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Testing the integral suspension pressure method for soil particle size analysis across a range of soil organic matter contents**

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A b s t r a c t. Particle-size distribution is a critical part of soil description, which is commonly measured using pipette and hydrometer methods. However, a recently developed technique, called the integral suspension pressure method, allows for the measurement of continuous particle-size distribution based on Stokes' law. The objective of this study was to evaluate the applicability of the integral suspension pressure method for measuring particle-size distribution, as an alternative to the standard hydrometer procedure. The integral suspension pressure method was tested by using a soil dataset with a wide range of organic matter contents (0.22-12.0%). Forty-nine samples were analysed with a hydrometer after organic matter removal and the results were compared with those obtained using the integral suspension pressure method. Through comparing the integral suspension pressure and hydrometer measurements, root mean square error values of 8.9, 8.1, and 11.9% were observed for sand, silt, and clay, respectively. The clay fraction was underestimated throughout the entire range of measurements. Conversely, the silt content was overestimated over the whole range of measurements, especially in samples with more than 36% silt. When compared to the hydrometer method, integral suspension pressure integral suspension pressure exhibited a tendency to misclassify the soil texture of clay loam samples but was accurate for sandy loams.

K e y w o r d s: integral suspension method, organic matter removal, particle-size distribution, soil texture

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INTRODUCTION

Soil texture and particle-size distribution (PSD), as well as the percentages of sand, silt, and clay are common soil properties reported in soil surveys and soil descriptions. PSD impacts several soil processes, including water movement and soil degradation, and consequently influences soil quality and productivity (Curcio *et al*., 2013). In addition, soil texture and PSD are commonly used as inputs in water flow and contaminant transport models; they are also considered when predicting soil hydraulic properties using pedotransfer functions (Contreras and Bonilla, 2018; Makovníková *et al*., 2017). Most PSD analysis methods, such as the hydrometer method (Bouyoucos, 1927) and pipette method (Robinson, 1922), are based on report specific point measurements. These two methods are based on Stokes' law, where samples are collected or measured at different sedimentation times. Because these methods depend on the soil texture classification system, which define the size limits between soil fractions (Martín *et al*., 2018), the results provide a limited number of size fractions. In addition, it is difficult and inaccurate to recalculate and convert this fraction data into another texture classification system which could easily provide PSD data compatible with any existing classification systems without the burden of extra labour (Nemes *et al*., 2002). Other methods provide a high-resolution continuous PSD curve, such as laser diffraction (Yang *et al*., 2015) that predicts grain sizes based on diffraction patterns (Konert and Vandenberghe, 1997). Although these continuous methods are more convenient for predicting soil hydraulic properties, sedimentation-based procedures are still used in soil surveys and routine analysis. One of the main reasons for this set of circumstances is the high cost associated with laser equipment and the differences in results compared to the sedimentation methods (Arriaga *et al*., 2006).

A more economical alternative for obtaining PSD curves, which is still based on sedimentation, is the integral suspension pressure (ISP) method. Unlike the hydrometer method, the ISP method computes PSD with Stokes' law continuously over time; additionally, it does not disturb the sample, unlike the pipette method. The ISP uses the temporal change in the pressure measured at a standard depth within the suspension to derive the PSD. This method integrates density throughout the suspension above the measuring depth. A mathematical model is used to calculate the pressure as a function of time; the pressure depends on the PSD as a function of the time series. The PSD of a sample is determined by adjusting the simulated time series of pressure to the measurements using inverse modelling and global optimization (Durner *et al*., 2017). Thus, the main feature of the ISP method is the generation of a quasi-continuous PSD curve, which is similar to the curves obtained through laser diffraction (Durner *et al*., 2017), but the same as the ones generated by the sedimentation and sieving-based pipette and hydrometer methods. In terms of the time taken for measurement, it takes over 6 h to obtain hydrometer readings, while the sand fraction method involves a sieving and drying procedure, which requires at least one extra day. The hydrometer method provides the three fractions (sand, silt, and clay) required to compute soil texture using a similar time window as the ISP method. The measurement-time requirements of the ISP method are similar; however, the process is semi-automated.

Removal of organic matter (OM) is recommended as a pretreatment to ensure the dispersion of soil microaggregates before PSD analysis takes place (Jensen *et al*., 2017). The OM aggregates elementary particles (Zimmermann and Horn, 2020), and not removing it, affects silt and clay determination (Jensen *et al*., 2017). Because the ISP and sedimentation methods are based on the same principle of buoyancy assumed in Stokes' law, soil particles from pretreated samples should exhibit a similar behaviour and measurement results in both techniques. Currently, the ISP method is used for reporting soil texture in soil studies related to land management and soil-water relationships (Foltran *et al*., 2021; Demand *et al*., 2019). However, to date no studies have compared the results of sedimentation analyses of pretreated soils with the ISP method. Therefore, this study aimed to evaluate the ISP method for soil particle-size measurement for several soil textures and samples with an extended OM content range. The PSD and soil textures were determined using the ISP method and compared with the hydrometer measurements which was used as a standard method, and OM was removed using H_2O_2.

MATERIALS AND METHODS

Forty-nine soil samples were collected from 37 sites in Chile. The soil samples were air-dried, manually crushed, sieved using a 2-mm mesh, and stored in polypropylene jars at room temperature until analysis. Bulk density (BD, Mg m^{-3}) was determined using the clod or core method (Zagal and Sadzawka, 2007), and OM (%) was measured using the Walkley and Black method (Walkley and Black, 1934). The water-stableilized aggregates (WSA, %) were measured using a wet sieving apparatus (Eijkelkamp Agrisearch Equipment, Giesbeek, The Netherlands) according to the methodology described by Kemper and Rosenau (1986). PSD was determined using hydrometer (Bouyoucos, 1927) and ISP methods (Durner *et al*., 2017). The ISP method was implemented using a PARIO device (Meter Group, Pullman, WA, USA). The operational ISP measurement range is 2-63 µm, where 63 µm is the upper limit for the silt fraction in the German soil classification system, KA 5. However, in this study, the USDA classification system was used for soil textural class determination, in which the upper limit for silt corresponds to 53 µm. Prior to hydrometer and ISP measurements, OM was removed using H_2O_2. Neither iron-rich nor carbonate-rich soils were found in the samples studied, so the pretreatment described below was the only one that was carried out. In a 1-L beaker, 60 mL of 15% H_2O_2 solution was added to 40 g (5%>OM content) or 50 g (5%<OM content) of sieved soil (<2 mm), the mixture was then heated to 80-90°C (Zagal and Sadzawka, 2007). Aliquots of 5 mL of 30% H_2O_2 were added until the H_2O_2 solution no longer yielded gas bubbles (Mikutta *et al*., 2005). If the OM reacted vigorously with the H_2O_2 solution at room temperature, the sample was allowed to react overnight. Two duplicated beakers were prepared for each sample. After removal, the first one was dispersed using 100 mL of sodium hexametaphosphate (50 g L^{-1}) and 100 mL of distilled water, and the soil solution was shaken overnight. The second one was oven-dried to 105°C and then weighed to determine the mass of OM loss due to its removal, and also to calculate the exact mass of the analysed solid. After that, the dispersed solution was placed in a standard 1-L sedimentation cylinder and stirred for 1 min, and then hydrometer measurements were obtained at 40 s and 7 h, while ISP techniques were applied for 12 h using a PARIO device (Durner *et al*., 2017). The parameters used for fitting the PSD in the PARIO device were as follows: the particle density was 2 650 kg m^{-3}, the dispersant concentration of 0.005 kg and the geometry of the setup was based on the default setup provided by the manufacturer. After the measurements were completed, the sand was wet sieved (>53 µm mesh), and the dry-oven sand was sieved through meshes of 2 000, 1 000, 500, 250, 105, and 53 µm for very coarse sand (VCS), coarse sand (CS), medium sand (MS), fine sand (FS), and very fine sand (VFS), respectively. The step of sand wet-sieving was repeated for each method. All of the laboratory duplicates were analysed at a frequency of one out of five samples (one per analytical batch) to verify any inconsistency and variation within a batch. The relative standard deviation (RSD) was used to determine reproducibility within each batch. The RSD was calculated as the ratio of the standard deviation to the mean for a set of values. The sand, silt, and clay fractions obtained using the ISP and hydrometer methods were compared using a paired *t*-test with a significance level of 0.05. Comparisons of the three fractions were obtained using coefficients of determination (R^2) and root mean square error (RMSE) (Mako *et al.*, 2019). The soil textures obtained using both methods were also compared by classifying them into three grouped textural classes: 1) clayey: clay and silty clay; 2) loamy: clay loam, loam, sandy clay loam, sandy loam, silt loam, and silty clay loam; and 3) sandy: sand and loamy sand (Thiam *et al.*, 2019). The *kappa* coefficient, accuracy, and sensitivity were determined based on a confusion matrix, a cross-tabulation comparison of soil textural classes (Salley *et al.*, 2018) determined using ISP measurements and a hydrometer. The *kappa* coefficients were categorized as proposed by Poppiel *et al.* (2019), where <0.00, 0.01-0.20, 0.21-0.40, 0.41-0.60, 0.61-0.80, and 0.81-1.00 indicated very bad, bad, fair, good, very good and excellent measurements of performance, respectively. According to the hydrometer measurements, the overall accuracy was defined as the total number of ISP soil texture classes that matched the hydrometer soil texture classes. Simultaneously, the sensitivity value was calculated using the number of ISP measurements that matched a specific soil texture class as compared to the hydrometer results (Salley *et al.*, 2018). The USDA-NCSS texture triangle representing the 12 textural classes was plotted using the ggplot2 and ggtern R packages (Hamilton and Ferry, 2018).

RESULTS AND DISCUSSION

Table 1 summarizes the main properties of the soil samples, including OM, BD, textural class (according to the hydrometer method), and WSA. The dataset used in this study includes basic soil properties (BD, pH, EC, and WSA) and soil properties related to the particle-size distribution over a broad range OM, which was critical for evaluating the methods used to determine PSD. The average content of OM for all of the samples was 4.31%, with minimum and maximum values of 0.22 and 12%, respectively. High OM content values (>10%) have been reported in soils from the same study area which also represent more than 50% of the arable land in Chile (Ellies *et al.*, 2005; Contreras and Bonilla, 2018). The BD in the topsoil samples ranged from 0.72 to 1.67 Mg m^{-3}, with an average value of 1.23 Mg m^{-3}. Lower BD values were observed for wildland soils, which correlated to a slight extent with the soil OM content.

Table 1. Summary of critical soil properties and land use of soils analysed in this study, based on the hydrometer method (n=49). Values are expressed in the form of an average ± standard deviation and range (minimum-maximum)

Class	Number of samples	Organic matter content (%)	Bulk density (Mg m^{-3})	Water stable aggregates (%)
C	4	3.60±2.61 (1.5-7.3)	1.18±0.25 (0.89-1.36)	72.6±14.2 (56.6-89.0)
CL	12	2.91±1.78 (0.7-7.1)	1.34±0.24 (0.88-1.56)	72.6±14.8 (43.1-94.7)
L	11	5.95±1.93 (3.1-9.1)	1.18±0.21 (0.86-1.60)	73.9±14.9 (51.4-97.1)
LS	3	2.13±1.88 (0.22-4.2)	1.44±0.05 (1.14-1.48)	56.7±38.4 (15.6-97.9)
SCL	3	2.73±1.25 (1.5-4.0)	1.29±0.07 (1.24-1.34)	55.8±11.2 (44.0-66.2)
SL	15	5.23±3.61 (1.2-12.0)	1.21±0.35 (0.72-1.67)	71.5±24.9 (0.00-94.1)
SIC	1	2.91 (-)	0.95 (-)	67.2 (-)

C – clay, CL – clay loam, L – loam, LS – loamy sand, SCL – sandy clay loam, SL – sandy loam, SIC – silty clay,

Because clay particles and OM are the main binding agents forming organo- mineral assemblages (Rivera and Bonilla, 2020), higher WSA values (over 95%) were observed for the clayey textural classes and also higher OM contents.

A comparison between the ISP and hydrometer measurements is shown in Fig. 1. As explained in the Materials and Methods section, OM was removed prior to analysis for both methods, which were performed separately. The average RSD of the ISP duplicates was 9.9%, a slightly higher value than the 8% reported for the hydrometer method (Gavlack *et al.*, 2005). Figure 1 compares the sand content measurements, where the R^2 value between the ISP and hydrometer results was 0.88, with an RMSE of 8.9%. Although the sand contents were not measured directly with the ISP device, they were necessary for PSD analysis and calculation when using the ISP method. In this method, the cumulative PSD function is determined using nonlinear regression in order to achieve the best fit between the model and measurements using the ISP method and independent sand fraction values obtained by sieving (Durner *et al.*, 2017). Durner *et al.* (2020) noted that measuring sand fractions independently could affect clay fraction determination. Durner *et al.* (2021) reported that this error significantly affects the results produced by samples with low clay contents, especially in sandy soils. Thus, although no statistically significant difference (p>0.05) was found between the sand contents determined using the hydrometer and ISP methods, an RMSE value of 8.9% may still be substantial, even if no statistically significant difference was found low variation in results, specifically at low clay contents. No statistically significant difference (p>0.05)

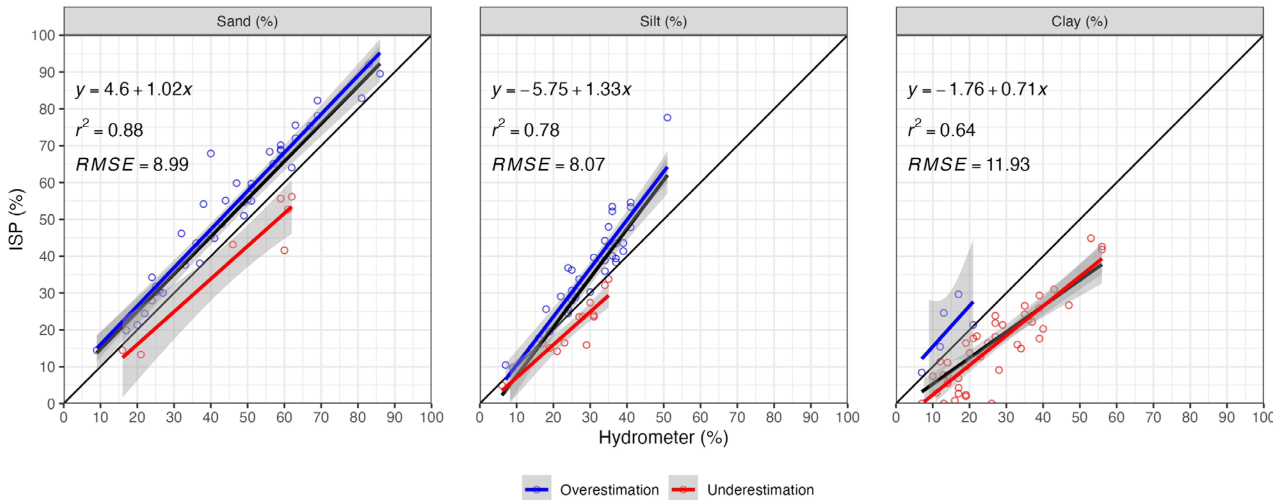

Fig. 1. Comparison between sand, silt, and clay contents measured using the ISP and hydrometer methods. The black line represents the linear regression expressed in each faceted plot for all data. The blue and red lines represent the linear regression expressed in each faceted plot for overestimated and underestimated data respectively.

was observed between the sand contents determined using the hydrometer and ISP methods as the same procedure (wet sieving) was used for both.

As shown in Fig. 1, the ISP silt values were not significantly different from those obtained using hydrometer measurements ($p>0.05$), and the silt fraction measurements of both methods were in agreement ($R^2=0.78$, RMSE = 8.1%). However, for all of the samples where the hydrometer measured a silt content of over 36% (n = 12), the ISP method exhibited an overestimation of this fraction. When compared to the hydrometer method, the ISP method also showed a statistically significant underestimation of the clay content ($p<0.05$), with 90% of the measured values occurring below the 1:1 line. The RMSE value for the silt content which may be used to compare the hydrometer and ISP methods (8.1%) was higher than those reported to compare the hydrometer and pipette methods (6%) (Faé *et al*., 2019). A high degree of uncertainty was observed in the measured clay contents when using the ISP method. A similar condition was reported by Nemes *et al*. (2020) when comparing the ISP and pipette methods. They found a high degree of uncertainty in the ISP clay measurement at low clay contents. An overestimation of silt and an underestimation of clay may be expected to occur if OM has not been removed previously as it causes an incomplete dispersion of soil aggregates <20 µm (Jensen *et al*., 2017). However, in both procedures, the OM was removed prior to analysis, hence, the differences in silt and clay content may be attributed to the ISP measurements. The USDA range for the silt size limit is 2-50 µm, which is within the measurement range for the ISP device (2-63 µm). Thus, the observed bias of the clay fraction may be understood to be primarily due to the silt measurements. The silt and clay fractions measured using ISP showed a higher RMSE than the comparison between the pipette and hydrometer methods (3-6% respectively) but a lower RMSE than that produced using laser diffraction and hydrometer measurements (18-22% respectively) (Faé *et al*., 2019). The sand content used as an input when fitting the PSD measurements for ISP, could be a source of error when measuring the clay fraction (Durner *et al*., 2020). Indeed, the error in the sand fractions determined by sieving will propagate linearly sfor the finer fractions in the ISP measurements (Durner *et al*., 2021). However, this source of error was dismissed in this dataset as no statistical differences in sand measurements were found between the ISP and hydrometer sieved fractions. The immersion of the device and the covered sampling area could be another potential source of differences in the results produced by the two measurement procedures. Disturbances in the soil columns could be more severe with either method, altering the dispersion (*e.g.*, cylinder wall flow after suspension mixing) due to device immersion to a different extent for each method (Durner *et al*., 2017). The use of different sampling zones in previous studies have been used to explain the differences between sedimentation procedures, such as hydrometer and pipette measurement (Durner *et al*., 2017). For example, a pipette provides a slightly finer distribution than a hydrometer (Coates and Hulse, 1985). Also, the discrepancies in silt content results between the ISP and hydrometer could also be explained by the errors resulting from subsampling due to the increasing prevalence of coarser particles (Ramsey and Suggs, 2001), and potential changes in temperature while performing different sets of measurements (Durner *et al*., 2021).

Differences in measurement results between the hydrometer and ISP methods were grouped into clayey, loamy and sandy classes (Fig. 2a). Sand content results did not produce any statistical differences (NS) for any textural group. Silt content results exhibited statistical differences between clayey and loamy ($p<0.001$) and clayey and sandy samples ($p<0.001$), with no statistically significant differences

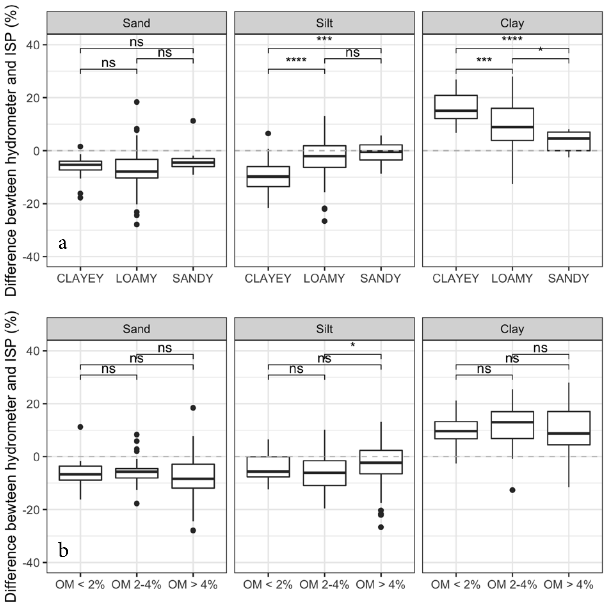

Fig. 2. Sand, silt, and clay fraction differences between the ISP and the hydrometer methods. The effects are presented for: a) various soil textural groups (clayey, loamy, and sandy) and b) OM ranges (<2, 2-4, and >4%). Data above the horizontal line represents an underestimation in the ISP measurements (as compared to the hydrometer measurements). Data below the line represents an overestimation. * p<0.05, ** p<0.01, *** p<0.001, ns – not significant.

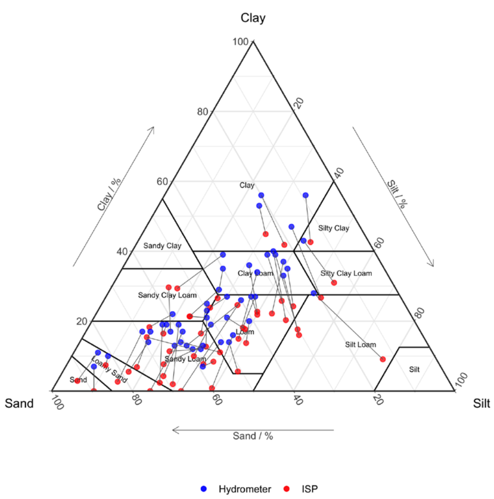

Fig. 3. USDA soil textural classes based on sand, silt, and clay contents measured using the ISP and hydrometer methods. Red and blue dots correspond to ISP and hydrometer measurements, respectively; the same samples are connected with a gray line. Red half-circles on the sand axis indicate measurements with a clay content <1%.

between the sandy and loamy groups (NS). Clay content results exhibited statistical differences among all of the textural groups. The statistical differences were observed for clayey and loamy (p<0.001), clayey and sandy (p<0.001), and loamy and sandy (p<0.05) samples. The overestimation of silt and underestimation of clay were larger in the clayey group than in the sandy and loamy soils. Differences between the results obtaithe ned using different methodologies, appear to be caused to a greater extent by differences in sample pretreatments than by the PSD analysis itself (Durner *et al*., 2021), a grouped analysis using OM content range was performed in order to verify any measurement bias involving OM removal. When grouping the soil samples by OM content range (<2, 2-4, and >4%), the differences between the fractions (silt and clay) measured using the hydrometer and ISP methods were not statistically different (Fig. 2b). The difference in ISP measurements between the silt and clay fractions relates to the silt content and its overestimation, but not to the OM content. Figure 3 shows the soil textures identified using the ISP and hydrometer methods. An underestimation was observed for the clay in the ISP method as the measurements progressed toward higher silt values. Figure 3 also indicates that the ISP device could not detect clay (<1% measured) in the three loamy soils. Due to the fact that OM removal requires a decanting step, there is a chance that a small portion of clay and fine particles may have been removed in this process (Fisher *et al*., 2017). This possible source of errors in the ISP method should be considered for improvement in future studies due to the low degree of precision found at low clay values. The accuracy of the ISP method in determining soil texture classes compared with the hydrometer method is shown in Fig. 4. The results produced by the ISP method matched those produced by the hydrometer method in 17 of the 49 samples analysed. The best performance was observed for the SL (sandy loam) class, where the ISP measurement results matched for 10 out of 15 samples that were analysed. By contrast, the worst performance was observed for the CL (clay loam) class, where none of the 12 samples classified as CL using the hydrometer method matched the classification obtained using the ISP method. The confusion matrix exhibited an

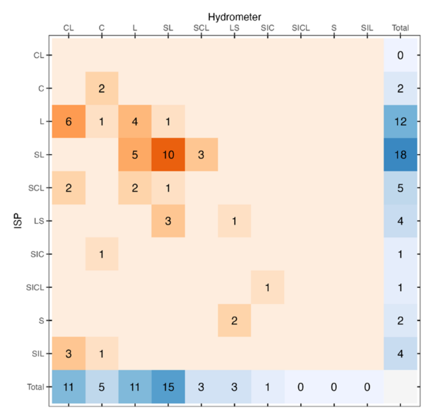

Fig. 4. Confusion matrix for accuracy in matching the textural class group using the ISP and hydrometer methods. SCL, S, and SIL classes were not measured using a hydrometer but rather they were classified using the ISP method. The intensity of colour in the red tiles is based on the number of matches, for the blue tiles it is based on the number of observations for each method. C – clay, SIC – silty clay, CL – clay loam, SICL – silty clay loam, SCL – sandy clay loam, L – loam, SIL – silt loam, SL – sandy loam, LS – loamy sand, S – sand.

overall accuracy of 0.35, regardless of the *kappa* value (=0.21), which was adequate according to Poppiel *et al.* (2019). The highest number of samples measured using a hydrometer (n=12, 11, and 15, respectively) belonged to the CL, L (loam), and SL classes, with sensitivities of 0.00, 0.34, and 0.67, respectively. The lack of agreement may be attributed to the combined effect of the underestimation of clay and the overestimation of silt, which shifts the texture classification in 65% of observations. Also, although no statistical differences in sand measurements were found between the ISP and hydrometer sieved fractions, the different measuring techniques can still affect the soil texture classification, as observed with classification changes from CL to SL. From the 32 texture classification differences between the hydrometer and the ISP method, 31 occurred between the adjacent classes, except for one soil sample that moved from CL to SL. A combined approach of the qualitative evaluation of soil textural classes and quantitative evaluation for the determination of the percent or mass of each fraction, must be followed in order to compare the hydrometer and ISP methods. The discrepancies between the sand, silt and clay values between the ISP and hydrometer techniques (RMSE 8.9, 8.1 and 11.9%, respectively) are translated into significant discrepancies in the assigned soil textural classes (Yang *et al*., 2015). The ability to determine the PSD value in a precise curve represents the advantage of the ISP method over the hydrometer method using the same principle. Finally, soil textural classes may be determined with a fair degree of accuracy (Poppiel *et al*., 2019) using the ISP method in contrast to the hydrometer method.

CONCLUSIONS

1. A dataset of soil samples comprising a wide range (0.22-12%) of organic matter content was analysed using integral suspension pressure and hydrometer methods.

2. The performances of the integral suspension pressure and hydrometer methods were compared and root mean square error of 8.9, 8.1, and 11.9% were observed for sand, silt, and clay, respectively. According to previous studies, root mean square error for silt values are higher in comparison to the pipette method but lower than that produced by laser diffraction.

3. The underestimation of clay content and the overestimation of silt content were observed when comparing the results obtained from the sand, silt, and clay fractions that were measured using the integral suspension pressure and hydrometer methods.

4. The underestimation of clay content translated into a misclassification of most textural classes, while the sandy loam and loam classes were determined with a fair degree of accuracy using the integral suspension pressure method.

5. Plans for future work includes a) a comparison between the integral suspension pressure method using a pipette and laser measurements and b) methodological improvements including sand separation before analysis and clay determination in a suspension.

Conflict of interest: The authors declare no conflict of interest.

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