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Variability and compatibility in determining soil particle size distribution by sieving, sedimentation and laser diffraction methods

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ABSTRACT

A range of methods and applications are in use to determine soil particle size distribution. Due to the differences in measurement technology, the analytical results may deviate more or less from each other, which has implications for the matching with historical soil databases. There is a need for studies to critically evaluate their results, both concerning subsample variabilities and compatibilities. In the present study the more recent integral suspension pressure (ISP) and laser diffraction (LDM) methods were compared with the reference sieve and pipette (SPM) method. Samples from topsoil and subsoil of four agricultural soils with sandy clay loam to clay textures were analyzed. A protocol, comparing alternative pre-sievings at the meshes 0.063 (ps0.063), 0.2 (ps0.2) and 2.0 mm (ps2) for the sedimentation (SPM, ISP) and laser diffraction (LDM) measurements, was used. Here we report, based on particle size fraction contents for clay (<0.002 mm), silt (0.002–0.063 mm) and sand (0.063–2.0 mm), i) apparent deviations between pre-sieving options for each method, ii) variabilities between sample replicates (three subsamples), and iii) relationships (linear regression) and iv) texture class differences between SPM, ISP and LDM analyses. Overall, SPM showed smallest deviations between pre-sieving options, LDM largest, and ISP intermediate. Higher silt content, for ISP, and higher sand content, for LDM, seemed to be critical in the choice of optimum pre-sieving. Regarding variabilities between replicates, SPM showed smallest variabilities, ISP (especially ISP-ps0.2 and ISP-ps2) and LDM-ps2 largest, and LDM-ps0.063 and LDM-ps0.2 intermediate. SPM-ps0.063, SPM-ps2, ISP-ps2 and ISP-ps0.2 showed strongest relationships (i.e. largest R^2) with the reference SPM-ps0.2, LDM-ps0.063 intermediate and LDM-ps2 weakest. Regarding texture classification, compared to the reference SPM-ps0.2, SPM-ps2 and ISP-ps2 showed largest (good, i.e. 80–100% of the cases) agreement, whereas LDM pre-sievings showed smallest (LDM-ps0.063, poor agreement, i.e. <55%). Linear-transfer transformed LDMt-ps0.063 improved the texture compatibility with SPM-ps0.2 to intermediate (63%) agreement, and SPMt-ps0.063 and ISPt-ps0.2 from intermediate (75%) to good (88%) agreement. Also clay-silt cutoff modified LDMc-ps0.063 and LDMc-ps0.2 improved the texture compatibility with SPM-ps0.2, to intermediate (63%) agreement. There is a need to continue fine-tuning methodologies to align particle size distribution composition from one method to the other, especially regarding the influence of equivalent and efficient particle shape and pre-treatment procedures on the results.

1. Introduction

Determining soil particle size distribution is a classic challenge in soil science, but yet a critical component for the determination and explanation of soil systems and functions. The common practices for determining soil particle size distribution balance between the needs for representativeness (as in sampling and replicability in analysis), compatibility (as in particle size classification scheme) and effort (as in labor time consumed for analysis).

The combination of direct determination of masses from sieving and gravitational sedimentation, like the sieve and pipette method (SPM) in the present study, has been in use for a long time as international reference method to estimate particle size distribution in soils (e.g. ISO 11277, 2009). The SPM procedure is labor intensive, not the least concerning the manual extraction of soil fractions by pipetting at given depth and time intervals during the sedimentation process of the clay

Abbreviations: ISP, integral suspension pressure method; LDM, laser diffraction method; SPM, sieve and pipette method; ps0.063, pre-sieving 0.063 mm; ps0.2, pre-sieving 0.2 mm; ps2, pre-sieving 2.0 mm; KUN, soil site Kungsängen; NAN, soil site Nåntuna; SAB, soil site Säby; ULT, soil site Ultuna.

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and silt fractions, and their subsequent drying and weighing. The more recent methods of integral suspension pressure (ISP) (e.g. Durner et al., 2017a) and laser diffraction (LDM) (e.g. Faé et al., 2019) are promising alternatives for soil particle size and distribution determination. ISP, like SPM, rely on sieving to obtain the sand fractions. ISP and LDM have the advantages that the extraction-drying-weighing procedure for the finer soil fractions (clay and silt) is replaced by automatic registration of, for ISP, pressures at given depth during the sedimentation process and, for LDM, particle diameter distribution in a pipetted sub-sample. Furthermore, ISP and LDM provide particle size distributions with a finer resolution compared to the coarse size increments obtained with SPM. Due to these differences in measurement technologies, particle size distributions determined with ISP and LDM often deviate more or less from those of SPM (e.g. Durner and Iden, 2021; Yang et al., 2019). This has implications for the matching with historical SPM derived soil texture data, which currently dominates soil data bases locally and globally, and could potentially result in reclassification of soils (e.g. Nimblad Svensson et al., 2022).

In terms of ISP comparisons with SPM, there is a need for additional comparative measurements, representing a variety of soil types and texture ranges, in order to explain part of the uncertainty and variability in soil particle size distribution (Nemes et al., 2020). Durner and Iden (2021) presented a further developed ISP method, called ISP+ , which improved the accuracy and precision considerably. Also in terms of LDM, the optimal conversion of LDM measurements to correlate well with SPM measurements needs further research (Nemes et al., 2020). LDM measured soil fraction contents may serve as base for soil textural–class determination compatible with SPM, if they are converted with linear regression relationships (linear-transfer functions) into transformed soil fraction contents (Taubner et al., 2009; Makó et al., 2017). Wet-sieving and weighing the sand fraction, and modification of the clay-silt cutoff from 0.002 mm to approximately 0.006 mm, resulted in LDM values that differed only about as much from SPM as the hydrometer method (Faé et al., 2019). The selection of an appropriate conversion system, however, is complex. For the clay-size fraction, for example, both similarities and dissimilarities between soil samples from different regions and types of sediment can be found (Buurman et al., 2001). Furthermore, the procedures applied in the sample preparations and actual measurements need to be taken into account (Taubner et al., 2009; Nimblad Svensson et al., 2022).

Both sedimentation of particles using Stokes ́law, as applied in SPM and ISP, and the conversion of diffraction angles into particle sizes, applied in LDM, are based on the concept of equivalent spherical particles. The fact that soil particles generally show various shapes, from nearly spherical to platy, will in itself influence the resulting equivalent particle size distributions differently for each of the analysis methods. For SPM and ISP, platy or other irregular particles reach lower settling velocities than spheres of comparable volume leading to that the particle diameter may be underestimated (Syvitski, 1991). For LDM, a mean particle diameter is averaged out of different axes of view, so that for example a platy particle is described by a cross-sectional area being larger than that of a sphere of equal volume (Jonasz, 1987). Thus, the effect of shape is working in opposite direction in LDM as compared with SPM and ISP (Taubner et al., 2009).

The proportions of sand fractions (in the present study defined in the range 0.063–2 mm) are for SPM and ISP generally analyzed by aid of dry- and wet-sieving, and combined with a fitting procedure for estimating the entire particle size distribution. It is cumbersome to pass fine earth material through sieves with openings smaller than 0.100 mm (Becher, 2011). Therefore particles with diameters smaller than 0.063 mm, representing the silt and clay fractions, are generally classified by sedimentation (Gee and Or, 2002). Laser diffraction analyses started out with measurement of the entire fine-earth fraction (<2 mm) without sieving for sand fractions. However, it has been difficult to get reliable particle size distribution results due to the difficulties to get the sand homogeneously distributed during the measuring procedures (e.g. Faé et al., 2019). LDM has often returned substantially different data as compared with SPM, due to differences in its core theory, sample pre-treatment process, as well as the fact that sand content has been measured together with clay and silt contents in the laser diffraction analyses (Nemes et al., 2020). Sieving of the sand fractions combined with laser diffraction analyses of the silt and clay fractions have been shown to result in better agreement between the two methods (Taubner et al., 2009; Faé et al., 2019).

Whereas the sieving analyses are often made for the default sand subfractions (e.g. 0.063–0.2, 0.2–0.6, 0.6–2 mm), the fractions that are brought to the sedimentation analyses have generally been pre-sieved to, for example, fractions < 0.063 (e.g. ISO 11277, 2009), < 0.2 (e.g. for SPM by our laboratory and for Centeri et al., 2015) or < 2 mm (e.g. for ISP by Meter Group, 2018–, 2019). Using pre-sieved < 0.063 mm material put high quality requirements on proper sieving procedures. If pre-sieved < 0.2 or < 2 mm material is used, however, the sand-silt limit (0.063 mm) is obtained from the sedimentation and laser diffraction measurements. This limit can be double-checked if, in parallel, the sand-silt limit is also sieved.

As outlined above, there has been several studies comparing, on one hand, ISP with SPM and, on the other, LDM with SPM. However, there are no or only very few studies comparing the three methods simultaneously on the same soil samples. Consequently, there is a need to compare particle size distributions obtained from the three different methods (SPM, ISP, LDM) using the same soil samples. Furthermore, there is a need to further investigate boundaries between the practice of sieving for the coarser particles, and sedimentation (SPM, ISP) or laser diffraction (LDM) measurements for the finer particles, i.e. optimum pre-sieving, for each of the three methods. The objective of the present study was to compare alternative protocol procedures for pre-sieving to determine soil particle size distribution with SPM, ISP and LDM methods for soils with a range of clay contents (25–55%). The comparisons were based on soil samples from four agricultural field sites at two depths (topsoil, subsoil), applying three alternative pre-sievings (0.063, 0.2 and 2.0 mm) in the sedimentation (SPM, ISP) and laser diffraction (LDM) analyses. Apparent deviations between pre-sievings of each method as well as variability between replicate subsamples were investigated. Alternative ways of processing the outcomes from the measurements, by aid of linear-transfer transformations and clay-silt cutoff modifications, were performed to find improved relationships and compatibility with our reference method, i.e. with SPM using pre-sieving option 0.2 mm.

2. Materials and methods

2.1. Sites and samples

The study was performed using soil samples from four agricultural field sites, in the nemoral environmental zone in Europe (Metzger et al., 2005), that had been under continuous crop cultivation for more than 50 years. They are all formed from postglacial sedimentary parent material, having clay contents higher than 25%, being classified into clay and silty and sandy clay loam textures and characterized as clay soils with varying degrees of silt, sand and gyttja contents (Table 1). The sites were spaced 1.5 to 3.5 km from each other around Uppsala city in Sweden. At each site, approximately 2 kg (after air drying) soil was sampled from an area of about 0.2 m^2 at each of two depths, topsoil 10–20 cm and subsoil 40–50 cm. All samples were taken within one week in late summer 2020 and analyzed in laboratory during the consecutive autumn and winter.

2.2. Laboratory procedures and analyses

Particle size distributions were determined with the methods of sieve and pipette (SPM) (sedimentation run in the laboratory standard 20 cylinder set-up), integral suspension pressure (ISP) (sedimentation process measured with Pario, Meter Group (2018–2019), using six devices) and laser diffraction (LDM) (measured in a Horiba Partica La-950

Table 1
Characteristics of the four sites.

	Position in decimal degrees	Land form	Texture and organic matter content (OM)[a])		Characteristic based on Ekström (1927) classes
			topsoil	subsoil	
Nåntuna (NAN)	59.81202 N, 17.67982 E	Crest of a slightly undulating valley bottom	Sandy clay loam OM 3.4%	Clay loam OM 1.3%	Sandy intermediate clay
Säby (SAB)	59.83205 N, 17.70548 E	Intermediate part of a large plain (level land)	Silt loam OM 3.6%	Silty clay loam OM 2.3%	Silty intemediate clay
Ultuna (ULT)	59.81275 N, 17.65185 E	Central part of a small plain (level land)	Silty clay loam OM 1.8%	Clay OM 0.4%	Heavy clay
Kungsängen (KUN)	59.83784 N, 17.66710 E	Lower part of a large plain (level land)	Silty clay OM 4.3%	Silty clay with gyttja OM 2.6%	Heavy clay with gyttja

[a]) Texture classes according to WRB (FAO, 2015) soil fraction limits, i.e. clay to 0.002 mm, silt to 0.063 mm and sand to 2.0 mm, and organic matter content calculated from loss on ignition (see also Table 4, SPM-ps0.2, for more detailed soil fraction size distribution).

Table 2
Laboratory pre-treatments.

	Sieve and pipette (SPM)	Integral suspension pressure (ISP)	Laser diffraction (LDM)
Fine earth (<2 mm) used for each replicate sample and pre-sieving (sub-sample)	20 g	30 g (in parallell 50 g for determination of organic matter plus air-dried water contents)	10 g
Pre-treatment to remove organic matter	45 ml deionized water[a]) and 10 ml hydrogen peroxide (H_2O_2) (35%)[b])	Like SPM	Like SPM, but 20 ml deionized water and 5 ml of $H_2O_2^{b}$)
Chemical dispersion	25 ml dispersant - metaphosphate $(NaO_3P)_n$ (33 g l^{-1}) and sodium carbonate Na_2CO_3 (7 g l^{-1})	Like SPM	Like SPM, but 12.5 ml
Physical dispersion	Mechanical rotator overnight	Like SPM	Reciprocating shaker overnight (200 strokes min^{-1})

[a]) Two drops of 1 M HCl was initially added to check carbonate content, but no effervescence was noted.
[b]) More water added if needed to replace evaporated water during boiling procedure.

v2, Horiba, Ltd., maximum 24 samples at each set-up). Preparation of the soil samples, i.e. air-drying, grinding and sieving to fine-earth fraction < 2 mm, was common to the three methods. Only the prepared fine-earth fraction was analyzed in the present study. In addition to the fine-earth fraction < 2 mm (here called ps2), two further pre-sievings were applied to obtain the sub-samples used for sedimentation (SPM, ISP) and laser (LDM) measurements, i.e. 0.063 mm (called ps0.063) and 0.2 mm (called ps0.2). Preparation of the fine-earth samples, all SPM and ISP analyses and LDM analyses for ps2 were performed by one operator, and LDM analyses for ps0.063 and ps0.2 by another operator. The reference method in the present study is SPM in general, and SPM with 0.2 mm pre-sieving (called SPM-ps0.2 and referred to as 'reference method') in particular, being the protocol used in our laboratory for the past 20 years. It should be acknowledged that SPM does not necessarily represent the 'true' particle size distribution (Nemes et al., 2020), but is referred to as it is also a general international reference method.

The procedures of the pre-treatments for each of the three methods are presented in Table 2, and the procedures in the subsequent measurements and processing were performed as in Table 3.

2.3. Processing and evaluation

In the present study, thus, the clay and silt fractions were for all three methods (i.e. SPM, ISP and LDM) determined from each of the pre-sieved < 0.063, < 0.2 and < 2 mm fractions, three subsamples from each sample (referred to as replicates in the following). The sand fractions were determined as presented in Table 3. The estimation of the cumulative fraction of the laser diffraction determined sand-silt fraction cutoff at 0.063 mm (i.e. for LDM-ps0.2 and ps2) was based on the assumption of a log-linear relationship between the two neighboring particle size limits with measured fractions (Nemes et al., 1999), i.e. 0.0590 and 0.0675 mm.

In the evaluation of LDM, an extra test was performed in line with Faé et al. (2019), i.e. modifying the clay-silt cutoff from the standard 0.0020 mm. In the present study the modified cutoff was set at 0.0039 mm, adapted from an optimum value reported for a large soil data base (n = 44) in Nimblad Svensson et al. (2022). In the following, the LDM cases with standard 0.0020 mm clay-silt cutoff will be called LDM and those with modified 0.0039 mm will be referred to as LDMc.

The results are presented and evaluated based on four aspects, i.e. i) apparent deviations between pre-sieving options for each of the methods (visually observed in the graphs), ii) sample replicate variabilities (three subsamples from each sample), iii) linear regression relationships versus the reference SPM-ps0.2 for the other method-pre-sieving combinations (i.e. SPM-ps0.063, SPM-ps2, ISP (all pre-sievings) and LDM (all pre-sievings), respectively), and iv) texture classification versus the reference SPM-ps0.2 for the other method-pre-sieving combinations. Regarding aspect ii, the range (maximum minus minimum value) was chosen as a convenient measure of variability (Dixon, 1986), due to that only three replicate samples were measured for each combination of method-soil-depth-pre-sieving. The compatibility between each method-pre-sieving combination, respectively, and the reference method SPM-ps0.2, was evaluated by aid of the linear regression slopes in aspect iii. Linear regression slopes with intercept set to zero were utilized in the evaluations, in order to avoid negative values and also being relevant from a physical point of view. Transformed values were estimated by dividing the measured SPM, ISP and LDM with the slope of respective linear regression relationship with the reference SPM-ps0.2. These transformed values will be referred to as SPMt, ISPt and LDMt. They were estimated for each of the main fraction classes (clay, silt, sand). If needed, the fractions were proportionally adjusted to make their sum 100. Resulting soil textural classes for the three methods and pre-sievings were compared, in order to indicate any compatibilities between them and the reference SPM-ps0.2. The classification was based on WRB (0.002–0.063-2.0 mm) particle-size fractions (FAO, 2015) and estimated for both measured and transformed fraction values.

3. Results

3.1. Pre-sieving deviations

Regarding apparent deviations between pre-sieving options for each of the methods SPM, ISP and LDM (visually observed in Figs. 1 and 2), the SPM cases showed almost no such deviations. Only minor ones were

Table 3
Laboratory measurements and processing.

	Sieve and pipette (SPM)	Integral suspension pressure (ISP)	Laser diffraction (LDM)
Type of measurement	Sedimentation/ pipetting (based on Stokes´ law, assuming spherical particles and particle density 2.65 kg l^{-1}).	Sedimentation/ pressure measurement.	Laser diffraction in particle size analyser device.
Number of samples and devices	Initially, altogether up to 15 samples were run at each measurement set-up.	Altogether up to six Pario pressure sensor devices were run simultaneously at each measurement set-up.	Altogether up to 16 samples were run at each measurement set-up.
Sedimentation and laser diffraction measurements - time aspects (on the <0.063, <0.2 and <2 mm pre-sieved fractions, respectively)	Pipetting at the time steps 56 s at 20 cm depth, 4 min 38 s at 10 cm, 51 min 29 s at 10 cm, 5 h 48 min at 7.5 cm (for <0.063, < 0.020, < 0.006 and < 0.002 mm, respectively). Oven drying at 105 °C overnight.	Performed 10 h during night, with the exception of two cases (out of the totally 72 cases) which were run for 8 h.	Approximately 5 min per sample.
Sedimentation and laser diffraction measurements - procedure (after shaking)	The pipette was lowered from above into the sediment cylinder at each time step to sample the aliquot.	The pressure sensor device, after being stored in water in a cylinder adjacent to the measurement cylinders to minimize temperature differences, was in the start of the run lowered from above into the measurement cylinder (opting for maximum 20 s after start of sedimentation) and then kept there during the recording procedure.	An aliquot was withdrawn with a pipette (while the sample was being stirred), and enough sample was added to reach a transmittance of 80 ± 0.5% (red laser). The measuring sequence used a pump speed of setting 7 (1633 rpm), and agitator setting 5 (2000 rpm). Each measurement run was saved in 93 size increments (bins).
Sand fractions measurements	Determination after the pipette run, by mixing the fractions solution from the pipette run (<0.063, <0.2 and <2 mm, respectively) with the remaining pre-sieved fractions (0.063-2 and 0.2-2 mm, respectively) and sieving this mixture to get the sand fractions 0.063-0.2 and 0.2-0.6 (wet-sieving) and 0.6-2 mm (dry-sieving). Oven drying at 105 °C overnight.	Determination after the pressure run like SPM.	Determination of LDM-ps0.063 from the pre-sieved 0.063-2 mm fraction, whereas for LDM-ps0.2, the laser measurements defined the 0.063-0.2 fraction and the pre-sieving the 0.2-2 mm fraction, and for ps2 all the range to 2 mm was determined by the laser measurements. Oven drying at 105 °C overnight.
Input data for calculations	Mass values from the aliquots with different fraction classes (from the pipette and wet- and dry-sievings), masses of the soil after drying to 105 and 550 °C (for estimation of loss on ignition), and a correction factor for the mass of the dispersant agent.	Particle density 2.65 kg l^{-1}, net mass of particles (g) (from gross mass of air-dried soil minus organic matter content and water content in air-dried soil), mass of dispersant 1 g l^{-1}, and sand fraction (0.063-0.2, 0.2-0.6 and 0.6-2 mm) percentages from the wet- and dry-sievings.	Setting of scattering parameters refractive index (RI) to 1.52 and absorption coefficient (AC) to 0.1 (proposed by Bieganowski et al. (2018) as an international standard to improve comparability between laboratories).
Estimation of particle size distribution	Determined from the oven-dried clay, silt and sand aliquots masses, adjusted for the mass of dispersant in the pipette sample volume and for organic matter content (calculated from loss on ignition minus a correction factor related to clay content).	Determined from the integral calculations of the pressure changes in the clay and silt fractions combined with weighed masses for the sand fractions according to the concepts in Durner et al. (2017a) and Pario manual (Meter Group, 2018-, 2019).	The volumetric clay and silt fractions were recalculated to mass units, as proposed by Faé et al. (2019), based on assumptions of equal particle density and spherical particles (as for the sedimentation aspects of SPM and ISP), combined with weighed masses for the sand fractions range.

observed for ps0.063 for SAB-silt topsoil (5–7% points smaller than ps0.2 and ps2) (Fig. 1b). For ISP, large deviations were observed for ps0.063 for SAB-clay (9–29% points larger than ps0.2 and ps2) and silt (9–28% points smaller than ps0.2 and ps2) (Fig. 1d and e). This was possibly due to the relatively higher silt/clay ratios for SAB compared

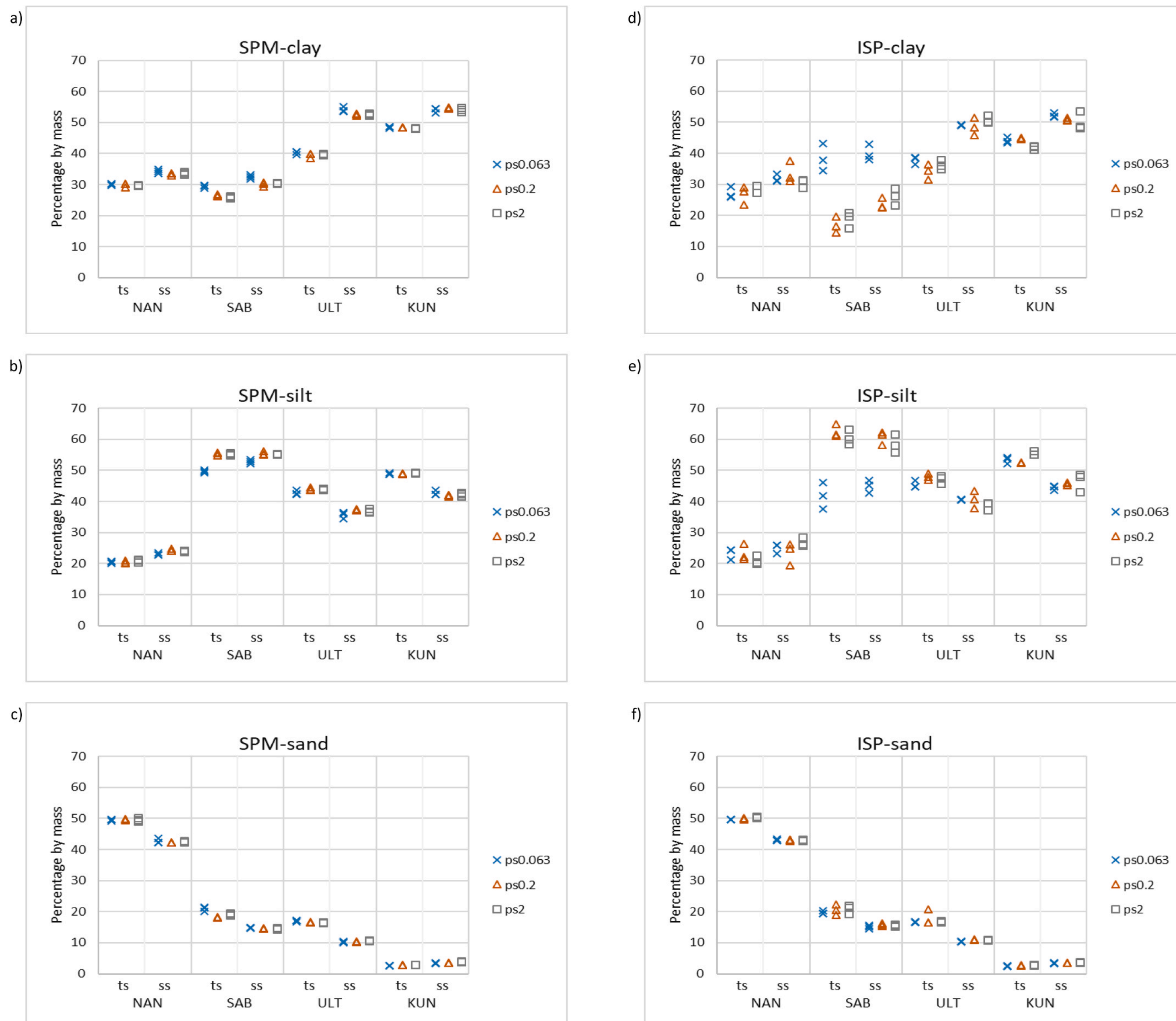

Fig. 1. Presentation of all replicate mass fraction percentages (n = 3) in topsoil (ts) and subsoil (ss) of each combination of soil and pre-sieving for each main particle size fraction (clay= a, d; silt= b, e; sand= c, f), for the methods of sieve and pipette (SPM= a, b, c) and integral suspension pressure (ISP= d, e, f).

with the other soils (see mean values of clay, silt and sand contents for the reference SPM–ps0.2 in Table 4).

For LDM and LDMc, large deviations were observed for ps2 for NAN-silt (from slightly smaller to 21–31% points larger than ps0.063 and ps0.2) and sand (from slightly larger to 36% points smaller than ps0.063 and ps0.2), and for some of the clay replicates (up to 22% points larger than ps0.063 and ps0.2) (Fig. 2). This is in line with the known issues of analyzing sand with laser diffraction methodology (Faé et al., 2019). For NAN-silt and sand as well as ULT-silt and sand, furthermore, ps0.063 and ps0.2 differed consistently between each other, but to a much smaller extent. Also SAB-sand showed some smaller deviations for ps0.2 and ps2 in the topsoil samples.

Of the soils, and for all three methods, KUN showed the smallest number of apparent deviations between pre-sievings (Figs. 1 and 2). This may implicate that the large clay content, and thereby relatively smaller silt and sand contents, combined with the gyttja content, stabilized the measurements.

Based on these results, a ranking of the methods regarding apparent deviations between pre-sievings for each of the methods, from small to large number of deviations (observed in Figs. 1 and 2), showed: SPM < ISP (except ps0.063-SAB which had large deviations from ps0.2 and ps2) < LDM = LDMc.

3.2. Replicate variability

The replicate values for each combination of site-depth-pre-sieving (n = 3) are found in Figs. 1 and 2 for each method and particle size class, and their respective mean values in Table 4 and Table 5. Generalized range classes for each mean value (i.e. ‘small’ for ≤2% points, ‘intermediate’ for 2–4% points, ‘large’ for >4% points) are in Tables 4 and 5 represented by colour and font as explained in the table captions.

For SPM, out of all 72 cases (i.e. combination of 4 sites, 2 depths, 3 pre-sievings, 3 particle size classes) in Table 4, the ranges were ‘small’ in all 72 cases. Based on this observation of ‘small’ replicate variabilities for the SPM cases in general and the reference SPM-ps0.2 in particular, it can be assumed that any larger variabilities than ‘small’ for the other methods, i.e. > 2% points, were due to methodology rather than to soil property variability.

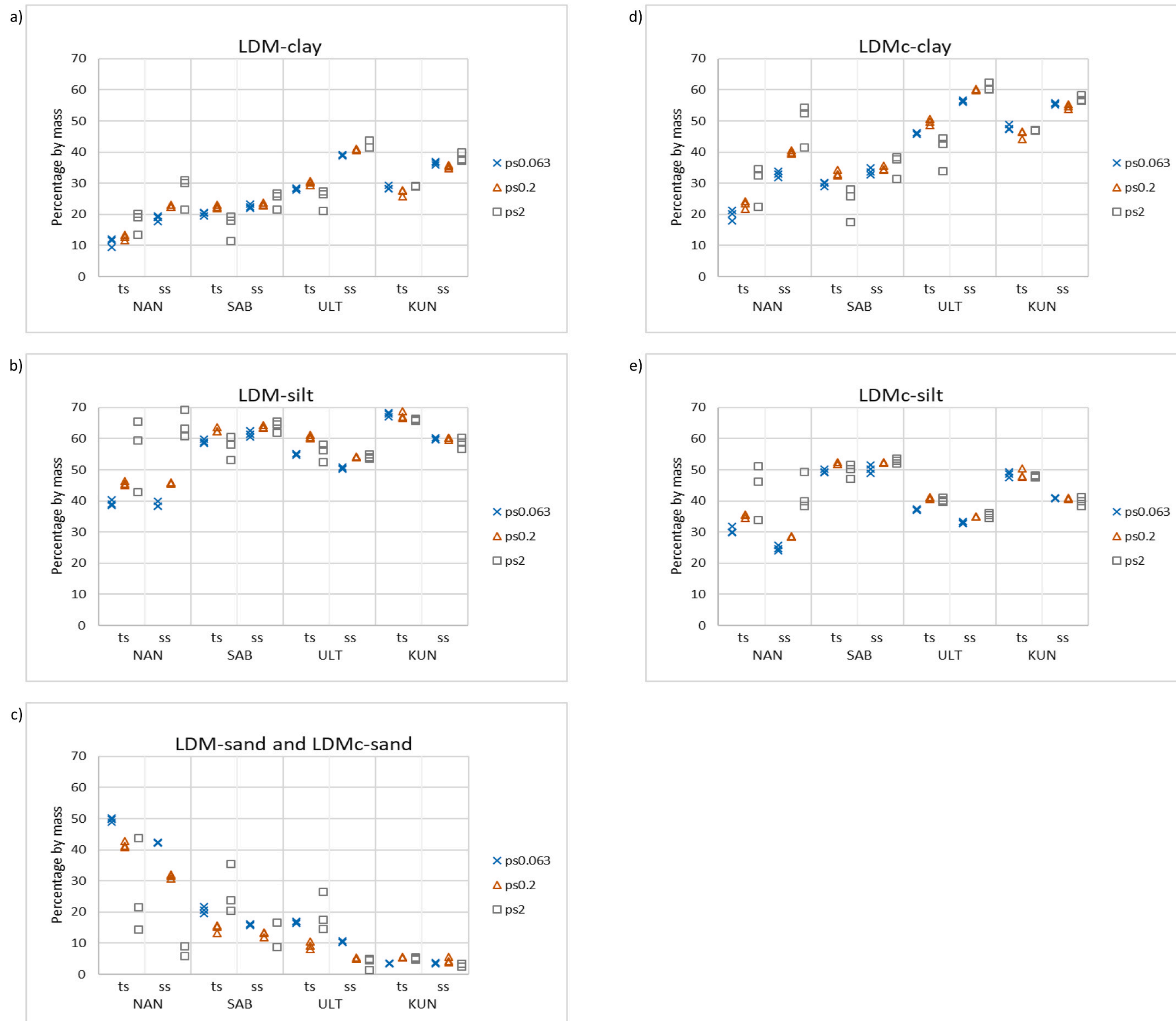

Fig. 2. Presentation of all replicate values (n = 3) in topsoil (ts) and subsoil (ss) of each combination of soil and pre-sieving for each main particle size fraction (clay= a, d; silt= b, e; sand= c), for the laser diffraction method with the standard (LDM= a, b, c) and clay-silt cutoff modified (LDMc= d, e, c) solutions.

For ISP, the ranges were generally 'small' for sand (6–8 out of 8 cases for each pre-sieving), whereas for clay and silt there were a greater number of 'large' ranges (4–8 out of 16 cases) (Table 4). KUN had, compared with the other soils, a greater number of 'small' ranges (16 out of 18 cases). As a whole for ISP, based on number of 'small' and 'large' ranges, ISP-ps0.063 was the optimum pre-sieving (14 'small' and 4 'large' ranges out of totally 24 cases).

For LDM, the majority of the ranges were 'small' for LDM-ps0.063 and LDM-ps0.2 (42 out of 48 cases) (Table 5), and there were no 'large' ranges for those pre-sievings. For LDM-ps2, on the contrary, cases with 'large' ranges predominated (13 out of 24), indicating that this pre-sieving was not suitable. An exception was KUN which had a greater number of 'small' ranges (4 out of 6 cases) than the other soils had and, furthermore, had no 'large' ranges. So for LDM, based on number of 'small' and 'large' ranges, LDM-ps0.063 (22 'small') and LDM-ps.0.2 (20 'small') were equally suitable optimum pre-sievings. Clay-silt cutoff modified LDMc had ranges fairly equal to LDM, i.e. the majority of cases were 'small' for LDMc-ps0.063 and LDMc-ps0.2 (39 out of 48 cases). Thus, there were no 'large' ranges for those pre-sievings, and LDMc-ps0.063 (20 'small') and LDMc-ps.0.2 (19 'small') were almost equally suitable optimum pre-sievings. LDMc-ps2 had, similarly to LDM-ps2, much greater number of 'large' ranges (i.e. 12 out of 24 cases) than the other pre-sievings.

Based on these results, a ranking between methods and pre-sievings regarding replicate variability, from small to large degree of ranges (Figs. 1 and 2 and Tables 4 and 5), showed: SPM-ps0.2 = SPM-ps2 $\leq$ SPM-ps0.063 < LDM-ps0.063 $\leq$ LDMc-ps0.063 = LDM-ps0.2 $\leq$ LDMc-ps0.2 < ISP-ps0.063 < ISP-ps0.2 $\leq$ ISP-ps2 < LDMc-ps2 $\leq$ LDM-ps2.

3.3. Relationships

Regarding the linear regression relationships with the reference SPM-ps0.2, for intercept set to zero (throughout this section), SPM-ps2 showed 1:1 relationships for all fraction classes, and SPM-ps0.063 showed close to 1:1 relationships (i.e. slopes for clay 1.02, silt 0.96, sand 1.01) (Table 6).

ISP, in relation with the reference SPM-ps0.2, showed strongest

Table 4
Mean of replicates (n = 3) for SPM and ISP, where their ranges (seen in Fig. 1) are classified into: 'small' range (≤2% points) as green-underlined normal font, 'intermediate' range (2–4% points) as black-normal font, and 'large' range (>4% points) as red-underlined italics font.

		NAN_ts	NAN_ss	SAB_ts	SAB_ss	ULT_ts	ULT_ss	KUN_ts	KUN_ss
SPM-ps0.063	Clay	30.0	34.3	29.5	32.4	40.2	54.1	48.5	54.0
	Silt	20.4	23.0	49.7	52.7	42.8	35.6	48.9	42.7
	Sand	49.5	42.8	20.8	14.8	17.0	10.2	2.6	3.4
SPM-ps0.2	Clay	29.9	33.4	26.5	30.1	39.4	52.6	48.4	54.6
	Silt	20.5	24.3	55.3	55.4	44.0	37.2	48.9	41.9
	Sand	49.6	42.3	18.2	14.5	16.6	10.3	2.7	3.5
SPM-ps2	Clay	29.6	33.6	25.8	30.4	39.7	52.5	48.0	54.0
	Silt	20.8	24.0	55.2	55.1	43.9	36.9	49.2	42.1
	Sand	49.6	42.5	19.0	14.5	16.4	10.6	2.8	3.9
ISP-ps0.063	Clay	27.1	31.9	*38.5*	*40.0*	37.9	49.0	44.2	52.2
	Silt	23.3	25.0	*41.8*	*44.8*	45.4	40.6	53.3	44.4
	Sand	49.6	43.1	19.7	15.1	16.7	10.4	2.5	3.3
ISP-ps0.2	Clay	*26.7*	*33.6*	*16.9*	23.6	*34.2*	*48.5*	44.8	50.9
	Silt	*23.3*	*23.4*	62.6	60.6	48.0	*40.6*	52.4	45.7
	Sand	50.0	43.0	20.6	15.8	*17.9*	10.9	2.7	3.4
ISP-ps2	Clay	28.8	30.4	*18.7*	*26.0*	36.2	50.7	41.9	*50.0*
	Silt	20.9	26.7	*60.5*	*58.5*	47.1	38.6	55.4	*46.4*
	Sand	50.3	42.8	20.8	15.5	16.7	10.7	2.7	3.6

Table 5
Mean of replicates (n = 3) for LDM and LDMc, where their ranges (seen in Fig. 2) are classified into: 'small' range (≤2% points) as green-underlined normal font, 'intermediate' range (2–4% points) as black-normal font, and 'large' range (>4% points) as red-underlined italics font.

		NAN_ts	NAN_ss	SAB_ts	SAB_ss	ULT_ts	ULT_ss	KUN_ts	KUN_ss
LDM-ps0.063	Clay	11.1	18.8	20.2	22.5	28.2	39.0	28.7	36.5
	Silt	39.3	38.9	59.1	61.5	55.1	50.5	67.8	60.0
	Sand	49.6	42.3	20.7	16.0	16.7	10.5	3.5	3.6
LDM-ps0.2	Clay	12.7	22.7	22.5	23.3	30.1	40.9	27.0	35.3
	Silt	45.6	45.8	62.8	63.9	60.6	54.1	67.5	60.1
	Sand	41.6	31.6	14.6	12.8	9.3	5.1	5.5	4.6
LDM-ps2	Clay	*17.6*	*27.6*	*16.2*	*24.6*	*24.9*	42.2	29.0	38.3
	Silt	*55.9*	*64.4*	*57.2*	64.0	*55.6*	54.2	66.0	58.6
	Sand	*26.5*	8.0	*26.5*	*11.4*	*19.5*	3.6	5.0	3.0
LDMc-ps0.063	Clay	19.8	32.8	29.8	33.8	46.0	56.4	48.0	55.5
	Silt	30.6	24.8	49.5	50.3	37.3	33.1	48.5	40.9
	Sand	49.6	42.3	20.7	16.0	16.7	10.5	3.5	3.6
LDMc-ps0.2	Clay	23.2	39.9	33.3	34.8	49.8	60.0	45.8	54.6
	Silt	35.2	28.5	52.1	52.3	40.9	34.9	48.7	40.8
	Sand	41.6	31.6	14.6	12.8	9.3	5.1	5.5	4.6
LDMc-ps2	Clay	*29.8*	*49.4*	*23.8*	*35.7*	*40.3*	61.0	47.1	57.1
	Silt	*43.7*	*42.6*	*49.7*	52.9	40.2	35.4	47.9	39.9
	Sand	*26.5*	8.0	*26.5*	*11.4*	*19.5*	3.6	5.0	3.0

relationships (largest R^2) for ISP-ps0.2 and ISP-ps2. Based on the slopes in Fig. 3, they underestimated clay contents with 9% (ISP-ps2, R^2 = 0.95) to 10% (ISP-ps0.2, R^2 = 0.92) (Fig. 3d, g) and overestimated silt contents with 8% (ISP-ps2, R^2 = 0.99) to 9% (ISP-ps0.2, R^2 = 0.99) (Fig. 3e, h). The sand contents were close to 1:1 relationship with SPM-ps0.2 (<3% overestimations) for all three ISP pre-sievings (Fig. 3c, f, i). The ISP-ps0.063 cases were closer to 1:1 relationship with SPM-ps0.2 than the other pre-sievings, but at the same time, for clay and silt, had weaker relationships (R^2 = 0.41 and 0.60, respectively) (Fig. 3a, b, c).

LDM, overall, showed larger differences in relation to SPM-ps0.2 reference values, than ISP did. LDM indicated strongest relationships (with SPM-ps0.2) for LDM-ps0.063, with 34% underestimated clay (R^2 = 0.78) and 27% overestimated silt (R^2 = 0.30) contents, and with sand contents almost equal to 1:1 relationship (Fig. 3a, b, c). Also LDM-ps0.2 and LDM-ps2, for clay, showed equally strong and similar relationships with SPM-ps0.2 as SPM-ps0.063, i.e. 32% (R^2 = 0.69) and 30% (R^2 = 0.82) underestimated values, respectively (Fig. 3d, g). For silt, however, LDM-ps0.2 and LDM-ps2 zero-intercept relationships with SPM-ps0.2 were non-significant, but, nevertheless, they showed similar slopes as LDM-ps0.063 (i.e. corresponding to 34% overestimations) (Fig. 3e, h). For sand, LDM-ps063 showed equally strong close to 1:1 relationship with SPM-ps0.2 as ISP, whereas LDM-ps0.2 showed 21%

Table 6
Relationships between pre-sievings of methods of sieve and pipette (SPM-ps0.063, SPM-ps2) and clay-silt cutoff modified laser diffraction (LDMc-ps0.063, LDMc-ps0.2, LDMc-ps2) versus (vs) reference SPM-ps0.2 for mean values (n = 3) of each combination of soil - depth - pre-sieving and each main particle size fraction (clay, silt, sand).

Pre-sieving/Fraction	Equation	R^2	Equation (intercept zero)	R^2
SPM-ps0.063 (y) vs SPM-ps0.2 (x)				
Clay	y = 0.937 x + 3.501	0.991	y = 1.020 x	0.983
Silt	y = 0.921 x + 1.778	0.980	y = 0.961 x	0.978
Sand	y = 1.003 x + 0.389	0.997	y = 1.014 x	0.997
SPM-ps2 (y) vs SPM-ps0.2 (x)				
Clay	y = 0.994 x + 0.053	0.999	y = 0.996 x	0.999
Silt	y = 0.996 x + 0.146	1.000	y = 0.999 x	1.000
Sand	y = 0.996 x + 0.276	1.000	y = 1.005 x	1.000
LDMc-ps0.063 (y) vs SPM-ps0.2 (x)				
Clay	y = 1.096 x - 2.895	0.859	y = 1.028 x	0.855
Silt	y = 0.680 x + 11.522	0.869	y = 0.939 x	0.732
Sand[a])	y = 0.986 x + 0.945	0.997	y = 1.015 x	0.996
LDMc-ps0.2 (y) vs SPM-ps0.2 (x)				
Clay	y = 0.965 x + 4.681	0.773	y = 1.076 x	0.762
Silt	y = 0.611 x + 16.693	0.841	y = 0.985 x	0.497
Sand[b])	y = 0.779 x + 0.291	0.963	y = 0.788 x	0.963
LDMc-ps2 (y) vs SPM-ps0.2 (x)				
Clay	y = 1.030 x + 2.479	0.775	y = 1.089 x	0.772
Silt	y = 0.239 x + 34.235	0.295	y = 1.007 x	-3.006
Sand[c])	y = 0.309 x + 6.878	0.290	y = 0.517 x	0.068

[a]) Identical with LDM-sand in Fig. 3c. [b]) Identical with LDM-sand in Fig. 3f. [c]) Identical with LDM-sand in Fig. 3i.

underestimated sand (R^2 = 0.96) and LDM-ps2 48% underestimation (however weak, R^2 = 0.07) (Fig. 3c, f, i). LDMc (Table 6), as compared with LDM (Fig. 3a, b, d, e, g, h), both for clay and silt shifted closer to 1:1 relationship with SPM-ps0.2 and resulted in larger R^2:s for LDMc-ps0.063 (R^2 = 0.86 for clay and 0.73 for silt) and LDMc-ps0.2 (R^2 = 0.76 for clay and 0.50 for silt). For sand, mean LDMc-SPM-ps0.2 ratios (Table 6) were by definition identical to those of LDM-SPM-ps0.2 ratios (Fig. 3c, f, i) and R^2 values were consequently also identical.

Based on these results, a ranking between methods and pre-sievings regarding relationships with SPM-ps0.2, from large to small R^2 (zero-intercept) (Table 6 and Fig. 3), showed: SPM-ps2 ≥ SPM-ps0.063 > ISP-ps2 = ISP-ps0.2 > LDMc-ps0.063 > LDM-ps0.063 = ISP-ps0.063 ≥ LDMc-ps0.2 > LDM-ps0.2 > LDM-ps2 = LDMc-ps2.

3.4. Impact on texture classification

Obtained textural classes as compared with the reference SPM-ps0.2 classes ('good', 'intermediate', 'poor' agreements) are in Table 7 and Table 8 represented by colour and font as explained in the table captions. For the original non-transformed clay-silt-sand percentages (Table 7), SPM-ps2 and ISP-ps2 showed 'good' agreement with the reference SPM-ps0.2, i.e. they fell in identical textural classes as SPM-ps0.2 in all or most cases (7–8 cases out of 8, i.e. 80–100%), whereas SPM-ps0.063, ISP-ps0.063 and ISP-ps0.2 showed 'intermediate' (5–6 cases; 55–80%) agreement, and LDM-ps0.063 showed 'poor' (≤4 cases; <55%) agreement.

For linear-transfer transformed clay-silt-sand percentages compared with the reference (non-transformed) SPM-ps0.2 (Table 8), the combinations SPMt-ps2 and ISPt-ps2 remained in 'good' agreement (7–8 out of 8 identical cases). SPMt-ps0.063 and ISPt-ps0.2 were upgraded (in relation to original classes in Table 7) from 'intermediate' to 'good' (7 cases) agreement (Table 8), whereas ISPt-ps0.063 remained in 'intermediate' (5–6 cases) agreement. LDMt-ps0.063 was upgraded from 'poor' (Table 7) to 'intermediate' (5 cases) agreement (Table 8). Clay-silt cutoff modified LDMc-ps0.063 and LDMc-ps0.2 showed 'intermediate' (5 cases) agreement with SPM-ps0.2. Thus, linear-transfer transformed LDMt-ps0.063 and cutoff modified LDMc-ps0.063 showed similar improvement of texture class agreement with SPM-ps0.2.

To summarize textural classification, a rating of agreement (identical cases) with SPM-ps0.2 based on non-transformed, linear-transfer transformed and clay-silt cutoff modified values (Tables 7 and 8), showed:

i) 'Good' (7–8 cases of totally 8, i.e. >80–100% agreement); SPM-ps2 = SPMt-ps2 = ISPt-ps2 ≥ SPMt-ps0.063 = ISPt-ps0.2 = ISP-ps2 > ,

ii) 'Intermediate' (5–6 cases, i.e. 55–80%); SPM-ps0.063 = ISP-ps0.2 ≥ ISPt-ps0.063 ≥ ISP-ps0.063 ≥ LDMt-ps0.063 = LDMc-ps0.063 = LDMc-ps0.2 > ,

iii) 'Poor' (≤4 cases, i.e. ≤55%); LDM-ps0.063.

4. Discussion

4.1. Pre-sieving deviations and replicate variability

The observations in the results (Sections 3.1 and 3.2) that SPM showed smaller number of apparent pre-sieving deviations and smaller replicate variabilities than the other two methods may to some extent be due to that SPM has been the standard method in our laboratory for several decades. Thereby, the variability caused by the analytical procedures management has been minimized. The larger extent of variabilities observed for ISP and LDM reveals that modifications in the procedures for ISP and LDM are still needed, if they are to be related to reference SPM values.

For ISP, the results in Sections 3.1 and 3.2 for the soil with higher silt content (SAB) resemble the results from Durner et al. (2017b) where pre-sieving with 0.063 mm resulted in smaller silt contents than pre-sieving with larger meshes. In the present study, however, this did not apply to the other clay soils with lower silt contents (i.e. NAN, ULT, KUN) for which ps0.063 ISP contents varied within the same ranges as ps0.2 and ps2. Since the sand contents were fairly similar between the three ISP pre-sievings (Fig. 1f), the sensitivity to silt content may be searched in the analytical procedures for estimating the finer fractions (clay and silt). Durner et al. (2017b) reported that homogenization of the suspension by overhead shaking gave lower reproducibility and smaller silt fractions, resulting in larger clay contents, than vertical stirring. This would in the present study, where overhead shaking was applied to ISP and vertical stirring to SPM, affect the results for ps0.063 but not for ps0.2 and ps2. For ISP, accuracy in temperature control and initial insertion of the probes in the test cylinders are vital and can give acceptable results (Nemes et al., 2020). The proposed modification in ISP methodology, i.e. ISP+ , is reported to minimize apparent deviations and replicate variability by reducing the uncertainty of the identified clay fractions (Durner and Iden, 2021). Furthermore, the measurement time could be reduced from 8 h (ISP) to about 2 h (ISP+) without affecting the accuracy. However, it brings about an extra physical weighing of the finest sediments, i.e., at the end of an ISP+ measurement run a part of the suspension is released laterally from the sedimentation cylinder through an outlet, collected in a beaker and oven-dried, and the dry mass of the collected soil particles is integrated into the calculations.

For LDM, the deviations and variability (Sections 3.1 and 3.2) for ps2 for the clay with high sand content (NAN) were probably due to the

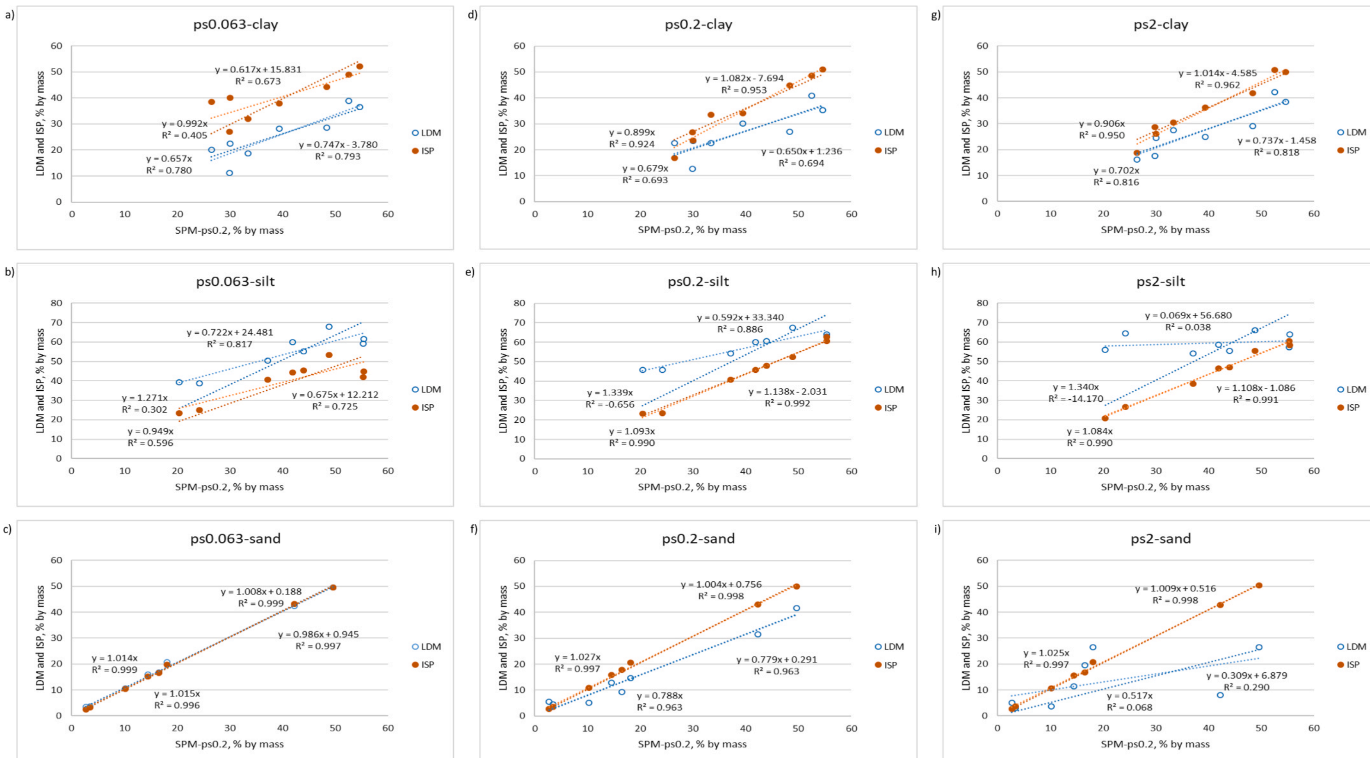

Fig. 3. Relationships between methods of integral suspension pressure (ISP) and standard laser diffraction (LDM) versus reference sieve and pipette (SPM-ps0.2) for mean values (n = 3) of each combination of soil - depth - pre-sieving (ps0.063 = a, b, c; ps0.2 = d, e, f; ps2 = g, h, i) and each main particle size fraction (clay= a, d, g; silt= b, e, h; sand= c, f, i).

Table 7
Textural classification, based on WRB (FAO, 2015) soil fraction limits, of SPM, ISP and LDM, where Cl= clay, Si= silt, si= silty, Lo= loam and sa= sandy. Classes as compared with the reference SPM-ps0.2 marked with: green-underlined normal font= 'good' (>80% agreement), black-normal font= 'intermediate' (55–80%), red-underlined italics font= 'poor' (<55%).

	NAN_ts	NAN_ss	SAB_ts	SAB_ss	ULT_ts	ULT_ss	KUN_ts	KUN_ss
SPM-ps0.063	saClLo	ClLo	ClLo (-siClLo)	siClLo	siCl (-siClLo)	Cl	siCl	siCl
SPM-ps0.2 [a]	saClLo	ClLo	SiLo (-siClLo)	siClLo	siClLo (-siCl)	Cl	siCl	siCl
SPM-ps2	saClLo	ClLo	SiLo	siClLo	siClLo (-siCl)	Cl	siCl	siCl
ISP-ps0.063	saClLo	ClLo	siClLo (-ClLo)	siCl (-siClLo)	siClLo	siCl (-Cl)	siCl	siCl
ISP-ps0.2	saClLo	ClLo	SiLo	*SiLo*	siClLo	siCl (-Cl)	siCl	siCl
ISP-ps2	saClLo	ClLo	SiLo	SiLo (-siClLo)	siClLo	Cl	siCl	siCl
LDM-ps0.063	*Lo*	*Lo*	SiLo	*Si Lo*	siClLo	*siClLo*	*siClLo*	*siClLo*
LDM-ps0.2	na [b]	na	na	na	na	na	na	na
LDM-ps2	na	na	na	na	na	na	na	na

[a]) Reference.
[b]) na= Not applicable (not classified due to the poor silt relationships with SPM-ps0.2 (see Fig. 3)).

turbidity in the measurement cell not corresponding to the optimal detection range. In this context, it is shown in the present and other studies (e.g. Buurman et al., 2001; Faé et al., 2019; Nimblad Svensson et al., 2022) that pre-sieving the fine-earth fraction further than ps2 (i.e. ps0.063 or ps0.2) may be needed, in order to reduce the variability induced by the coarsest sand particles. This variability is due to the complication of filling of a homogeneous aliquot and to possible settling of particles inside the instrument itself, or to possible obscuring of smaller particles from detection (Taubner et al., 2009). Another influencing factor was that in all LDM-ps0.063 samples, despite being pre-sieved at 0.063 mm, particles larger than 0.063 mm were detected by the laser diffraction in the suspension, and this to a larger degree for NAN and SAB than for ULT and KUN. Those particles were in the present study added to the coarse-silt fraction, like in Taubner et al. (2009), to hold on to the 0.063 mm sieve as reference. Furthermore, the smaller amount of sample used in LDM as compared with SPM and ISP (Table 2) may result in a larger uncertainty of measurement, and thereby a need to perform sufficient replicates to come up with representative results.

Other aspects may affect the results differently for the different combinations of methods and pre-sievings. One example is the limitation imposed by the fact that the sum of the composition of the three particle sizes, clay, silt and sand should add to 100%, for example in the cases when two particle size fractions determines the third one. Another example is soil characteristic (Table 1). It was observed that the clay soil with gyttja content (i.e. KUN) showed smaller replicate variabilities than the other soils (Figs. 1 and 2, and Table 4 and Table 5). It can be argued whether this is due to actually smaller replicate variabilities or that the gyttja content by its nature (i.e. consisting of organic compounds

Table 8
Textural classification, based on WRB (FAO, 2015) soil fraction limits, of linear-transfer transformed SPMt, ISPt and LDMt and clay-silt cutoff modified LDMc, where Cl= clay, Si= silt, si= silty, Lo= loam and sa= sandy. Classes as compared with the reference SPM-ps0.2 marked with: green-underlined normal font= 'good' (>80% agreement), black-normal font= 'intermediate' (55–80%), red-underlined italics font= 'poor' (<55%).

	NAN_ts	NAN_ss	SAB_ts	SAB_ss	ULT_ts	ULT_ss	KUN_ts	KUN_ss
SPMt-ps0.063	saClLo	ClLo	ClLo (-siClLo)	siClLo	siClLo (-siCl)	Cl	siCl	siCl
SPM-ps0.2 [a]	saClLo	ClLo	SiLo (-siClLo)	siClLo	siClLo (-siCl)	Cl	siCl	siCl
SPMt-ps2	saClLo	ClLo	SiLo	siClLo	siClLo (-siCl)	Cl	siCl	siCl
ISPt-ps0.063	saClLo	ClLo	siClLo (-ClLo)	siClLo (-siCl)	siClLo	*siCl*	siCl	siCl
ISPt-ps0.2	saClLo	ClLo	SiLo	SiLo-siClLo	siClLo	Cl	siCl	siCl
ISPt-ps2	saClLo	ClLo	SiLo	siClLo	siClLo (-siCl)	Cl	siCl	siCl
LDMt-ps0.063	*Lo*	ClLo	ClLo (-siClLo)	siClLo	siCl	Cl	siCl	siCl
LDMt-ps0.2	na [b]	na	na	na	na	na	na	na
LDMt-ps2	na	na	na	na	na	na	na	na
LDMc-ps0.063	*Lo*	ClLo	ClLo (-siClLo)	siClLo	*Cl*	Cl	siCl	siCl (-Cl)
LDMc-ps0.2	*Lo*	ClLo (-Cl)	siClLo	siClLo	siCl (-Cl)	Cl	siCl	siCl (-Cl)
LDMc-ps2	na	na	na	na	na	na	na	na

[a]) Reference.
[b]) na= Not applicable (not classified due to the poor silt relationships with SPM-ps0.2 (see Fig. 3)).

intimately mixed with inorganic particles) affects dispersion, sieving or other factors in the laboratory procedures.

4.2. Relationships and texture classification

Regarding ISP, Nemes et al. (2020) presented values having similar near 1:1 regression relationship with SPM for silt fraction, and slightly weaker relationship and smaller regression slope for clay, as compared with the present study (Section 3.3). They were using samples with a larger proportion of soils with clay content smaller than 25%. The weaker relationships for ISP-ps0.063 in our study - as compared with ps0.2 and ps2 - may be searched in the analytical procedures for estimating the finer fractions (clay and silt) as noted in Section 4.1. ISP showed 'good' texture class compatibility with the reference SPM-ps0.2 already for non-transformed ISP-ps2.0 values (Section 3.4.). Linear-transfer transformed values improved the compatibility to 'good' also for ISPt-ps0.2, but not for ISPt-ps0.063 (Table 8). ISP compatibility with SPM may nevertheless, as outlined in Section 4.1, be increased by improved temperature control and probe management during the tests (Nemes et al., 2020) or with application of the improved ISP+ method (Durner and Iden, 2021). For example, Durner and Iden (2021) showed a significantly improved precision of the ISP+ results in the range of silt and clay particles and very good agreement with the pipette method, i.e. all 5 soils and 6 replicates studied falling in identical texture class as pipette.

Regarding LDM, previous studies reported that the pipette method for the clay fraction generated results approximately two to three times larger than the laser method (Konert and Vandenberghe, 1997; Buurman et al., 2001; Taubner et al., 2009; Nemes et al., 2020; Bittelli et al., 2022). Buurman et al. (2001) also showed the influence of soil parent material in that laser diffraction gave 42% of pipette method clay in marine samples, and 62% in fluvial and loess samples. Compared with these studies, our study (Section 3.3) showed smaller discrepancies between the two methods, i.e. LDM-SPM slopes of 0.65–0.70 for the clay fraction (corresponding to only 1.4–1.5 times larger values for SPM as compared with LDM).

To compensate for the underestimate of the clay fraction by laser diffraction, Konert and Vandenberghe (1997) suggested using the laser diffraction fraction of < 0.008 mm as a proxy for pipette fraction < 0.002 mm. Buurman et al. (2001) argued that in many samples the 'real' fraction 0.002–0.008 mm will consist of both platy and non-platy particles while in other samples it does not contain any platy minerals. This is probably one of the reasons that, for example, Makó et al. (2017) detected the clay-silt cutoff limit at 0.0058 mm (with organic matter removed) and 0.0066 mm (without organic matter removal) and Nimblad Svensson et al. (2022) at 0.0039 mm. It may therefore be desirable to obtain the correct correlation for each type of sediment (Konert and Vandenberghe, 1997; Buurman et al., 2001). In the present study, we used < 0.0039 mm (adapted from Nimblad Svensson et al., 2022) as corresponding proxy to generate LDMc which resulted in, as compared with LDM, stronger and closer to 1:1 relationships with SPM-ps0.2 (Table 6 and Fig. 3a, b, d, e, g, h).

Sand contents were in the present study for all three methods based on sieving the 0.063 to 2.0 mm fraction with the exception of LDM (and thereby also LDMc) for ps0.2 and ps2. The relatively large deviations from the 1:1 relationship with SPM-ps0.2 for sand for LDM and LDMc, i. e. for ps0.2 a slope of 0.79 and for ps2 a slope of 0.52 (Fig. 3f, i and Table 6), were possibly due to the difficulties in creating homogeneous solutions for laser diffraction of particles > 0.063 mm (as outlined in Section 4.1). For the other methods in the present study, the relatively small deviations from 1:1 relationship with SPM-ps0.2 (Table 6 and Fig. 3) may be within the possible variability generated in the sieving procedures. Interestingly, Buurman et al. (2001) showed the influence of soil parent material by reporting sand fractions (>0.0050 mm) detected by laser diffraction being 107% of the sieve fraction in marine soil samples, and 99% in the fluvial soil samples.

Regarding texture classification, LDM-ps0.063 showed in the present study 'poor' texture class compatibility with SPM-ps0.2 for non-transformed values (Table 7), but improved it to 'intermediate' with linear-transfer transformed LDMt-ps0.063 and clay-silt cutoff modified LDMc-ps0.063 values ('intermediate' also for LDMc-ps0.2) (Table 8). For LDM-ps0.063 in the present study, applying the linear-transfer equation slopes from Nimblad Svensson et al. (2022) (i.e. for clay 0.662, silt 1.217 and sand 0.998), which were based on a much larger number of soil samples to transform the measured values (i.e. 44 soil samples from a national sample scheme), gave almost identical texture classes as linear-transfer transformed LDMt-ps0.063 using the slopes from Fig. 3a, b, c (i.e. for clay 0.657, silt 1.271 and sand 1.015) and as clay-silt cutoff modified LDMc-ps0.063 in the present study. This implicates that linear-transfer equation slopes from the large national sample scheme reported by Nimblad Svensson et al. (2022) may be considered suitable to apply to other smaller sample schemes of similar pedology and parent material, like the eight samples scheme in the present study. The Nimblad Svensson et al. (2022) study showed 77% (34 out of 44 samples) and 82% (36 out of 44) texture class compatibility with SPM-ps0.2 for linear-transfer transformed and clay-silt cutoff modified laser LDM-ps0.063, respectively. Taubner et al. (2009) showed for corresponding methods (linear transformed laser versus standard pipette) a compatibility of 62.5% (10 out of 16 samples), to be compared with, similarly, 62.5% (5 out of 8) in the present study (LDMt-ps0.063 in Table 8). Like in the present study, Bitelli et al. (2022) used regression equations and modified upper limit for the clay range to convert data. They demonstrated that the laser diffraction was in better agreement with an optical (digital imaging) method than traditional sedimentation methods, suggesting that the standards for particle size analysis be changed from sedimentation to laser diffraction methodologies.

The discrepancies between the methods have, as outlined in the introduction section, been attributed to the theoretical assumption of particles spherical shape irrespective of the actual particle mineralogy and shape. For SPM, for example, this assumption results in higher effective clay content (e.g. Eshel et al., 2004), and for LDM it results in higher effective silt content (Taubner et al., 2009). Both the 0.0020 mm (clay-silt) and the 0.0063 mm (silt-sand) boundaries cause problems in the comparisons, the first because of occurrences of platy shape of clay minerals, and the second due to both a change in method in the pipetting and sieving procedures and to non-sphericity of particles (Buurman et al., 2001). Furthermore, laser device type as well as settings of pump and agitator speed, and scattering parameters (refractive index, absorption coefficients) influence the output values as well as their proper modifications (Bieganowski et al., 2018; Nimblad Svensson et al., 2022).

5. Conclusions

A set of clay soils were compared for three particle size distribution methods (SPM, ISP, LDM), and tested for three pre-sieving options (ps0.063, ps0.2, ps2). The study shows that soil particle size estimations across different particle size classes, pre-sieving options and methods of measure were not straight-forwardly transferrable, and varied as a result of the underlying assumptions and measurement technologies of each method. They responded differently to variations in silt (ISP), sand (LDM) and gyttja (all three methods) contents in these clay soils. In comparison with the reference SPM-ps0.2, ISP clay contents were 1–10% smaller and LDM clay contents 30–34% smaller. Linear-transfer transformed ISP and LDM as well as clay-silt cutoff modified LDM decreased these differences and improved texture compatibility with SPM-ps0.2.

Pre-sieving options for the three methods, considering the combined effect of all aspects in the study (i.e. deviations, replicate variability and compatibility with the reference SPM-ps0.2 regarding linear relationships and textural classes), revealed that SPM-ps2 was equally suitable as the reference SPM-ps0.2. Regarding ISP, being less suitable than all SPM pre-sievings, linear-transfer transformed ISPt-ps2 and to almost the same degree, linear-transfer transformed ISPt-ps0.2, performed best. And for LDM, also being less suitable than all SPM pre-sievings, linear-transfer transformed LDMt-ps0.063 and clay-silt cutoff modified LDMc-ps0.063, and to almost the same degree LDMt-ps0.2 and LDMc-ps0.2, performed best. ISP has shown inaccurate results in previous studies but, nevertheless, performs somewhat better than LDM in some aspects in the present study. Further comparative studies may preferably be conducted with the improved ISP+ method.

Similar to previous studies, we identify the need for further efforts to create widely accepted procedures for soil sample treatment and measuring procedures to improve compatibility between methods for soil particle size analysis. Linear-transfer transformation is a logical approach to identify the strength in relationships between the methods, but may be context specific (non-universal) and dependent on soil parent material. Further studies of variations in such transformations as related to soil particle size distributions and soil origin are needed, including the need for exploring the influence of actual particle shape and size on the concept of equivalent and efficient particle.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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