Assessing Repeatability and Precision of Dosing Techniques in Soil Particle Size Distribution Analysis Using Laser Diffraction

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Abstract

This study investigates the precision and reliability of various dosing repeatability techniques for laser diffraction particle size analysis in soil samples, focusing on the impact of dosing methods on measurement reproducibility and accuracy. Three different dosing techniques (A) manual pipetting with a shaker, (B) a mash using a spatula, and (C) a dried sample using a spatula) were evaluated using a laser diffraction analyser. Soil samples representing sandy, loamy, and clayey types were analysed to assess the relative standard deviations (SD) for particle size measurements. The results were compared to traditional pipetting methods to identify discrepancies and evaluate the impact of dosing techniques on measurement precision. Significant variations in measurement precision were observed among the dosing techniques. Manual pipetting technique (A) exhibited higher relative SDs, with average values of 22.4%, indicating substantial variability and lower repeatability. In contrast, techniques B and C achieved lower relative SDs, averaging 8.1% and 7.9%, respectively. The study also confirmed that laser diffraction tends to underreport clay fractions and overreport silt fractions compared to pipetting. The results highlight the critical role of dosing technique in determining measurement precision for laser diffraction particle size analysis. Carefully optimized manual methods (such as techniques B or C) can still achieve high levels of precision, approaching those of automated dosing systems. These insights are essential for improving analytical practices and ensuring reliable soil particle size measurements in various applications.

Keywords: *soil particle size sistribution (PSD), laser diffraction analysis, sample dosing techniques, repeatability assessment, standard deviation analysis*

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1 Introduction

Soil particle size distribution (PSD) is a crucial parameter across various disciplines, including agronomy, environmental science, and civil engineering. An accurate understanding of soil PSD is essential for determining soil texture, permeability, water retention capacity, and overall fertility. Laser diffraction is a sophisticated method for PSD analysis that provides rapid, precise, and detailed characterization of soil particles, ranging from clay to sand sizes (Eshel *et al.*, 2004; Beuselinck *et al.*, 1998).

Laser diffraction analysers operate based on the principle of light scattering. As a laser beam passes through a dispersed soil sample, particles scatter light at angles inversely proportional to their size. This scattered light is detected and analysed to generate a particle size distribution profile (Murray, 2002). This technique is favoured for its high resolution and capability to analyse a wide range of particle sizes in a single measurement. However, achieving reproducible results with laser diffraction in soil PSD analysis can be challenging. Variability in sample preparation, dispersion, and measurement conditions can lead to discrepancies between repeated measurements. Consistency in these factors is critical for reliable data, particularly when classifying soil according to the USDA soil texture triangle (Gee and Or, 2002).

A common issue in laser diffraction PSD analysis is the variability observed in repeated measurements from different aliquots of the same sample. Such variations can stem from several sources: (i) Sample Homogeneity: Soil samples may contain aggregates or unevenly distributed particles, affecting the representativeness of small aliquots (Loizeau *et al.*, 1994); (ii) Dispersion Efficiency: Inadequate dispersion can result in the presence of flocs, while over-dispersion may break down natural aggregates, both of which can lead to inconsistent results (Ryżak and Bieganowski, 2010); and (iii) Aliquot Handling: Manual pipetting or sampling can introduce variability due to differences in the amount or composition of material taken for each measurement (Konert and Vandenberghe, 1997).

Achieving reproducible results in PSD measurements using laser diffraction involves addressing these multifaceted challenges. Numerous studies have focused on identifying and mitigating sources of variability. Eshel *et al.* (2004) critically evaluated laser diffraction for PSD analysis, highlighting the importance of uniform sample preparation procedures. They found that inconsistencies in sample pre-treatment could lead to significant deviations in measured particle size distributions, underscoring the need for standardized methodologies. Proper dispersion of soil particles is essential to prevent aggregation and ensure accurate measurement of individual particle sizes (Beuselinck, *et al.*, 1998).

Manual handling of soil samples can also contribute to variability due to differences in aliquoting and mixing techniques. Automated systems for sample handling and dispersion have been developed to minimize human-induced variability. Konert and Vandenberghe (1997) compared manual and automated sample handling methods, finding that automated systems offered greater consistency and reproducibility. Loizeau *et al.* (1994) assessed a wide-range laser diffraction grain size analyzer for sediments, noting the challenges of obtaining representative aliquots from heterogeneous samples. Their findings suggest that improving aliquoting techniques is crucial for reproducible PSD measurements. Di Stefano *et al.* (2010) emphasized the importance of aliquoting techniques and proposed using automated pipetting systems to enhance reproducibility, demonstrating that automated pipetting reduces variability introduced by manual methods. Xu and Di Guida (2003), confirmed by Callesen *et al.* (2018), found that automated sample dispersion and measurement systems significantly improved repeatability. Roberson and Weltje (2014) compared various particle size analyzers, highlighting the impact of sample introduction methods on measurement accuracy. Automated dosing systems generally reduce operator-induced variability and improve repeatability, while manual methods, though flexible, can be prone to inconsistencies based on operator technique. Miller and Schaetzl (2011) observed that the coefficient of variation

(CV) for repeated measurements of the same sample ranged from 1% to 15%, depending on soil type and preparation method, emphasizing the influence of manual handling and operator variability on measurement reproducibility.

The primary objective of this paper is to assess the repeatability of soil sample dosing techniques into the wet dispersion unit of a laser diffraction analyzer. Specifically, the study aims to evaluate the consistency of three different dosing methods (hand pipette with shaker, mash using a spatula, and dried sample using a spatula) by analyzing the particle size distribution (PSD) measurements. The evaluation will be conducted using the standard deviation (SD) as a measure of variability, with the goal of identifying the dosing technique that provides the most reliable and reproducible results. This research seeks to contribute to the standardization of soil sample preparation and dosing methods in laser diffraction analysis, ultimately improving the accuracy and reliability of soil texture classification.

2 Material and Methods

2.1 Laser Diffraction Particle Size Analysis

In this study, particle size distribution (PSD) measurements were conducted using the Analysette 22 NeXT Nano laser diffraction analyser from Fritsch. This advanced instrument features a wide measuring range from 0.01 to 3800 μm, allowing for precise analysis of various particle sizes. The Analysette 22 NeXT Nano utilizes the MaS control software and supports both Fraunhofer and Mie scattering theories, providing flexibility in data analysis and interpretation. The device is equipped with a green laser with a wavelength of 520 nm and an approximate power of 1 mW, ensuring accurate and reliable measurements. (Fritsch, 2020).

The optical system of the analyser is based on a reverse Fourier design, optimizing measurement accuracy. The instrument also includes additional modules such as a wet dispersion unit and an ultrasonic box, which enhance its capability to handle different sample types and preparation methods. The suspension volume for measurements ranges from 150 to 500 ml, and the device features a radial pump with an adjustable speed of up to 3.5 l/min. Large-angle detectors and backward scattering channels contribute to the comprehensive analysis of particle size distributions. (Fritsch, 2020).

2.2 Preparation of the Soil Sample

Before introducing the soil sample into the laser particle analyser, meticulous preparation is necessary. This preparation follows the methodology established by Lisá (2016) and is not standardized like the pipetting method (classic sedimentation method).

(i.) Natural Drying: The collected soil sample was left in laboratory conditions for natural drying to ensure consistent moisture content before further processing.

(ii.) Sample Grinding and Sieving: After drying, the soil was gently ground using a mortar and then sieved through a 2 mm sieve. This process was aimed at breaking down larger aggregates and standardizing the particle size for analysis.

(iii.) Chemical Treatment: Approximately 5-10 g of the sieved soil was transferred to a test tube and dispersed in a 10% potassium hydroxide (KOH) solution. This treatment was intended to remove organic materials, oxides, carbonates, and other potential contaminants.

(iv.) Boiling for Enhanced Effectiveness: The test tube containing the soil-KOH suspension was boiled in water to enhance the effectiveness of the KOH treatment and ensure the thorough removal of residual organic matter.

(v.) Centrifugation: Following boiling, the suspension was subjected to centrifugation to ensure thorough mixing and complete coating of each soil grain with the reagent.
(vi.) Finally, the KOH solution was decanted.

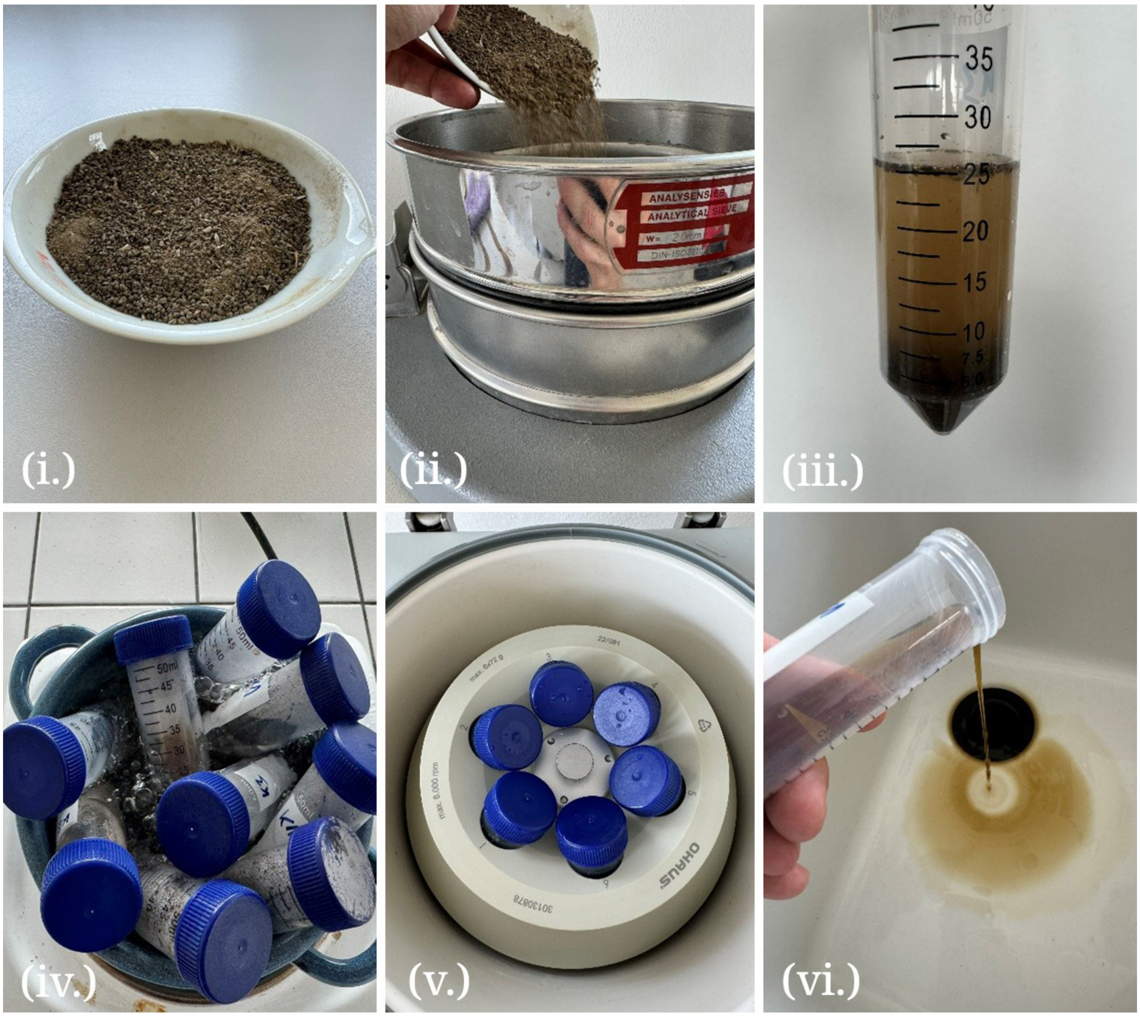

Fig. 1: *Soil sample preparation*

This procedure (step i-vi) is demonstrated in Figure 1. Soil samples were subsequently processed in the following three hand techniques:

- Technique A: After decantation, the suspension was replaced with distilled water. The resulting slurry was pipetted into the dispersion unit of the laser analyser, with a Vortex shaker used to achieve sample homogenization.
- Technique B: After decantation, a very small amount of distilled water was added to the soil to create a mash, which was then scooped into the dispersion unit of the laser analyser.
- echnique C: After decantation, the sample was allowed to dry before being dosed into the dispersion unit of the laser analyser.

These techniques (A-C) are demonstrated in Figure 2.

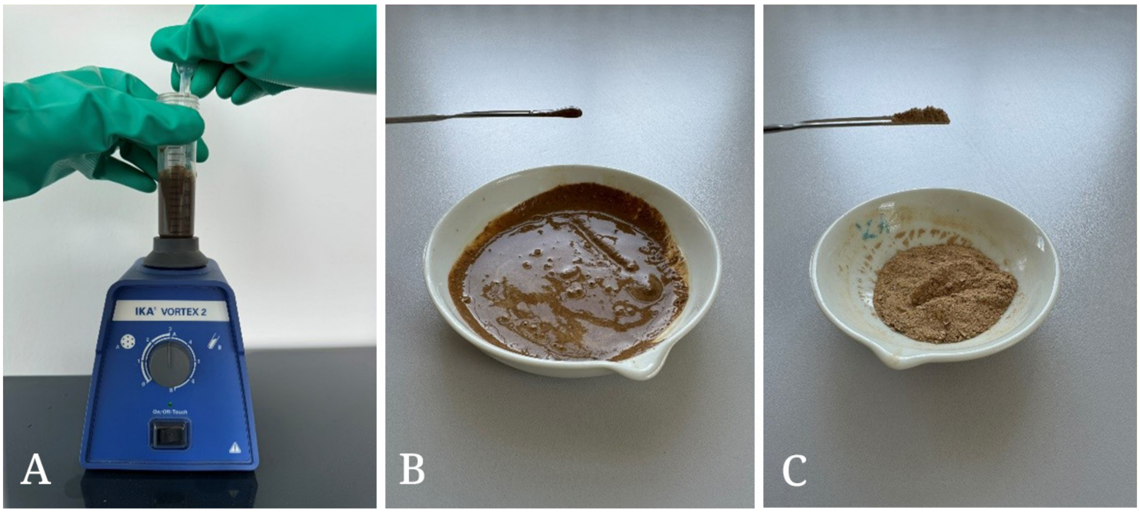

Fig. 2: *Schematic representation of soil sample processing and dosing techniques for laser diffraction analysis*
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2.3 Standard Operating Procedures

Standard Operating Procedures (SOPs) for sample measurements were established based on both practical experience and knowledge (Paseka, 2022) as well as research findings (Bieganowski *et al.*, 2018). Measurements were conducted under consistent SOP settings to ensure accuracy and reproducibility. The pump was set to operate at 80% capacity, corresponding to a flow rate of 2.8 l/min. The suspension volume was maintained at 90%, equating to 450 ml. Ultrasound power was consistently set to 50%, translating to 25 watts.

The obscuration level was controlled within the range of 15–20% to optimize signal detection and minimize measurement errors. Both the dark and background measurements were set to 10 seconds to address baseline noise and interference. The Mie scattering method was employed, with measurements lasting 25 seconds each and a total of 5 repetitions to ensure precision and statistical reliability.

For dispersion parameters, the refractive index (RI) for solid particles (silica) was set to 1.45, and for the liquid (distilled water), it was set to 1.33. The absorption coefficient (AC) was set to 0.3. These standardized settings provided a controlled environment for accurate and consistent sample analysis.

2.4 Soil Texture Classification and Measurement Procedures

Soil texture classifications were determined by analyzing the grain size distribution based on USDA soil taxonomy standards, with boundaries defined for sand (50–2000 μm), silt (2–50 μm), and clay (>2 μm). The grain size curve obtained from the analysis allowed for precise classification of soil textures. A total of 3 soil samples with varying textures, sandy, loamy and clayey, were tested to ensure comprehensive coverage.

Each sample was prepared uniformly and analyzed using three distinct dosing methods: Technique A) using a hand pipette with a shaker, Technique B) as a mash using a spatula, and Technique C) as a dried sample using a spatula. Each method (A-C) was tested by dispensing the sample into the dispersion unit of the laser diffractor 10 times, with each measurement repeated 5 times, resulting in a total of 450 individual measurements.

For comparative analysis, a case study was conducted using the classic sedimentation method, which involves pipetting and is based on the Stokes velocity relationship. In this case, a single measurement was performed for each soil sample.

2.5 Statistical Analysis

For the evaluation of the dosing methods (Techniques A, B, and C), basic statistical measures were employed to assess the repeatability and reliability of the measurements. The primary statistical tools used were the arithmetic mean and the standard deviation. These metrics were chosen for their simplicity and effectiveness in summarizing the data and identifying variability (Freedman *et al.*, 2007).

- Arithmetic Mean: The arithmetic mean, or average, is calculated by summing all the measurement values and dividing by the total number of measurements. It provides a central value (mean value denoted as E) that represents the typical measurement outcome for each dosing method.
- Standard Deviation: The standard deviation (SD) is a measure of the amount of variation or dispersion in a set of values. It is calculated by determining the average distance of each measurement from the mean. A lower standard deviation indicates that the measurements are closely clustered around the mean, suggesting high repeatability and a more reliable dosing mechanism. We analysed the data in the context of relative standard deviations and numerically in the context of ±1 SD from the mean. This approach is based on the principle that approximately 68% of the values in a normally distributed dataset lie within two standard deviations of the mean.

Assessment of Dosing Mechanisms: In this study, the repeatability of the dosing mechanisms (Techniques A, B, and C) was evaluated by comparing the standard deviations of the measurements. A smaller standard deviation indicates less variability and better repeatability, suggesting a more consistent and reliable dosing mechanism. This statistical approach ensures that the evaluation is both quantitative and objective, providing clear insights into the performance of each dosing mechanism.

3 Results

The data obtained from the Analysette 22 NeXT Nano laser diffraction analyzer offers a comprehensive overview of the particle size distribution (PSD) across three soil samples. The results, presented in graphs and tables, highlight key parameters such as mean particle size (E), standard deviation (SD), and the proportions of different particle size fractions (soil 1: sandy, soil 2: loamy, soil 3: clayey). This section details the findings, emphasizing the reproducibility of measurements, the impact of different dosing methods, and the implications for soil classification and analysis.

The analysis reveals significant variations in PSD depending on the dosing technique used. As illustrated in Figure 3, the highest variability in particle size distribution was observed with Technique A (manual pipetting into the laser), indicated by the broader range of values (E ± 1SD). In contrast, Techniques B and C produced more consistent results, suggesting that these methods offer more stable and reproducible measurements compared to manual pipetting.

Figure 3 shows that the laser diffraction method generally provided lower estimates for fine particles (clay fractions) compared to the traditional pipette method, particularly in Soil 3. This phenomenon carries important implications for soil classification and subsequent analyses. The differences in PSD results between laser diffraction and the pipette method could lead

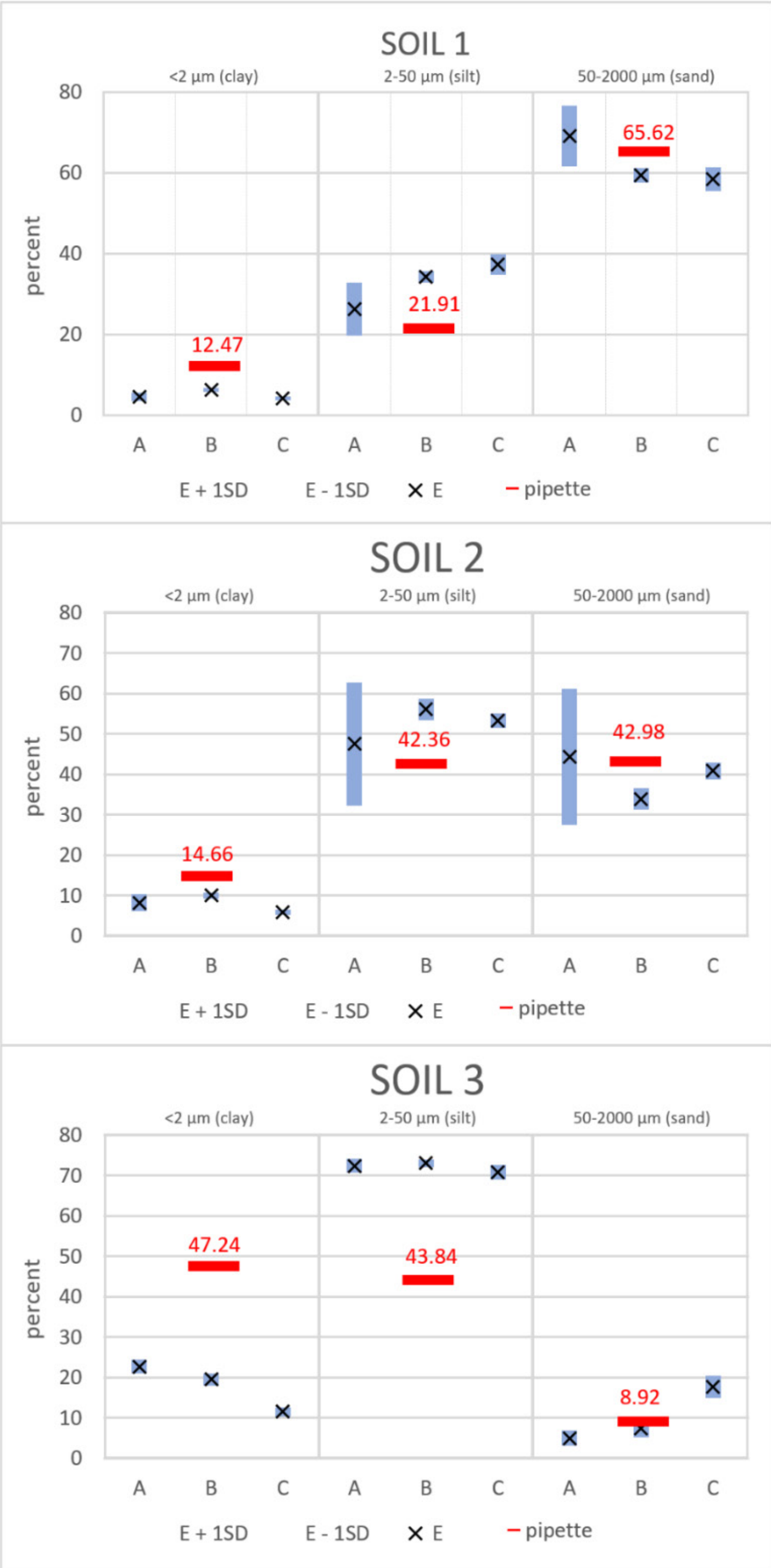

Fig. 3: *Evaluated results of the mean E values from the grain size curves for the cutoff values of clay, silt and nd sand including standard deviations for the 3 variants (A, B and C) of sample dosing into the laser and the measured value by the traditional pipetting method (in red in the graph)*

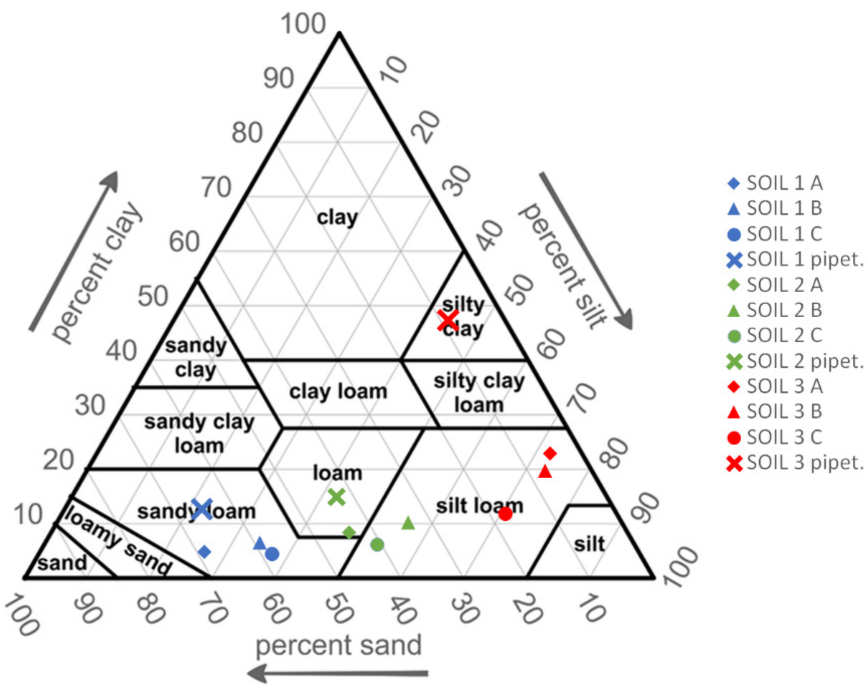

Fig. 4: *USDA Soil Texture Triangle Comparing Laser Diffraction and Pipette Methodologies across Different Dosing Techniques.*

to variations in soil texture classification, potentially affecting agricultural decision-making, environmental assessments, and other applications. However, this inaccuracy was not the primary focus of this study.

The analysis presented in Figure 4 illustrates the disparities in soil texture classification as determined by the USDA soil texture triangle, comparing results from three soil samples subjected to different dosing techniques into the laser diffraction analyser (Techniques A, B, and C) and the classical pipette method.

It is evident that the pipette method consistently yields higher clay content percentages compared to the laser diffraction technique, regardless of the dosing method used. This discrepancy is particularly pronounced in Sample 3, where the laser diffraction method underestimated the clay fraction by approximately 50%, leading to a significant shift in soil classification. In contrast, the sand fraction measurements obtained by the laser are relatively consistent with the pipette method, indicating that the laser method may reliably assess coarser fractions but struggles with finer particles.

While the primary aim of the study was not to compare the classical pipette method with laser diffraction but to determine the superiority of one dosing technique over another, conducting pipette measurements on the samples was essential. This allowed us to evaluate the bias introduced by each dosing technique when compared to the pipette method. The results indicate that all dosing techniques consistently diverged from the pipette method in a similar direction and to a comparable extent. Consequently, the conclusions will focus more on analysing the standard deviation (as a measure of repeatability) rather than the comparison of different techniques relative to their deviation from the pipette results. Furthermore, the limited number of soil samples tested is insufficient for drawing robust conclusions about the magnitude of this deviation.

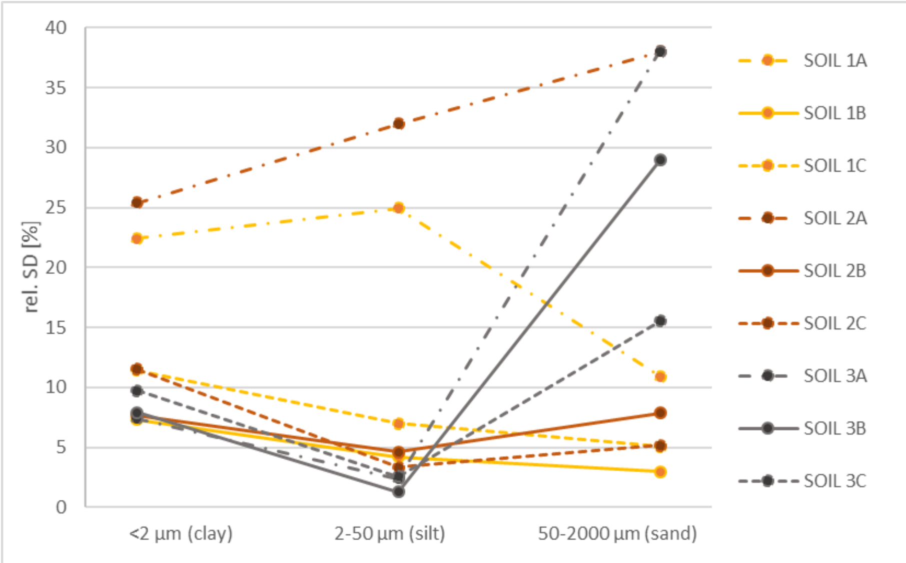

Fig. 5: *Relative standard deviation in percent across clay, silt, and sand fractions for three Dosing Techniques (Techniques A, B, and C)*

While the results in Figure 4 highlight the discrepancies in soil texture classification due to different dosing techniques, Figure 5 shifts the focus to evaluating the precision of these techniques. By analysing the relative standard deviations (SD) across various soil fractions, the study identifies which dosing methods yield the most consistent and reliable measurements, independent of the comparison between laser diffraction and the pipette method.

The analysis of relative standard deviation (SD) for various soil fractions (clay, silt, sand) across the three dosing techniques reveals critical insights into the precision of each method. The data indicate that Technique A consistently results in the highest relative SD, suggesting significantly greater variability and lower repeatability in measurements obtained using this method. Conversely, Techniques B and C exhibit markedly lower relative SD values, generally remaining within 10%, except for the sand fraction.

Furthermore, a substantial increase in SD is observed in the sand fraction of Soil Sample 3 (clay-rich), where variability is notably higher, particularly when using Technique A. This finding underscores the challenges of achieving consistent dosing with this method in soils with high clay content.

The results demonstrate that Techniques B and C provide more reliable and reproducible measurements, which are crucial for accurate soil texture classification and subsequent analyses. The lower variability observed with these techniques further supports their suitability for precise particle size distribution (PSD) analysis, especially in complex soil matrices such as those with high clay content. The significant differences in measurement accuracy among all three techniques also suggest that the choice of dosing method can substantially influence the outcome of soil texture classification and related applications.

Figure 6 provides a focused analysis on the precision of different techniques. By examining the relative standard deviations (SD) for each soil fraction across the three dosing methods, Figure 6 allows for a detailed comparison of the repeatability and reliability of the dosing techniques.

Figure 6 presents a column graph that clearly delineates the relative standard deviations for clay, silt, and sand fractions, as measured by the laser diffraction analyser, for each of the three dosing options (Techniques A, B, and C). The graph shows that Technique A (represented

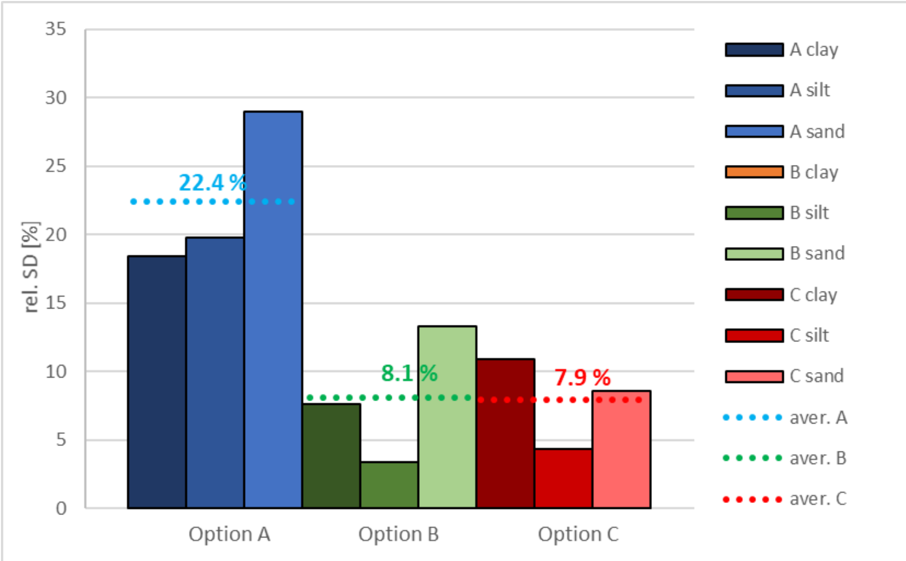

Fig. 6: *Relative standard deviation (%) across clay, silt, and sand fractions for three dosing techniques (Techniques A, B, and C)*

by blue columns) consistently results in the highest relative SD, with an average of 22.4%. This indicates substantial variability and lower repeatability in measurements for this technique. In contrast, Techniques B (green columns) and C (red columns) exhibit significantly lower relative SD values, averaging 8.1% and 7.8%, respectively. The lower variability for Techniques B and C underscores their superior repeatability compared to Technique A.

Among the three techniques, the silt fraction yields the lowest SD for both Techniques B and C, often falling below 5%, which highlights the effectiveness of these methods in providing consistent measurements for silt. The final data in Figure 6 reinforce the results obtained previously and highlight the superior performance of Techniques B and C in achieving reliable and reproducible measurements. This analysis is crucial for identifying the optimal dosing method for laser diffraction analysis, ensuring accurate soil texture classification and reliable particle size distribution data.

4 Discussion

Laser diffraction and pipetting are two commonly employed techniques for particle size analysis in soil samples, each with its own advantages and limitations. One of the major challenges associated with laser diffraction is the potential for overestimation or underestimation of specific particle fractions, particularly fine particles such as clay and silt. Svensson *et al.* (2022) reported significant discrepancies between results obtained using laser diffraction instruments, such as the Mastersizer, and traditional pipetting methods, particularly for clay and silt fractions. Their study highlights that discrepancies between these methods may arise due to suboptimal instrument settings and the selection of cutoff values between different particle fractions, such as clay and silt. According to Weiwen Qiu *et al.* (2021), laser diffraction can underreport clay fractions by up to 62% and overreport silt fractions by 23%, which aligns with earlier findings by Konert and Vandenberghe (1997) and Pieri *et al.* (2006). These differences are critical for soil texture classification as significant deviations in the measurement of clay and silt fractions can impact the final distribution of soil fractions.

The obtained results corroborate these findings, showing that laser diffraction tends to underreport the clay fraction compared to pipetting, with differences reaching up to 50% for clay fractions in this study. Additionally, the overestimation of silt fractions observed aligns with the results reported by Yang *et al.* (2015), indicating a notable percentage increase. These findings suggest that while laser diffraction offers a rapid and efficient measurement approach, its accuracy for fine particles can be influenced by various factors, including instrument settings and manufacturer specifications. It is crucial to recognize that optimizing the Standard Operating Procedures (SOPs) for laser diffraction instruments and selecting appropriate cutoff values for particle classification are essential for minimizing these discrepancies. Furthermore, different instruments may have varying characteristics and specifications that affect measurement results. This issue underscores the need for standardization of methodologies and thorough calibration of instruments to ensure the highest accuracy and reproducibility of measurements across different analytical techniques.

In this study, the evaluation of various dosing techniques for laser diffraction particle size analysis provided critical insights into their impact on measurement precision. This section focuses on the relative standard deviations (SD) of the different dosing methods and their implications for the reproducibility and reliability of soil texture measurements.

Miller and Schaetzl (2011) highlighted significant variability in the coefficient of variation (CV) based on different soil types and preparation methods. Their research demonstrated that CV for repeated measurements ranged from approximately 1% for sandy soils to up to 15% for clayey soils, indicating a substantial influence of soil texture on measurement precision. Similarly, Polakowski *et al.* (2021) examined the reproducibility of laser diffraction for soil particle size analysis and found that the CV was lowest for silt (3.44%) and highest for sand (23.28%). This variability underscores the impact of soil type on measurement outcomes. The obtained results corroborate these findings, showing significant differences in relative SD across the three tested soil samples. Specifically, for dosing Technique B, the average relative SD for clayey soil (Sample 3) was 12.7%, whereas for sandy soil (Sample 1), it was 4.9%. These results reflect the broader trend that soil texture affects measurement precision, with clayey soils exhibiting higher variability compared to sandy soils. Notably, the high SD observed in the sand fraction of clay-rich soils could influence the overall average SD, highlighting the complexities of achieving consistent measurements in heterogeneous soil matrices.

Further analysis revealed that manual dosing methods, such as those employed in techniques A, B and C, exhibited greater variability compared to automated dosing systems. Miller and Schaetzl (2011) noted that manual dosing methods generally show higher variability, with CVs typically ranging from 10% to 15%. In contrast, automated dosing systems tend to offer more consistent results, with CVs around 1–5%. This advantage of automated dosing in achieving reliable particle size distribution (PSD) measurements is evident in this study, where Techniques B and C, which involve more controlled dosing, approached the precision levels of automated methods with relative SDs of 8.1% and 7.9%, respectively.

The observed results underscore the importance of the dosing technique in influencing measurement precision. Techniques A, involving manual pipetting, displayed higher SDs due to the inherent variability in manual processes. In contrast, Techniques B and C, which provide more consistent dosing, resulted in lower relative SDs, although not as low as those typically seen with fully automated systems. This indicates that while automated dosing systems offer superior reproducibility, carefully optimized manual methods can still achieve reasonably high levels of precision.

Additionally, the precision of manual dosing methods can be affected by the operator‘s skill and the inherent variability in the dosing process. For instance, the use of a hand pipette for sample with a Vortex shaker (Technique A) was found to be particularly unsuitable, as it introduced significant variability and potential for human error. Additionally, the resulting average deviation for this technique exceeded the 10–15% range typically associated with manual dosing as reported by (Miller and Schaetzl, 2011). This highlights the need for a meticulous approach when using manual methods and suggests that automated dosing systems, which minimize human error and variability, offer a more reliable alternative for high-precision measurements.

In conclusion, while automated systems provide the highest consistency, carefully optimized manual methods can still yield reasonably high levels of precision. However, the role of the operator and the specific manual techniques used, such as pipetting from a tray, can significantly impact the results. These findings emphasize the need for selecting appropriate dosing techniques and considering operator skill to ensure accurate and reproducible measurements in soil texture analysis.

5 Summary

This study investigated the impact of different dosing techniques on the measurement precision of soil particle size analysis using laser diffraction. The evaluation focused on comparing the precision and repeatability of manual dosing methods, and their effects on the accuracy of soil texture classification.

The results demonstrated significant differences in measurement precision based on the manual dosing technique employed. Among the three manual dosing methods (Techniques A, B, and C), Techniques B and C achieved relatively lower relative standard deviations (SD) of 8.1% and 7.9%, respectively, indicating more consistent results. In contrast, Technique A showed higher variability, reflecting the inherent challenges associated with manual dosing processes.

Although all manual techniques offer valuable insights, Techniques B and C showed precision levels approaching those of automated systems, with relative SDs within 5% of automated benchmarks. In contrast, Technique A proved to be significantly less reliable, demonstrating excessive variability and making it less suitable for precise measurements. These findings suggest that while manual techniques B and C can achieve high levels of precision, Technique A is less effective and may be considered impractical for accurate soil particle size analysis.

The precision of particle size measurements varied notably with soil type. For instance, Technique B showed an average relative SD of 12.7% for clayey soil (Sample 3), compared to 4.9% for sandy soil (Sample 1). This finding highlights that soil texture, particularly high clay content, can significantly impact measurement variability, suggesting that soils with different textures may require tailored approaches to achieve consistent results.

The comparison between the traditional pipette method and all laser diffraction techniques revealed notable discrepancies, with the laser methods consistently showing deviations in particle size distribution. These inaccuracies were consistent across all manual dosing techniques, highlighting the inherent differences between the methods and the need for careful interpretation when comparing results from different analytical approaches.

Given that this study was conducted with only three soil samples, the findings are suggestive rather than definitive. Further research with a larger and more diverse set of soil samples is necessary to validate these results and refine dosing techniques. While manual dosing techniques can yield valuable insights into soil particle size analysis, their precision varies significantly. The study emphasizes the importance of carefully selecting dosing methods to ensure accurate and reproducible measurements.

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