional tests with NAPT soils confirmed that the 1-h dispersion yielded similar results to the overnight dispersion. Both treatments show low RMSE depending on the texture fraction and soil (Table 4). This is further evidence that the 1-h shaking can be used for LD soil texture analysis without loss of efficacy of the dispersion treatment.

Because we used both chemical and physical dispersion, we also consider their interactive effect. Some authors discourage the use of chemical and physical dispersion methods in the same soil sample, arguing that their combination can result in flocculation (Bieganowski et al., 2018). We argue that the chemical dispersion can be aided by applying the optimal time and power of sonication. For instance, we saw indications that the overnight chemical dispersion was sufficient to break aggregates, as additional time of sonication did not show a significant effect on the frequency distribution (Figure 4a,b). In these figures, there is no evident effect of the sonication time, contrary to Figure 4c,d that show clear differences of sonication time when using 1-h Na-HMP treatment. For clay and silt, the combination of 1-h Na-HMP and 180 s of sonication is similar to the overnight treatment with 60 s of sonication (Figure 5). Based on all evaluated parameters, we consider that the use of 180 s of sonication combined with 1 h of shaking with Na-HMP prior to the analysis is preferable. The separate effects of sonication time are further described below.

3.1.2 | Pre-analysis treatment: Carbonate removal

Overall, the effect of carbonate removal was minor and did not impact soil texture classification, as seen in Figure 6. The tested soils had contrasting calcium carbonate content to demonstrate the effect of the treatment on soils with high calcium carbonate levels ($\geq$50 g kg^{-1}) and low calcium carbonate levels. In both cases, no significant effect of carbonate removal on soil texture was observed.

Aggregation in soils from semi-arid regions, such as Southern Idaho, is commonly influenced by their high carbonate contents (Virto et al., 2011). However, authors generally do not remove cementing or flocculating agents for soil texture analysis, and dissolution of carbonates before LD analysis is rarely used (Bieganowski et al., 2018). The literature shows contrasting results when carbonate pre-treatment is employed, and the differences seem to be driven by soil type and the proportion of carbonates in the samples. For instance, Virto et al. (2011) tested decarbonation treatments prior to LD analysis and reported that the aggregating influence of OM and carbonates differs among soils and tillage management and



(a) Bahem (b) Declo

FIGURE 3 Effect of chemical dispersion on the silt fractions of two tested soils, (a) Bahem and (b) Declo. The sodium hexametaphosphate in bath treatment was tested only on samples with sand. The red line indicates the hydrometer-defined silt fractions.

TABLE 4 Comparison between chemical dispersion treatments: 1-h versus overnight shaking with sodium hexametaphosphate. The results indicate the difference (%) between the laser diffraction-derived classes compared to the values measured by the sieve–hydrometer method. The hydrometer-defined texture is shown in parenthesis.

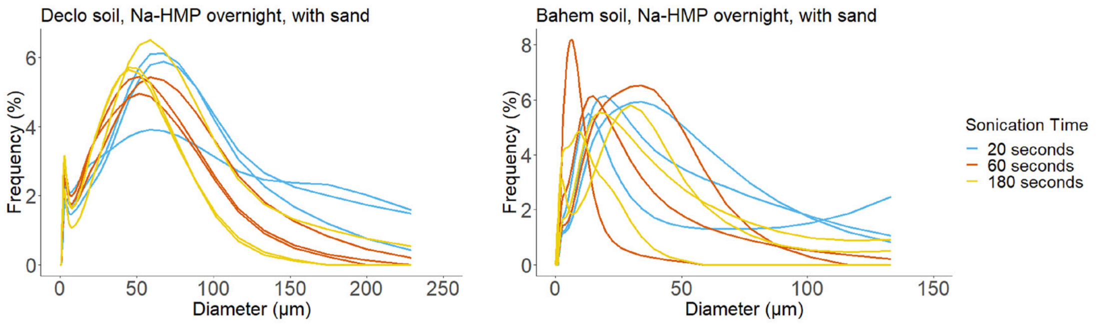
Pre-analysis treatment: Chemical dispersion		
	1-h prior	**Overnight**
Bauder (Clay Loam)		
Clay	10.99	9.50
Silt	8.02	7.44
Sand	3.66	2.71
Garrett (Sandy Clay)		
Clay	8.52	6.76
Silt	6.35	6.57
Sand	3.49	0.29
Paso (Clay)		
Clay	3.89	10.75
Silt	1.98	2.42
Sand	4.95	7.73
Watermelon (Loamy Sand/Sand Loam)		
Clay	3.39	3.22
Silt	1.90	0.74
Sand	4.13	4.92

depends on their relative proportion. The authors observed aggregating influence of carbonates on the grain-size distribution of two soils, and consider that the exchangeable calcium levels and carbonate mineralogy affect how significant that influence is. However, it is not clear whether the observed changes on grain-size distribution led to soil texture reclassification. Fisher et al. (2017) observed a minor effect of decarbonation on soils with low levels of carbonates. A minor effect of carbonate removal on particle size distribution was also observed in other works, even in samples with strong dominance of secondary carbonates (Lucke & Schmidt, 2015; Schulte et al., 2016). The results from this work are further evidence that carbonate removal do not appear to influence soil textural characterization even on soils with a higher carbonate content.

3.1.3 | Pre-analysis treatment: Sand separation

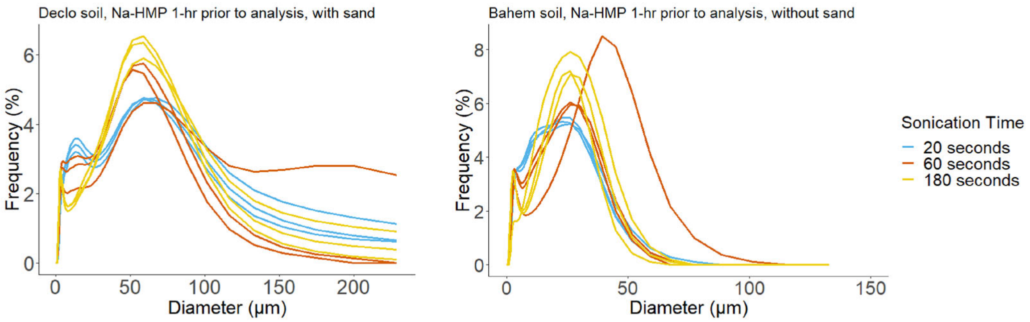
In conventional soil texture analysis, sand sieving is the first stage of the particle size characterization, after which the pipette method is conducted for determining the remaining fractions. In hydrometer analyses, sand is also determined by sieving (Gee & Or, 2002). We tested a similar sand separation approach in the LD analysis to examine whether the separation was still necessary when using the new technology. This approach has been tested before in LD analysis. Faé et al. (2019) noted that the sieving of the sand fraction is a key factor for obtaining precise results, because it limits particle size range, resulting in stable soil dispersed suspensions. By sieving the samples prior to laser detection, the hypothesis is that one can obtain a homogeneous aliquot and avoid obscuration of smaller particles from detection (Taubner et al., 2009). Here, we compare the results of the samples that were analyzed with and without sand.

The first observed effect of sand removal was the lower variability of the textural classes when compared to the samples analyzed with sand. Figure 7 shows the narrower boxplots (i.e., less scattered data) for all fractions when samples are run without sand. The sand fraction was the most affected by sand removal. The mean sand fraction across all Bahem samples without sand was 15.05% versus 8.74% for Bahem samples with sand. The effect of this difference is seen on the texture classification: 77% of the Bahem samples analyzed with sand were correctly assigned to the texture class silt loam, while 100% of the samples were assigned to the corrected class across all "no sand" samples. For the Declo soil, the sand removal resulted in significant differences across all particle size fractions. The texture of all Declo samples analyzed with sand was incorrectly identified as silt loam. When sand is removed, 56% of the samples were



(a) Declo soil

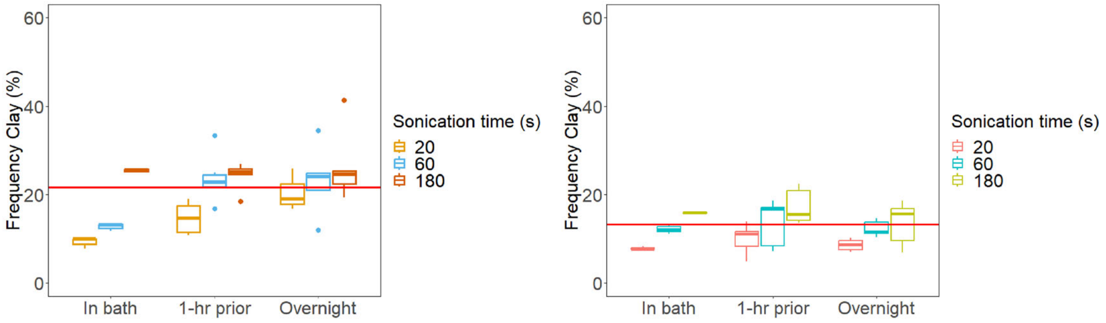
(b) Bahem Soil



(c) Declo soil

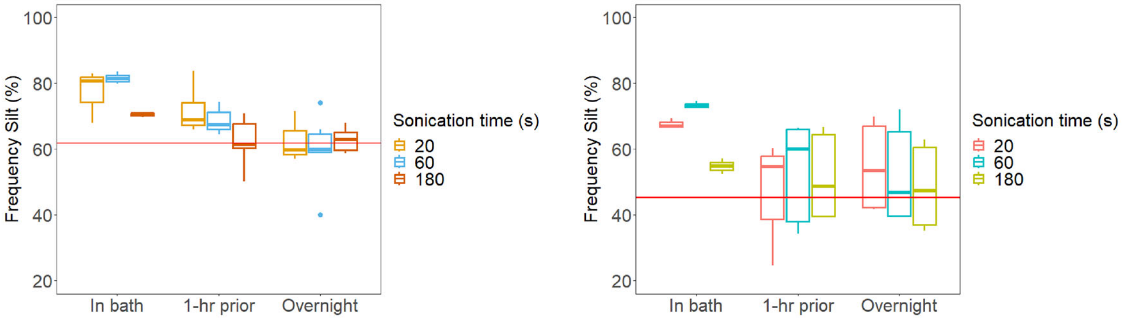
(d) Bahem Soil

FIGURE 4 Effect of time of sonication on the frequency distribution of the Declo soil (a and c) and Bahem soil (b and d). The analysis was conducted with RI = 1.44 - 0.100i, with the soil samples shaken overnight with sodium hexametaphosphate. Lines of the same color indicate procedure replicates.



(a) Clay - Bahem soil

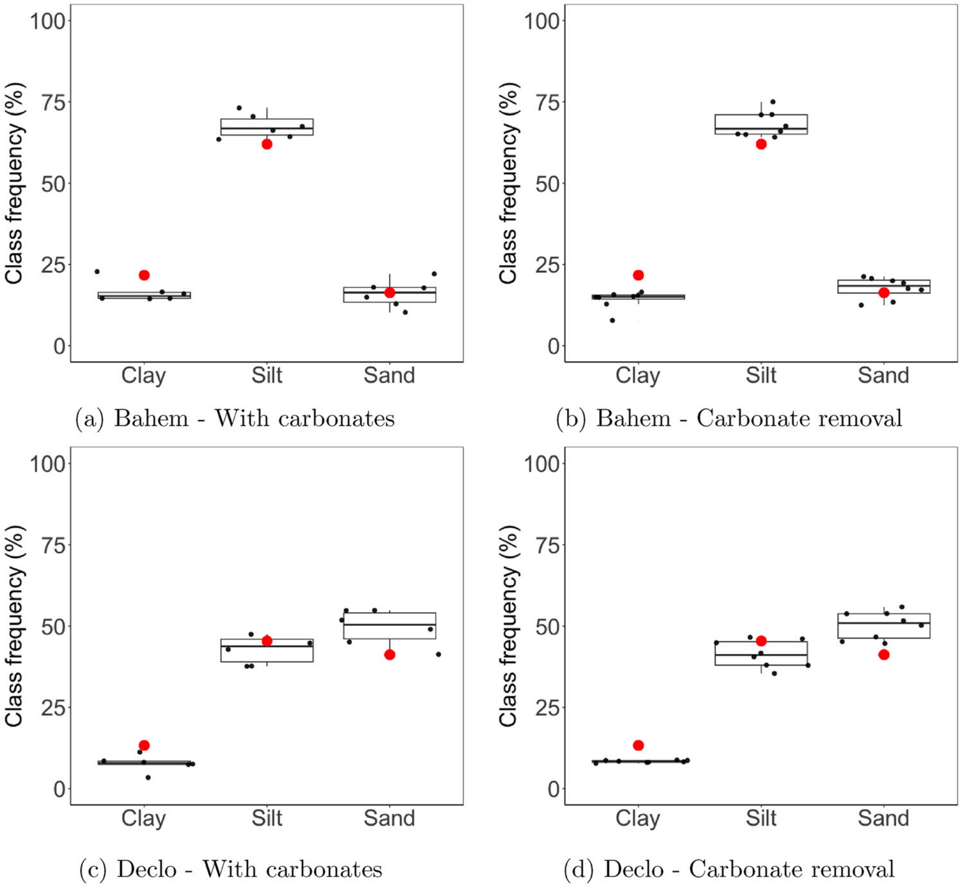
(b) Clay - Declo Soil



(c) Silt - Bahem soil

(d) Silt - Declo Soil

FIGURE 5 Combined effect of physical and chemical dispersion on the clay (a and b) and silt (c and d) fractions of tested soils. The red line indicates the hydrometer-measured clay or silt fractions. The boxplot displays the median and the first and third quartiles (lower and upper hinges). The whiskers extend from the hinge to |1.5 × interquartile range|.



(a) Bahem - With carbonates (b) Bahem - Carbonate removal

(c) Declo - With carbonates (d) Declo - Carbonate removal

FIGURE 6 Effect of carbonate removal on the clay, silt, and sand fractions of the Bahem (a and b) and Declo (c and d) soils ($n = 6$). The boxplot displays the median and the first and third quartiles (lower and upper hinges). The whiskers extend from the hinge to |1.5 × interquartile range|. The red dots show the frequencies as measured by the sieve–hydrometer method.

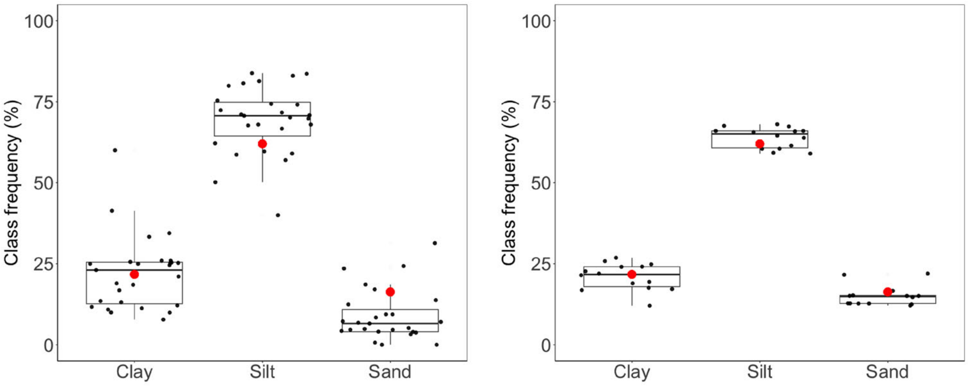
correctly classified as loam. These findings support sand removal before LD analysis to obtain more comparable results with traditional methods.

3.1.4 | Analysis parameters: Sample addition

Across all runs with pipette subsampling ($n = 237$), 44% of the samples were assigned to the same textural class as defined by the sieve–hydrometer method. In comparison, by adding the entire sample to the analyzer ($n = 223$), 71% of the samples were correctly classified. This result suggests that adding the entire soil sample and conducting a transmittance correction through dilution is a superior sample addition method when compared to pipetting, as subsampling can be a significant source of error. For instance, Miller and Schaetzl (2012) repeated the LD analysis for 1485 soil samples and reported that 11.5% changed textural class compared to the first analysis, most likely due to intra-subsample variability. The authors observed that the precision of laser-generated particle size data generally decreases for silty/loamy samples as particle size gets coarser. In this analysis, we observed that the difference between pipetting and adding the entire sample was less notable for coarser samples, such as Deerfield, Felton, Maumee, and Watermelon, soils with sand fraction greater than 75%. This could be due to the described loss of precision, and also because these samples had the sand fraction removed, so the pipette subsampling is more representative in the coarser samples (less soil to sample from) and therefore will produce more similar results compared to when the entire sample is added to the analyzer. The RMSE for the soils tested are shown in Table 5.

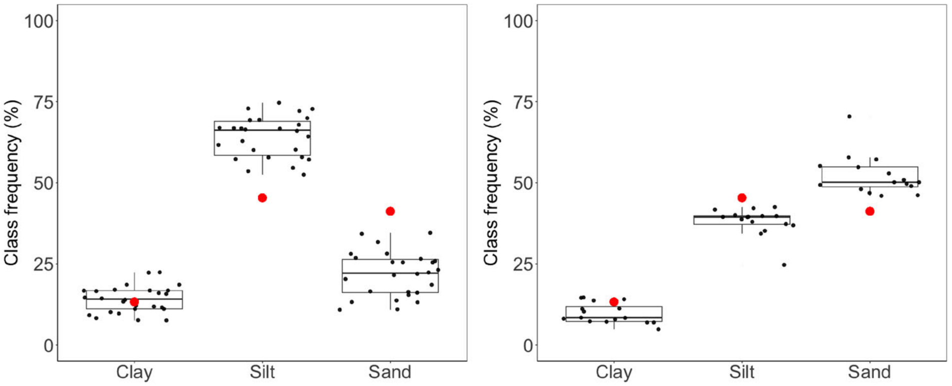
The addition of the entire sample has the potential to diminish sampling error and result in a more representative sample for all particle sizes. One limitation for adding the entire sample to the analyzer is the rapid decrease of the levels of transmittance below appropriate levels. To counteract, we used the “auto-dilution” function, that adds water to the bath until the transmittance levels are back to the recommended levels. In analyzers that do not have this function, the same could be accomplished by manually adding water to the bath and allowing the machine to drain the excess.

The soil paste did not produce satisfactory results and is not recommended as a mode of addition of soil samples to



(a) Bahem - With sand

(b) Bahem - Without sand



(c) Declo - With sand

(d) Declo - Without sand

FIGURE 7 Effect of sieving out the sand on the clay, silt, and sand fractions of the Bahem (a and b) and Declo (c and d) soils. The boxplot displays the median and the first and third quartiles (lower and upper hinges). The whiskers extend from the hinge to |1.5 × interquartile range|. The red dots show the frequencies as measured by the sieve–hydrometer method.

TABLE 5 Dilution (transmittance matching) versus subsampling (pipette): root mean square error (RMSE) comparing the hydrometer and the laser diffraction-defined fractions.

	Pipette			**Dilution (transmittance matching)**		
Soil	**Clay**	**Silt**	**Sand**	**Clay**	**Silt**	**Sand**
Arbuckle	8.62	13.98	4.94	5.89	3.72	2.20
Bahem	11.85	15.57	8.90	7.26	6.83	3.64
Bakey	11.01	8.75	6.74	14.38	5.87	10.74
Bauder	12.60	9.44	3.69	9.10	6.29	3.63
Belen	11.68	8.85	3.64	4.63	7.09	3.91
Cecil	6.80	0.97	7.33	6.39	1.44	8.20
Declo	7.02	18.32	16.59	5.47	5.22	9.86
Deerfield	3.72	1.21	3.59	3.84	2.07	4.13
Felton	2.97	1.05	3.88	2.51	3.07	3.54
Garrett	9.69	7.22	4.38	7.15	5.33	2.27
Maumee	6.37	1.02	6.70	6.50	2.07	4.39
Mendon	9.46	5.54	4.00	10.60	6.89	2.82
Paso	4.44	2.56	6.41	3.26	1.14	2.81
Watermelon	3.62	0.93	4.78	3.14	2.52	3.35

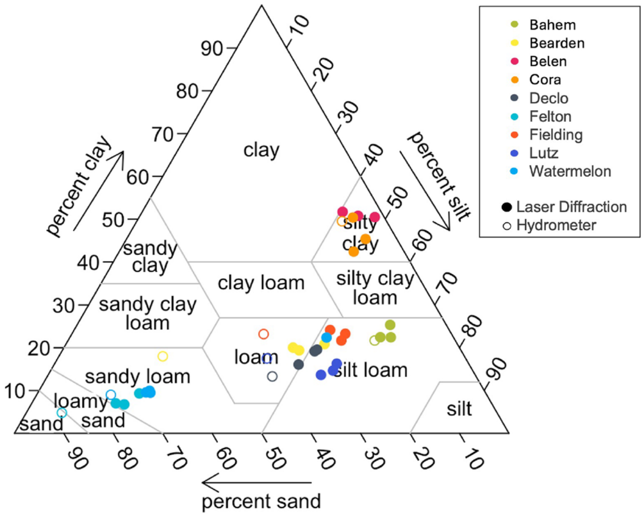


FIGURE 8 Soil texture triangle showing the results of the soil paste addition treatment.

LD analyzers. Although it is a methodology suggested by the ISO for LD analysis of powders (there is no mention of soils in the standard), the addition of the soil sample as paste failed to reproduce the hydrometer-defined classes (Figure 8), as only three of the soils were assigned to their hydrometer-defined texture class. This is possibly due to the inappropriate dispersion treatment. The dispersant is added to the soil prior to analysis, but the concentration and/or time and type of interaction with the soil are insufficient.

3.1.5 | Analysis parameters: Refractive index

The refractive index of 1.440 - 0.100i resulted in a better agreement between LD and the hydrometer method for most of the textural classes. Figure 9 shows two of the tested soils in which we can see that the higher RI further underestimates the clay content of the soil sample, a tendency also observed in the other soils. Eshel et al. (2004) observed a similar behavior of the clay and silt fractions when RI increases. The authors defend the use of lower RIs to offset the likely overestimation of non-spherical particles by laser diffraction analysis. By using the 1.44 - 0.100i RI, LD correctly classified the texture of the five soils tested in 56% of the samples compared to 29% of the samples when using 1.543 - 0.010i.

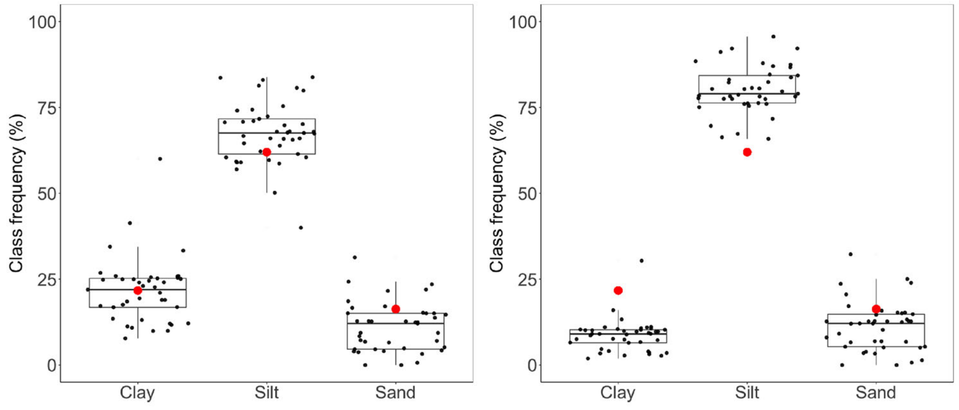
Refractive index is a user-input parameter for LD that uses Mie scattering as the measurement principle. The RI of a material is a function of its particle size and composition (Di Stefano et al., 2010). Because soils are composed of many minerals and particles of different compositions and structures, it is difficult to establish one RI to perfectly encapsulate the optical properties of any soil. Approximately half of the known minerals have real RIs between 1.475 and 1.700 (Frost, 1983), and real RI values ranging from 1.42 to 1.6 have been reported in the literature for laser diffraction applications (Arriaga et al., 2006; Eshel et al., 2004; Faé et al., 2019).

Because it is impossible to determine the actual optical coefficients of soils and sediments, different assumptions and approaches have been reported in the literature (Bieganowski et al., 2018). Some works adopted the established RI of known media. For instance, Thomas et al. (2021) used 1.6 as the real RI, which is the RI for standard polystyrene latex spheres. Faé et al. (2019) used 1.543, the RI for quartz. In a comparison between LD and sedimentation methodologies, Arriaga et al. (2006) reported a real RI of 1.42 producing comparable results with the pipette method after testing RI values between 1.40 and 1.54. In an evaluation of the residual between the fitted and corrected data in an LD analysis, Ryżak and Bieganowski (2011) reported that RIs of 1.43, 1.533, 1.444, and 1.577 showed comparable small values for the residuals.

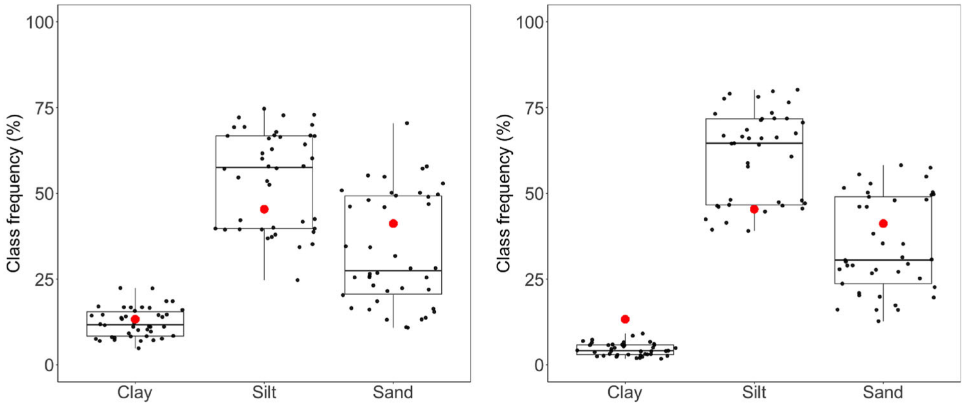
The RI of 1.44 - 0.100i is the manufacturer-recommended RI to use for soil and sediment applications (Horiba Scientific, 2023). However, some authors have argued that values outside the 1.475 and 1.700 range mischaracterize soil particles and their optical properties (Eshel & Levy, 2007). We argue that because soils are not only composed by mineral particles, and organic matter was not removed in this analysis, the RI of 1.44 - 0.100i is appropriate for soils and sediments. Although the optical properties of soil organic matter are not well-known, organic matter in marine sediments has an RI between 1.01 and 1.10 (Bieganowski et al., 2018; Jonasz, 1991). Additionally, the Chi-square and R parameter confirmed the choice for 1.44. These parameters compare the measured scattered light with the calculated scattered light based on the chosen RI. A lower value for both Chi-square and R parameter was obtained for 1.44 compared to 1.543 for the majority of the samples tested, indicating a better fit of the distribution produced by using 1.44 as the RI. However, when considering the imaginary term of the RI, that is, the term of the RI that correlates to the degree of transparency of the particle, the R parameter and Chi-square show contrasting results. The Chi-square indicates that 1i is superior to 0.100i in 77% of the samples; the R parameter indicates that 0.100i is the most appropriate in 72% of the tested samples. The different imaginary terms did not result in change on the textural classification (Figure 10), meaning that for the purpose of this analysis, the imaginary term did not have a significant effect on the texture classes. We opted to adopt 1.44 - 0.100i as the RI because it produced the most comparable clay, silt, and sand fractions to the hydrometer method, as measured by the RMSE.

3.1.6 | Analysis parameters: Sonication time

By testing different times of sonication (20, 60, and 180 s), we found that, for silt and clay, 180 s of sonication resulted in most comparable results with the hydrometer method



(a) Bahem: 1.440 - 0.100i (b) Bahem: 1.543 - 0.010i



(c) Declo: 1.440 - 0.100i (d) Declo: 1.543 - 0.010i

FIGURE 9 Frequency of clay, silt, and sand of the Bahem (a, b) and Declo soils (c, d) as measured by the laser diffraction method in comparison to the hydrometer method (%). The red dots represent the frequency of clay, silt, and sand as measured in the hydrometer method. The boxplot displays the median and the first and third quartiles (lower and upper hinges). The whiskers extend from the hinge to |1.5 × interquartile range|.

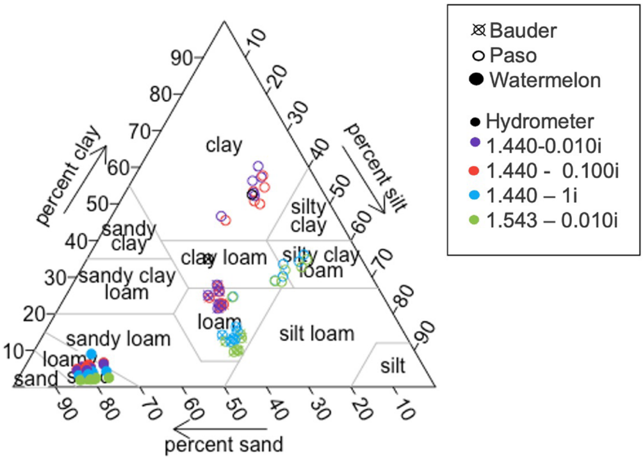


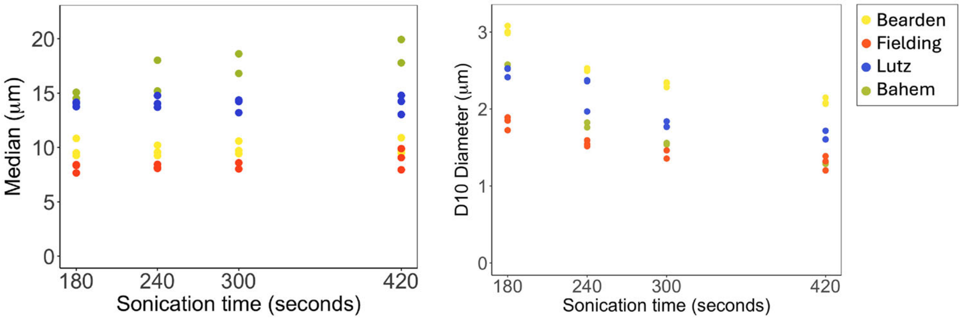
FIGURE 10 Soil texture classification of different soils using different refractive indices (RI) in the laser diffraction (LD) analysis.

TABLE 6 Root mean square error for different sonication times for Bahem and Declo soils. The results indicate the difference (%) between the laser diffraction-derived classes compared to the values measured by the sieve–hydrometer method.

	Bahem soil			Declo soil		
	20 s	60 s	180 s	20 s	60 s	180 s
Clay	8.85	11.77	8.56	7.78	6.47	6.13
Silt	16.34	16.99	13.13	18.13	21.53	15.60
Sand	9.92	14.80	12.82	13.96	19.18	15.86

(Table 6). More time of sonication produced a higher mean of the clay fraction, closer to the clay value measured by the hydrometer, accompanied by a slight decrease in the silt fraction.

Because the analysis indicated that the maximum sonication time tested was the most appropriate, we also tested 240, 300, and 420 s of sonication, aiming to understand the marginal effect of additional sonication time. Out of the six soils tested in this analysis, five showed RMSE for the clay fraction decreasing as time of sonication increased. Contrarily, the RMSE for silt shows that 180 s produces the



(a) Median diameter (b) D10 diameter

FIGURE 11 Mean D10 diameter and median diameter size according to sonication time. The D10 diameter is the diameter at which 10% of a sample is comprised of smaller particles.

most comparable results to the hydrometer classes (data not shown). Sand was removed prior to this analysis and therefore is not directly affected by the sonication. These results show that the clay fraction increases with a longer sonication time, which is supported by the lower D10 observed in these conditions (Figure 11b). However, because the increase on sonication time negatively affected the silt fraction, the choice for the most appropriate sonication requires balancing the impacts on the different soil fractions.

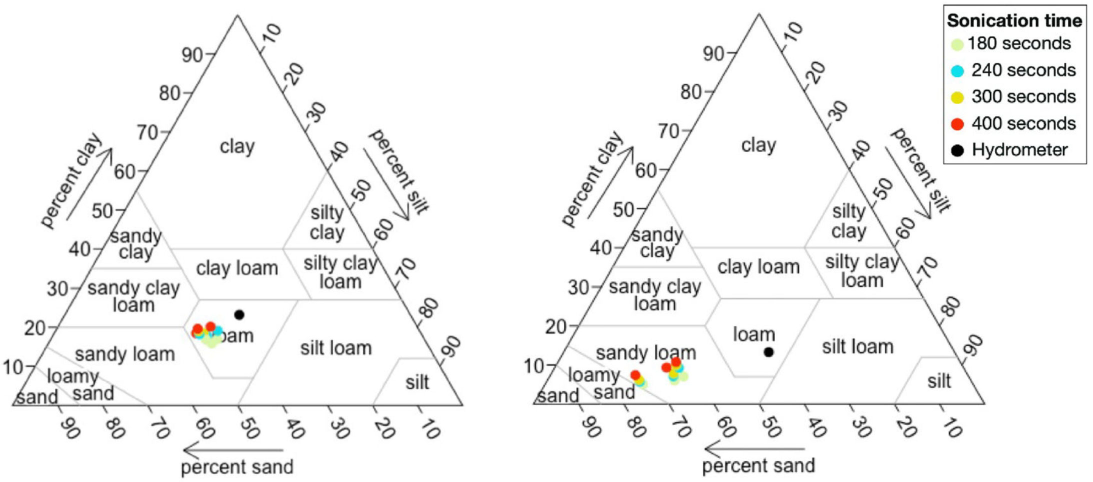
Most of the reported sonication time in the literature varies from 60 to 240 s (Eshel et al., 2004; Faé et al., 2019; Makó et al., 2017; Thomas et al., 2021). Di Stefano et al. (2010) evaluated the effect of sonication time, comparing 60, 120, and 180 s. They found no significant difference in the particle size distributions and opted for using a 120-s sonication time. Svensson et al. (2022) also found little to no effect of sonication on the particle size distribution pattern. The authors used 90 s of sonication (3 x 30 s, power 24 W) in samples that were pre-treated and shaken overnight with a chemical dispersant. Arriaga et al. (2006) also tested different times of physical dispersion, and observed that 360 s yielded similar cumulative frequency distributions compared to 540 s of sonication. They observed that chemical dispersion with no sonication was almost as effective as sonicating for 540 s. In this work, we found that increasing the time of sonication did not impact the soil texture classification, because the changes in silt and clay fractions are marginal as sonication times vary. Figure 12 shows the soil texture classification for two of the soils tested.

The appropriate level of sonication is an amount of time and power able to disperse the sample to the single particle state without fracturing individual particles. To further examine the effects of the sonication time, we evaluated the frequency distribution for each treatment. By examining the tails of the frequency distribution, we wanted to identify larger particles disappearing (shorter tail on the right side of the frequency distribution) and being redistributed throughout the particle size distribution curve. This behavior typically indicates the desired sonication time for an appropriate dispersion. Based on the previous literature, the power of sonication applied is unlikely to have caused primary particles to fracture (Callesen et al., 2018; Kaiser & Asefaw Berhe, 2014). We show two examples in Figure 4c,d. The sonication of 180 s moves the distribution tails to the center of the distribution, as evidenced by the higher peak compared to the others. For the longer sonication times, Figure 13 exemplifies how sonication time affects the frequency distribution. The longer sonication times do not show signs of unwanted breakage of individual particles, but they affect soils differently.

The effects of sonication time on soil texture classification are marginal. Although longer times resulted in higher clay fractions, the opposite effect on the silt fraction also needs to be considered. Because of that and the fact that 180 s resulted in an effective dispersion treatment when summed to the chemical dispersion, we considered 180 s to be the most appropriate sonication time. Additionally, as time of sonication increases, the median diameter does not decrease with each measurement (Figure 11a), which is an indication that disaggregation was effective (Bieganowski et al., 2018). We observed that sonication times as high as 420 s did not show signs of fracturing primary particles.

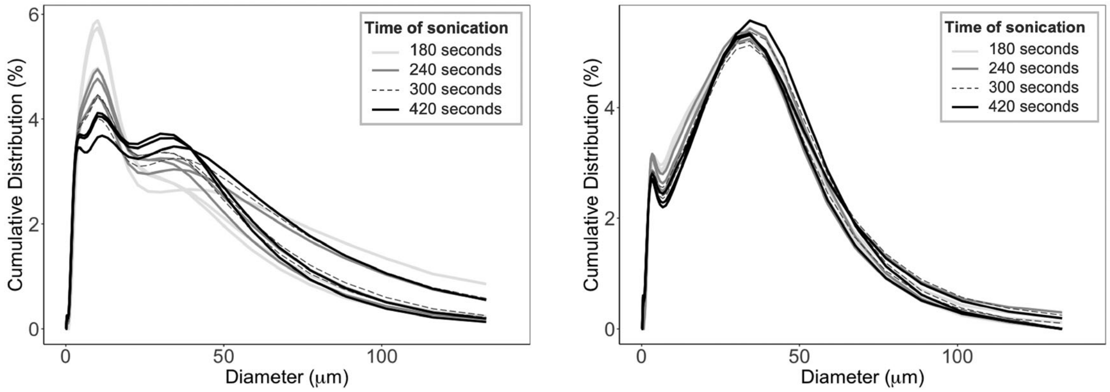
3.2 | Laser diffraction final protocol

The analysis parameters that produced LD-derived classes most comparable to the hydrometer-defined classes are: (1) chemical dispersion by shaking the soil with 5% Na-HMP for 1 h (2) sand removal, (3) addition of entire sample to analyzer followed by transmittance matching, (4) RI of 1.44 - 0.100i, (5) 180 s of sonication. Out of these parameters, we observed that mode of sample addition is one of the most impactful parameters for matching the LD analysis to the widely used hydrometer. By adding the entire sample, we avoided subsampling errors and we generated reproducible results. The final protocol is available in Figure S1. We encourage its use as an standard operating procedure for LD analysis of soil texture.



(a) Fielding (b) Declo

FIGURE 12 Soil texture triangles showing the impact of different sonication times to the soil texture classification of two of the soils tested, (a) Fielding and (b) Declo.



(a) Bearden (b) Lutz

FIGURE 13 Effect of time of sonication on the cumulative frequency distribution of the Bearden (a) and Lutz (b) soils.

Figure 14 shows the LD classification of all 18 soils for the LD analysis conducted with the established parameters. This analysis resulted in 14 soils being correctly classified and it attests the high replicability of the proposed LD method; for 12 soils, the same texture classification was observed for all replicates. For the soils that were incorrectly classified, the underestimation of the clay factor is the common cause, as mentioned before. The comparison between LD and hydrometer produced similar results compared to that between the hydrometer and the pipette method.

The comparison between soil texture methodologies is a long-sought research topic. The sometimes contrasting results in the literature stem from the inherent biases and sources of error from each methodology as well as the different physical principles that govern sedimentation and LD methods (Eshel et al., 2004). Given the many parameters of the LD analysis and the complex nature of soils, standardizing LD for soil texture analysis is fundamental for routine analysis as well as for building a soil database in which different characteristics of the soil particle distribution can be explored. In this work, we contribute to the literature by proposing a common protocol based on previous results in the literature and our own detailed analyses that can be adopted in routine soil texture analysis. While we tested a range of soils, further validation of this study will improve the confidence in its reliability. The use of a common protocol is essential for reproducible results and comparison between works.

3.3 | Applying reported relationships between particle size analysis methods

After establishing the optimal levels of the tested parameters, we calculated the mean clay, silt, and sand fractions for all samples analyzed using those parameters (Table 7). These

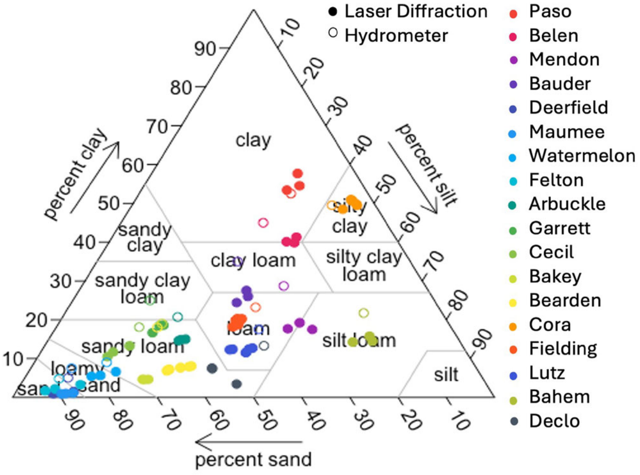


FIGURE 14 Soil texture of all soils as established by the laser diffraction analyses conducted with the parameters that produced laser diffraction-defined classes most comparable with the hydrometer method.

TABLE 7 Mean and standard deviation of clay, silt, and sand fractions as measured by laser diffraction analysis using the optimal levels of the tested parameters.

Soil	Frequency clay (%)	Frequency silt (%)	Frequency sand (%)
Arbuckle	14.81 ± 0.19	27.69 ± 0.63	57.50 ± 0.81
Bahem	14.82 ± 0.81	65.70 ± 2.03	19.48 ± 2.44
Bakey	4.62 ± 0.02	25.15 ± 0.62	70.23 ± 0.62
Bauder	25.98 ± 1.55	34.96 ± 0.78	39.06 ± 1.95
Bearden	7.43 ± 0.45	30.33 ± 1.85	62.25 ± 2.29
Belen	40.41 ± 0.74	37.85 ± 0.94	21.74 ± 1.32
Cecil	11.81 ± 1.40	15.69 ± 1.72	72.51 ± 3.08
Cora	50.02 ± 0.96	45.40 ± 0.98	4.58 ± 1.33
Declo	6.11 ± 2.32	40.03 ± 4.09	53.86 ± 1.77
Deerfield	1.19 ± 0.26	9.26 ± 2.31	89.56 ± 2.57
Felton	2.38 ± 0.82	8.68 ± 3.36	88.94 ± 4.17
Fielding	19.50 ± 0.86	36.95 ± 0.54	43.56 ± 1.02
Garrett	17.90 ± 1.09	21.30 ± 0.66	60.80 ± 1.69
Lutz	12.15 ± 0.50	41.64 ± 1.94	46.21 ± 1.92
Maumee	1.00 ± 0.11	10.74 ± 1.17	88.26 ± 1.28
Mendon	18.13 ± 0.95	50.54 ± 2.64	31.32 ± 2.53
Paso	55.20 ± 2.23	30.92 ± 1.16	13.87 ± 2.26
Watermelon	5.90 ± 0.63	15.71 ± 2.26	78.39 ± 2.87

values were used as input data for the predictive models shown in Table 8.

In the mathematical functions established by Faé et al. (2019), the regression of LD data against the hydrometer method in the original research resulted in R^2 values of 0.92, 0.92, and 0.99, for clay, silt, and sand, respectively, and was formulated based on the analysis of 54 NAPT soil standards. In their analysis, the authors also removed the sand fraction via sieving prior to the LD analysis (53 mm), and employed a refractive index of 1.543 - 0.01i with a clay–silt cutoff of 5.92 µm. The analyses used pre-treated subsamples (5% Na-HMP, overnight soaking).

TABLE 8 Reported quantitative relationships between hydrometer and laser diffraction. Laser diffraction is being referred to as LD and sieve–hydrometer by SH; CL_{LD} and SA_{LD} stand for the clay and sand fractions measured by LD, respectively.

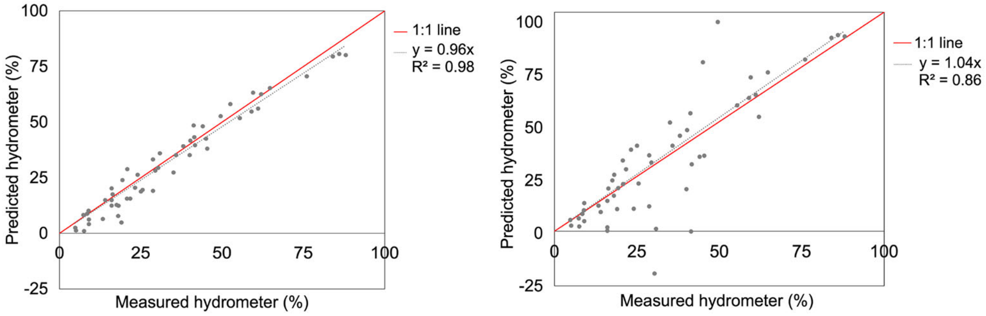
Size fraction	Faé et al. (2019)	Di Stefano et al. (2010)
Clay	$LD = 0.95 \times SH - 0.004$	$SH = a^{a} \times LD$
Silt	$LD = 1.05 \times SH + 0.02$	$SH = 100 - a \times CL_{LD} - SA_{LD}$
Sand	$LD = 1.11 \times SH - 0.08$	$SH \approx LD$

[a] a is 2.18 for samples with sand >50%, and 1.91 for samples with sand ≤ 50%.

In the second set of equations, Di Stefano et al. (2010) reported an RMSE of 9.27%, 9.05%, and 2.16%, for clay, silt, and sand, respectively. The authors also removed the coarser fraction of the sample (≥710 µm), and the 1.5-mL soil suspension aliquot was introduced into the dispersion unit after overnight shaking with Na-HMP. The equations by Di Stefano et al. (2010) show a greater scatter comparatively and were developed based on the analysis of 228 soil samples.

In this attempt to apply mathematical functions formulated based on other datasets to our data (Figure 15), we identified a good fit with one set of equations (Faé et al., 2019), the relationship between the measured and predicted hydrometer measurements resulted in an R^2 of 0.98. We hypothesize that this is because Faé et al. (2019) used similar parameters in the LD analysis compared to the optimal parameters proposed from the results of the current research. This reinforces the importance of having a common set of parameters for LD analysis. We showed that one can predict hydrometer-derived clay, silt, and sand fractions based on the LD-derived classes with a high degree of certainty if similar parameters are used in the analysis. The model by Di Stefano et al. (2010) showed a higher variability and it evidences the difficulty in having a unique model for relating hydrometer and LD-based data, given the variability of parameters in the LD analysis.

There are strong arguments in the literature against the approach of relating and matching particle size distribution data from different methods (Eshel & Levy, 2007). Some researchers argue that there is an inherent method dependence for particle size distributions and any attempt to compare methodologies would be an oversimplification. We argue that although a quantitative relationship between hydrometer-derived data and LD data cannot be easily generalized, creating optimal conditions for LD to produce similar results to sedimentation methods is critical to contribute to the advancement of new methods and to establish new standards for particle size analysis.



(a) Faé et al. (2019)

(b) Di Stefano et al. (2010)

FIGURE 15 Relationship between the hydrometer-derived clay, silt, and sand fractions and the predicted fractions by the laser diffraction method using the equations developed by (a) Faé et al. (2019) and (b) Di Stefano et al. (2010).

The use of more advanced technologies such as SEM and transmission electron microscopy (TEM) allows the direct measurements of the absolute surface area of individual particles and therefore, has the potential to resolve any methodological debates. A work comparing sedimentation methods, LD, and SEM indicated that the standard sedimentation methods wrongly classified silt-sized particles in the clay fraction, while similar particle size diameters were observed between SEM and LD (Yang et al., 2019). This evidences the need to advance the currently used methodologies for soil textural analysis.

4 | CONCLUSION

The use of LD for soil and sediment applications has been studied for a few decades, and findings evidence the potential effectiveness of LD in determining particle size distribution. However, adoption of this methodology as standard soil texture analysis is still incipient. One limiting practical aspect for its adoption is the costly investment, which means laser diffraction analyzers may not be economically feasible in many laboratories. Aside from that, researchers who already use LD still need to adopt a common methodology that allows for direct comparisons between soil texture analyses.

The methodology identified in this work for LD routine soil texture analysis consists of (1) 1 g of ≤2 mm soil sample is mixed with 100 mL of 5% Na-HMP solution and shaken (end-over-end shaker) for 1 h, (2) sand separation, (3) sample is added entirely to analyzer, (4) analysis is conducted with sample constant agitation, (5) transmittance levels are adjusted by diluting sample in bath, (6) an RI of 1.44 - 0.100i, and (7) 180 s of sonication. Carbonate removal did not have significant effects on soil texture classification in this research, and literature supports that it is only necessary in cases in which these are primary components in soils and sediments, which was not the case in this analysis. The standard operating developed from this work is included in Figure S1.

We found that empirical relationships between soil textural methods are largely restricted to the dataset used if LD parameters are not standardized, that is, a better agreement can be expected for datasets that were produced using similar LD analysis parameters. Using a common LD protocol while concurrently building a direct link between LD results and soil properties are necessary approaches to advance particle size analysis. Future works should include the testing of this protocol for other soils and regions, accompanied by the continuous development of a database with comparable results from LD analysis.

AUTHOR CONTRIBUTIONS

Isis S. P. C. Scott: Conceptualization; data curation; formal analysis; investigation; methodology; validation; writing—original draft; writing—review and editing. **Kossi Nouwakpo**: Conceptualization; investigation; methodology; supervision; writing—review and editing. **Dave Bjorneberg**: Methodology; resources; supervision; writing—review and editing. **Christopher Rogers**: Data curation; resources; writing–review and editing. **Lauren Vitko**: Conceptualization; investigation; methodology; writing—review and editing.

CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

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SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

How to cite this article: Scott, I. S. P. C., Nouwakpo, K., Bjorneberg, D., Rogers, C., & Vitko, L. (2024). Establishing a standard protocol for soil texture analysis using the laser diffraction technique. *Soil Science Society of America Journal*, 1–18. https://doi.org/10.1002/saj2.20738