



Recommendations for soil sample preparation, pretreatment, and data conversion for texture classification in laser diffraction particle size analysis

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ABSTRACT

With regard to the differences between soil particle size distribution (PSD) obtained by sieve-sedimentation methods (SSMs), e.g., the sieve-pipette method (SPM) and the laser diffraction method (LDM), usually, the clay fraction content in LDM measurements is lower than in SSM. Two groups of reasons can be identified for this. Firstly, differences resulting from the features of the methods themselves. Secondly, differences resulting from the soil sample preparation (after sampling) and pretreatment (disaggregation). These differences not only cause difficulties in the PSDs comparability but also make it difficult, and sometimes even impossible, to apply the LDM results to soil texture classification. The solution to this difficulty may be to use pedotransfer functions and standardization of measurement procedures. The aim of this work was to validate the pedotransfer function proposed by Makó et al. (2017) on a Polish soil physics database for recalculating the results from LDM to compare them with SPM results and assess how the soil preparation and pretreatment influenced the obtained PSD and resulting soil texture classification. Using the pedotransfer function, 74 % accuracy was achieved, which is comparable with the results of other pedotransfer functions reported in the literature. Comparing the PSD results for the methods of sample preparation found in the literature after sampling (analysis of fresh, air dry and oven-dried – in 105 °C – soil) and pretreatment before the measurement (breaking soil aggregates chemically and physically) allows recommending the conducting of LDM PSD measurements using air dry soils and soil ultrasound disaggregation.

1. Introduction

Particle size distribution (PSD) is one of the basic quantities used to describe the properties of soil. PSD directly or indirectly influences chemical (Huang et al., 2020; Tkaczyk et al., 2017), biological (Frąc et al., 2020; Hemkemeyer et al., 2018; Uzarowicz et al., 2020), and physical (Sokołowska et al., 2020; Szyplowska et al., 2019; Wang et al., 2021) soil properties such as water retention, aeration, fluid/gas exchange, and sorption (Brogowski and Chojnicki, 2019; Hu et al., 2011).

PSD is important or even necessary in many soil investigations such

as: water dynamic modelling (Lamorski et al., 2013; Rastgou et al., 2019), pedotransfer functions (Hernádi and Makó, 2014; Lamorski et al., 2008; Tóth et al., 2015), geomorphological processes (Dobrowolski et al., 2012; Tanaka and Watanabe, 2015). Additionally, PSD provides the input data to define fertilization recommendations (Watroś, 2019). However, it seems that the most important issue for which PSD is necessary is soil classification (Kabała et al., 2019; USDA, 1999; IUSS Working Group WRB, 2015).

The most commonly used PSD measurement methods are originally the sieve-sedimentation methods (SSMs), i.e., sieve-pipette (SPM) and

Abbreviations: LDM, laser diffraction method; SSM, sieve-sedimentation method; SPM, sieve-pipette method; LDMmod, modified laser diffraction method; LDMadj, adjusted laser diffraction method; PSD, particle size distribution; OA, overall accuracy; UA, user's accuracy; PA, producer's accuracy.

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hydrometer methods, and lately the laser diffraction method (LDM) (Makó et al., 2019). There are also other useful approaches such as the integral suspension pressure method (Durner et al., 2017) and the automated dynamometer method integrated with an x-y sampler changer (Kaszubiewicz et al., 2020). SSMs are based on a two-step procedure; firstly, the sand fraction is separated from the pretreated and shaken soil suspension by sieving, then dried and weighted. In the second step, Stokes' law is the basis of measurement, whereby the sand fraction-free suspension is left for sedimentation (ISO 11277:2020, 2020). After this, the change of suspension density is measured at strictly defined time intervals (hydrometer method), or the dry mass of the soil sample is collected with a calibrated pipette from a given height of the cylinder, also after strictly defined time intervals (SPM).

The LDM is based on the measurement of light intensity scattered on the soil particles intensively pumped through the measuring cell on which a laser beam is incident. The scattered light is registered on the detector array placed angularly around the measuring cell. The signal recorded on the detectors allows the calculation of the PSD. The Mie theory (which requires knowledge of the optical parameters of the sample) or Fraunhofer approximation is usually used for these calculations (ISO 13320:2020, 2020).

There were also attempts to combine LDM with the sieve method (von Suchodoletz et al., 2013; Nimblad Svensson et al., 2022; Faé et al., 2019). The sieve-laser method consists in separating and measuring sand fractions on sieves and measuring finer fractions using laser diffraction. When measuring PSD, the primary particles should be present in the suspension. This means that all soil aggregates should be broken down to primary particles. In practice, however, it is often impossible to destroy all aggregates because some of them are very stable, which is why the pretreatment procedure is so important. The repeatable procedure minimizes random errors and enables comparing the results obtained by different laboratories. There are two main procedures of disaggregation of soil samples:

- physical, i.e., mixing, shaking, use of ultrasounds;
- chemical, i.e., addition of dispersant.

Chemical procedures are used both as a disaggregation method and also as a method of adhesives release – to prevent particles from sticking together.

Reagents recommended by ISO 11277:2020 (2020) used for chemical dispersion are sodium hexametaphosphate in anhydrous sodium carbonate or sodium pyrophosphate solution. However several other dispersants are used in soil laboratories such as sodium bicarbonate, sodium hydroxide, and sodium oxalate (Bieganowski et al., 2018). The advantage of chemical methods is that, theoretically, only the aggregates are dispersed, while primary particles remain unaffected. However, the effectiveness of these methods may vary depending on the sample (aggregate stability, cementing agents, etc.) and reagents used. The use of pyrophosphate for samples containing calcium ions results in the formation of persistent insoluble salt crystals that interfere with the measurement (Shein, 2009). Moreover, the pyrophosphate effect can also be impermanent, and subsequent flocculation may occur (Gorączko and Topoliński, 2020). Similar limitations also exist in the application of hexametaphosphate. Different results can also be achieved depending on the type of mineral dispersed (Tan et al., 2017).

The removal of organic matter by H_2O_2 treatment is also widely discussed (Allen and Thornley, 2004; Gray et al., 2010; Jaijell et al., 2021). It was found to be sufficient to destroy most of the adhesives but the effect of this depends highly on the sample (Vasskog et al., 2016). Generally, organic removal is recommended if PSD is measured by standardized sieve-sedimentation methods (ISO 11277:2020, 2020). However, recent studies measuring PSD by LDM show that the removal of organic matter can be omitted for soils with low and medium amounts of carbon (Koza et al., 2021) or for using PTFs (Makó et al., 2017). The use of chemical methods is also quite labor-intensive, which reduces

their attractiveness.

The advantage of the ultrasound method (often along with mixing) is that the sample remains chemically unmodified. Moreover, the method is relatively easy to apply, which in practice can lead to the replacement of cumbersome adhesive (humus, carbonates, iron oxides) removal procedures. The disadvantage of the ultrasound method is the fact that some soil minerals can be milled under the influence of ultrasound waves (Boels et al., 2011; Wagterveld et al., 2011), overstating the proportion of smaller fractions in the sample. Ultrasounds may also be incapable of breaking certain permanent aggregates (Tyugai et al., 2010). Another problem with this method is the lack of equipment standardization (probe power, and the shape and volume of dispersing unit vessels), which determines the actual amount of energy used during dispersion (Bieganowski et al., 2018).

Another unresolved issue is the effect of sample preparation before measurement. For example, ISO 11277:2020 (2020) states that if sample properties can be changed by drying, fresh soil can be used. Makó et al. (2017) used air dried soil material, Fisher et al. (2017) preferred drying the soil at 40 °C, Sperazza et al. (2004) tested sediments dried at 50 °C, while Segal et al. (2009) and Jaijell et al. (2021) used a temperature of 105 °C for drying soils and sediments respectively. Drying at different temperatures may influence the effectiveness of dispersion and there are studies examining the effect of drying on aggregate stability (which can be indirectly interpreted as being important in the context of disaggregation efficiency) (Amézqueta, 1999). However, there are no literature reports that show the effect directly in the context of PSD measurements, especially in combination with aggregate disaggregation.

Regardless of the sample preparation method used, the results obtained from SSM methods are not identical to those from LDM. Usually, the clay fraction content in LDM measurements is lower than in SSM, so work has been undertaken over the years to resolve this problem. Initially, linear equations were obtained by comparing the results from both methods (Cooper et al., 1984; Loizeau et al., 1994; Muggler et al., 1997), but these were not universal, so new equations were sought to convert the results from the LDM to the SSM (Arriaga et al., 2006; Bittelli et al., 2022; Eshel et al., 2004; Taubner et al., 2009). An alternative way of dealing with the problem of comparability of results using SSM and LDM was to change the clay fraction upper threshold to, for example, 8 μ m (Konert and Vandenberghe, 1997).

Fisher et al. (2017) used Lin's concordance correlation coefficient to determine the sand, silt, and clay fraction thresholds for LDM and proposed a 9 μ m threshold for clay-silt fraction. Based on the same parameter, Qiu et al. (2021) specified the same limit in the range 4–6 μ m and also between 45 and 64 μ m for silt-sand. In turn, Makó et al. (2017) proposed shifting the borders to 6.6 μ m for clay-silt fraction and 60.3 μ m for silt-sand fraction. The clay, silt and sand fractions thus modified were then used as input parameters for the pedotransfer functions they developed and suggested.

The question that should be asked relates to the main objective of this work: is it possible using LDM to obtain soil classification comparable with that obtained by SPM? For the purposes of this work, the PSDs obtained by SPM are considered as the correct results and considered to be the absolute values referred to. However, in reality, these results are not absolute as they are fundamentally dependent on the measurement procedure adopted.

Recently, Bittelli et al. (2022) showed that LDM measurements are actually in better agreement with the direct method of size measurement (digital imaging) than SPM. They also suggested that for LDM/SPM comparison and texture classification, either results need to be recalculated with linear regression or the clay fraction limit should be changed to 8 μ m.

Finding a solution to the problem of comparison of the results obtained by both methods (i.e., SSMs and LDM) becomes more and more pressing because i) a lot of historical soil data is based on the PSD measurement by SSMs, ii) SSMs are still very often used in soil laboratories, while iii) LDM popularity is increasing. As the above-mentioned

issues (i.e., the soil pretreatment, aggregates disintegration, and recalculation of the LDM results to make them comparable with SSMs results) influence the obtained soil PSD results, the authors of this paper undertook studies to assess how the adopted procedures affect comparability.

The main objective of this work was to evaluate the possibility of using LDM for soil texture classification – as compared with SPM – and to check whether the method of sample preparation changes the soil texture classification results obtained by the increasingly popular LDM. The research was carried out in two stages. The aim of the first was to validate the pedotransfer function proposed by Makó et al. (2017) for recalculating the results from LDM to compare them with SPM results, tested on Polish soils representing Central Europe. In the second stage, the selected soils (with different textures) were investigated to assess how the soil preparation and pretreatment influenced the obtained PSD and resulting soil texture classification.

2. Materials & methods

2.1. Materials

116 soils from the arable horizon (5–25 cm), representative of Poland (Bieganski et al., 2013) were taken and sieved using a 2 mm mesh sieve and then air dried. Total organic carbon (TOC) and inorganic carbon (IC) content of all soils was measured (n = 3) with a TOC V_{CPH} analyzer coupled with an SSM5000 solid sample combustion unit (Shimadzu, Japan). Soil textures assessed by SPM, according to ISO 11277:2020 (2020) are presented in Fig. 1A.

One of the aims of this work was to validate the pedotransfer function proposed by Makó et al. (2017) which was developed on the set of 400 soils from the LUCAS (Land Use/Land Cover Area Frame Survey) database representative of Europe. Although the total number of soils in the LUCAS investigation was large, the number of samples representative for individual regions of Europe was relatively small. Therefore, in the 1st stage, it is necessary to validate this pedotransfer function by focusing on the soil sets more representative of such regions. To meet this challenge, the 116 soil samples representative of Poland (Bieganski et al., 2013) in the present study were recalculated with the proposed pedotransfer function and analyzed.

In the 2nd stage, the impact of sample preparation and pretreatment on a selection (based on texture, from the set of soils measured in the 1st stage) of 16 fresh (not-dried in laboratory) soils, having different textures, sieved using a 2 mm mesh sieve, were analyzed. The basic information for these soils is presented in Table 1.

Table 1
Basic information about soils used for the sample preparation and pretreatment impact.

Soil No.	Soil unit according to IUSS-WRB 2015	TOC*	IC*	SPM* clay content [mass %]
		[mass %]	[mass %]	
1	Haplic Cambisol (Siltic)	2.22 ± 0.03	0.00	6
2	Haplic Cambisol (Siltic)	4.23 ± 0.08	1.87 ± 0.04	19
3	Rendzic Leptosol	2.58 ± 0.07	2.12 ± 0.07	18
4	Rendzic Leptosol	2.12 ± 0.12	0.26 ± 0.02	16
5	Haplic Luvisol (Loamic)	2.32 ± 0.19	0.01 ± 0.00	2
6	Haplic Cambisol (Siltic)	1.63 ± 0.03	0.00	19
7	Stagnic Fluvisol (Ochric)	3.89 ± 0.07	0.00	23
8	Eutric Mollic Gleysol (Loamic)	4.40 ± 0.15	0.00	32
9	Eutric Cambic Hyperskeletal Leptosol (Loamic, Ochric)	1.72 ± 0.01	0.01 ± 0.01	16
10	Pellic Vertisol (Aric, Humic, Endostagnic)	1.81 ± 0.02	0.00	27
11	Haplic Vertisol (Aric, Drainic, Hypereutric Humic)	1.68 ± 0.11	0.39 ± 0.17	27
12	Pellic Vertisol (Aric, Drainic, Hypereutric, Humic, Stagnic)	3.81 ± 0.05	0.00	33
13	Haplic Vertisol (Gleyic)	2.75 ± 0.05	0.02 ± 0.01	45
14	Haplic Vertisol (Gleyic)	2.62 ± 0.02	0.00	53
15	Haplic Vertisol (Gleyic)	2.43 ± 0.03	0.00	30
16	Haplic Vertisol (Gleyic)	2.50 ± 0.11	0.00	24

*TOC – total organic carbon mean content; IC – inorganic carbon mean content; SPM – sieve-pipette method; ± – standard deviation.

2.2. Sample preparations and pretreatments

For the pedotransfer function validation (1st stage), air dried soils with calgon solution for disaggregation (33 g of sodium hexameta-phosphate and 7 g of sodium carbonate in 1000 cm³ of distilled water) were used (ISO 11277:2020, 2020). For the impact of sample preparation and pretreatment (2nd stage), fresh soils were sampled, and a combination of different soil preparations (drying) and pretreatments (dispersion) were used (Table 2). Preparation and pretreatment variants were used only with LDM.

The physical (ultrasound) dispersion was done by built-in ultrasound

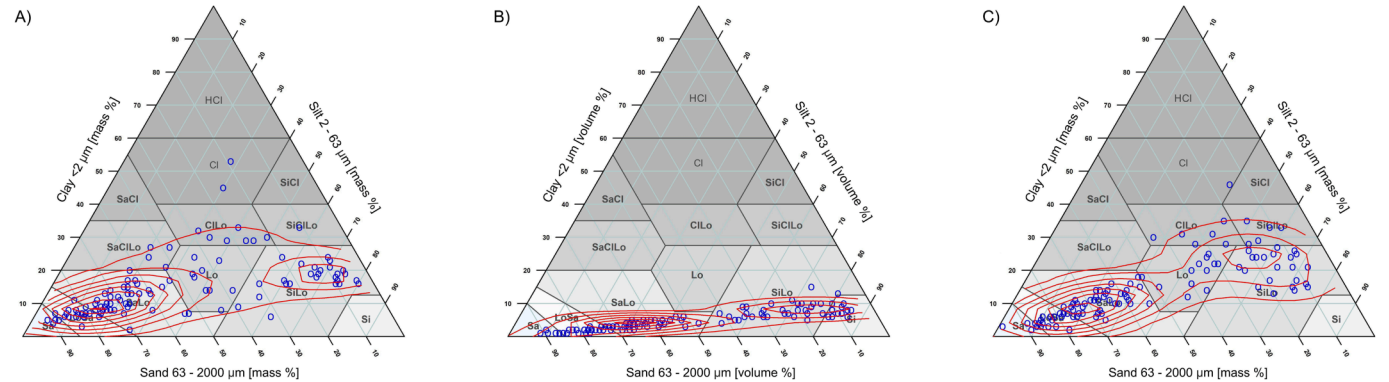


Fig. 1. The Ferret's Triangles for 116 soils investigated in the first stage. PSD measured by: A) SPM; B) LDM; C) adjusted LDM (LDMadj). Abbreviations for the textural classes are: HCl – heavy clay, Cl – clay, ClLo – clay loam, L – loam, LoSa – loamy sand; Sa – sand; SaCl – sandy clay, SaClLo – sandy clay loam, SaLo – sandy loam; Si – silt, SiLo – silt loam, SiCl – silty clay, SiClLo – silty clay loam. Contour lines denote the points with the same occurrence frequency.

Table 2

Combinations of soil samples preparation and pretreatment in the sample preparation and pretreatment impact stage.

Soil	Drying method	Dispersion method
Fresh*	N/A	Chemical (calgon) Physical (ultrasound)
Dried	Air drying 105 °C drying	Chemical (calgon) Physical (ultrasound) Chemical (calgon) Physical (ultrasound)

*Fresh soil – the measurement was carried out on the day following the collection at the latest. If the measurement was on the following day, the sample was stored in an airtight plastic bag to avoid evaporation.

probe in the sample dispersion unit of a laser diffractometer, in which maximum power (35 W) was used for 4 min (Polakowski et al., 2021). The calgon solution used for the chemical dispersion was the same as described in the pedotransfer function validation (1st stage).

2.3. PSD measurements

PSDs of all soils in the pedotransfer function validation stage (1st stage) were measured by two methods: SPM and LDM. In the sample preparation and pretreatment stage (2nd stage), LDM was used.

SPM was used according to the ISO standard (ISO 11277:2020, 2020) – 0.063–2 mm fraction was analyzed by sieving and <0.063 mm, by pipette. In the case of LDM, the fractions were not separated, and thus all fractions <2 mm were analyzed by the laser diffraction methodology. A Mastersizer 2000 (Malvern, UK) with Hydro G dispersion unit was used for LDM measurements with the following settings: i) the light intensity from two sources (red and blue light), ii) Mie mathematical model for calculations, iii) refractive index (RI) 1.52, iv) absorption index (AI) 0.1, v) dispersant (water) refractive index RI = 1.33, vi) measurement time 60 s (30 s for red light and 30 s for blue light), vii) five laser diffraction spectra measurements for one replication, viii) three physical replications (physical replication means a one-time addition of soil to the measuring system), ix) stirrer speed – 700 rpm, and x) pump speed – 1750 rpm (Bieganski et al., 2018). PSD results were calculated using the Malvern Mastersizer 2000 software ver. 5.61. Organic matter was removed in the case of SPM measurements and was not removed in the LDM measurements. The decision to not remove the organic matter in LDM measuring was due to two facts: i) the content of these substances was relatively low; ii) the suspended soils were intensively mixed and pumped (Bartmiński et al., 2011). Also, previous experience (Makó et al., 2017) shows that ultrasound dispersion is sufficient to disrupt aggregates adhered to by organic matter and its removal impact on PSD in low and medium-carbon soils is generally minor (Koza et al., 2021).

2.4. Pedotransfer function to recalculate measured LDM to predicted SPM texture

For converting PSD obtained by measured LDM into predicted SPM PSD, an approach suggested by Makó et al. (2017) was used. Makó et al. (2017) proposed several pedotransfer functions, but since the differences between them were low (~55 to ~58 % agreement with SPM), it was decided to use the simplest ones. The rationale for this approach was a desire to validate the equations when additional soil properties are not known and thus to test the method in the “worst” case scenario on as wide a sample set as possible.

Normally, texture classification on the basis of the SPM and LDM applies standard boundaries of silt and clay content – i.e., clay <2 µm and silt in the range 2–63 µm (ISO 11277:2020, 2020). Makó et al. (2017) used modified fractions boundaries: clay (<6.6 µm) and silt in the range 6.6–60.3 µm. In the present study, we also applied this type of

fraction size limit change (LDMmod) and then used the empirical equations proposed by Makó et al. (2017) to refine the LDM-SPM correspondence.

$$\text{clay}_{\text{LDMadj}} = 0.69 + 0.92 \times \text{clay}_{\text{LDMmod}} \quad (1)$$

$$\text{silt}_{\text{LDMadj}} = -6.10 + 1.10 \times \text{silt}_{\text{LDMmod}} \quad (2)$$

where:

$\text{clay}_{\text{LDMadj}}$ – adjusted LDM clay content [%].

$\text{clay}_{\text{LDMmod}}$ – modified LDM clay content [%] – clay fraction size limit 6.6 µm.

$\text{silt}_{\text{LDMadj}}$ – adjusted LDM silt content [%].

$\text{silt}_{\text{LDMmod}}$ – modified LDM silt content [%] – silt fraction size range 6.6–60.3 µm.

In order to distinguish between the values obtained directly from LDM and the values converted according to Makó et al. (2017), the converted values are referred to as “LDMadj”.

2.5. Comparison of the soil texture classification in both stages

Classification of the soil texture based on SPM, LDM, and LDMadj was used in the pedotransfer function validation stage. For the sample preparation and pretreatment stage, only soil texture classifications obtained by SPM and LDMadj were compared.

Samples from both datasets (SPM and LDMadj) were assigned to WRB textural classes in R (R Core Team, 2013) using the ‘soiltexture’ package (Moeys, 2018). Textural classes defined using the LDM method were compared with the measured SPM data. The percentage agreement between the textural classes was then evaluated. To specify the individual soil texture class accuracy of LDM-determined textures with SPM method derived textures, a confusion matrix was calculated (Salley et al., 2018). To statistically evaluate the agreement of the soil textures determined by the different methods, four types of indicators were trained based on the confusion matrix, being the overall accuracy (OA), user’s accuracy (UA), producer’s accuracy (PA), and kappa coefficient (κ) indicators. OA represents the absolute number of LDM-derived texture classes that match texture classes according to SPM-derived methods. UA shows the proportion of LDM-derived texture classes that match a given SPM-derived class relative to the total number of that texture class (error of commission). PA is a measure of the proportion of SPM-derived soil texture classes correctly classified by the LDM-derived results relative to the total number of observed points within each SPM-derived texture class (error of omission). κ (kappa) was calculated by taking into account the imbalanced distribution of the classes in the sample and measures the classification accuracy after taking into account the probability of random matching between the 13 texture classes. The kappa index can be calculated from the proportion of correctly classified samples and the probability of random agreement (Congalton and Green, 2008). Its results range from –1 to 1, where values <0.0 indicate poor agreement, values between 0.0 and 0.2 indicate slight agreement, values between 0.2 and 0.4 indicate fair agreement, values between 0.4 and 0.6 indicate moderate agreement, values between 0.6 and 0.8 indicate substantial agreement, and values between 0.8 and 1.0 indicate almost perfect agreement (Landis and Koch, 1977).

The differences between the WRB texture classes were plotted as texture triangles, also using the ‘soiltexture’ package. We used the two-dimensional kernel density estimation procedure in R to visualize and evaluate the structure of the soil texture data (Moeys, 2018). Kernel density estimation approximates the probability density function of a selected variable and is a possible tool for plotting the shape of the distribution (Venables and Ripley, 2002).

Lastly, for comparative reasons, we measured the shift on the Ferret’s Triangle, depending on the recalculation method or preparation/

pretreatment variant. This shift $|\vec{W}|$ was calculated according to the formula:

$$|\vec{W}|[\%] = \sqrt{(clay_a - clay_b)^2 + (silt_a - silt_b)^2 + (sand_a - sand_b)^2} \quad (3)$$

where:

clay_{a,b} – clay fraction content (%) measured with (a) SPM and (b) LDM or LDMadj
 silt_{a,b} – silt fraction content (%) measured with (a) SPM and (b) LDM or LDMadj
 sand_{a,b} – sand fraction content (%) measured with (a) SPM and (b) LDM or LDMadj

2.6. Statistical analysis

The results were statistically processed using Statistica 13.3. The normality of results was tested using the Shapiro–Wilk test. One-way ANOVA, Tukey HSD post hoc test, and two-way ANOVA were used to check the significance of the soil pretreatment differences. For non-Gaussian results, the non-parametric Kruskal–Wallis H test was used.

3. Results

3.1. Validation of pedotransfer function (1st stage)

The first issue which should be presented in the context of the aim of this work is the direct comparison of soil texture classification obtained by SPM and LDM (Fig. 1A and B) for all 116 soils. It can be seen from this figure that the clay fraction content was substantially lower for LDM. Generally, the highest clay content measured by LDM was 15 %, while the highest content measured by SPM was 53 %. The results of LDM texture classification after recalculating clay and silt fractions according to Makó et al. (2017) (i.e., LDMadj) are presented in Fig. 1C. It can be seen that the general distribution of points on the triangle obtained using the SPM and the LDMadj method were similar. Kernel density contours showing the frequency of occurrence of the samples indicate that, in both cases, the majority of the samples are found in the triangular sand, loamy sand, and sandy loam fields. Another common

“densification field” is the group of loamy silt textured soils. The texture of soils with the highest clay (Fig. 1A – Cl area) content could not be identified by the LDMadj PSD method (Fig. 1C – Cl area) in the same way as by the SPM method.

As mentioned previously, according to numerous authors, the biggest problem with LDM results is with the content of measured clay fraction (i.e., clay underestimation and as a result, silt overestimation by LDM in comparison with SPM). This opinion was confirmed in Fig. 1B. Therefore, in Fig. 2 the clay fraction content is compared between SPM vs LDM and SPM vs LDMadj.

As could be expected, LDM in general underestimated the clay fraction content. This is confirmed by the large value (~2.8) of the slope for the results constituting the comparison between SPM and LDM (Fig. 2). However, an interpolated line in the relationship between the clay content obtained by the SPM in relation to the content of this fraction obtained with LDMadj (Fig. 2), showed a slope close to the 1:1 line (~0.95) with a determination coefficient of 0.73.

Comparing the results between LDM and SPM for all 116 soils, ~57 % (66 out of 116) were correctly classified (Fig. 1B). The largest number of correctly classified soils belonged to the loamy sand (LoSa) and sandy loam (SaLo) texture groups. No soil belonging to the clay loam (ClLo), clay (Cl), sandy clay loam (SaClLo), loam (Lo), or silt clay loam (SiClLo) texture groups was correctly classified. A shift in the clay-silt boundary to 6.6 µm resulted in an increase in the content of the clay fraction. As a result, the percentage of correct classification increased to ~74 % (86 out of 116) when LDMadj was compared with SPM (Fig. 1C), but still no samples belonging to sandy clay loam or silt clay loam were correctly classified. The differences in the classification made by LDM and LDMadj for incorrectly classified soils (in relation to SPM) are shown in Fig. 3. It can be seen that in the case of LDM (not modified – Fig. 3A), the shifting was greater than when LDMadj was used for classification (Fig. 3B) and only a shift to the nearest neighboring texture group occurred. Also, a jump by two texture groups only took place in five cases for LDM (Fig. 3A).

In the case of LDM, the trend of the shifting was clearly visible – most shifts (35) were towards the lower right corner of the triangle (Fig. 3A), i.e., the increase of silt fraction was observed. Such a trend was not observed when LDMadj was used for soil classifications, however, more shifting was observed horizontally (right – 11; left – 8) than vertically (down – 6; up – 5) (Fig. 3B).

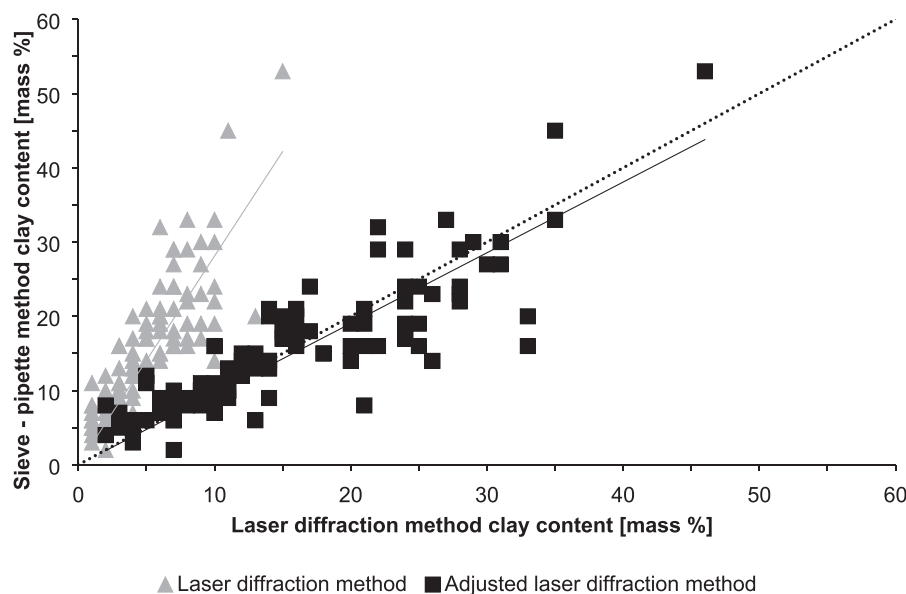


Fig. 2. The comparison of the clay content measured by SPM, LDM and LDMadj methods in all investigated soils. The trend lines were entered, forcing the line to pass through the origin of the coordinate system. The dotted line indicates the ratio of 1:1. SPM – sieve-pipette method; LDM – laser diffraction method.

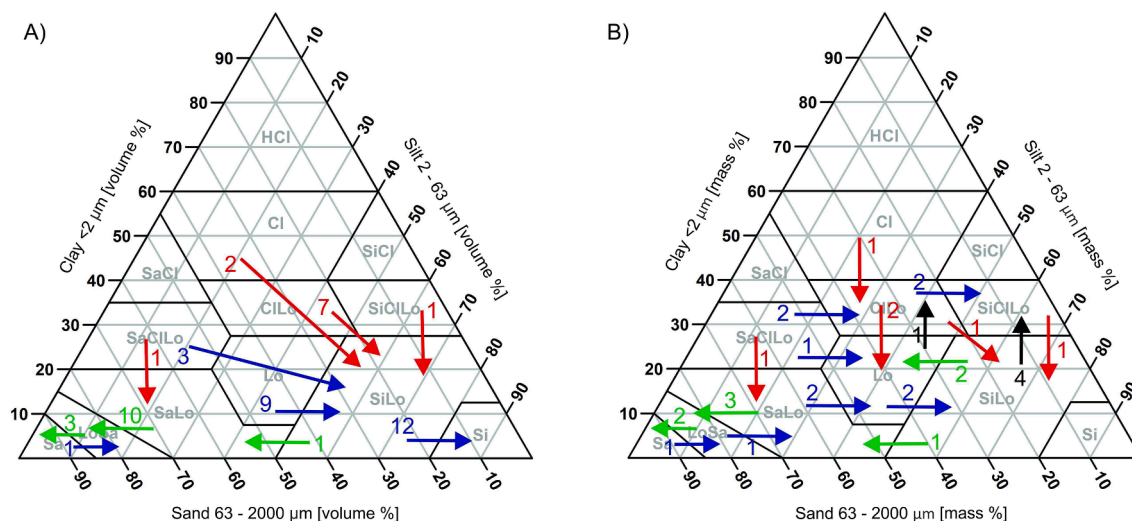


Fig. 3. Comparison of soil classification between SPM (beginning of the arrow) and A) LDM and B) LDMadj (end of the arrow). Color of the arrows denotes: red – shifting of the classification down the Ferret's Triangle; blue – right; green – left; black – up. The numbers represent the number of times the shift occurred. Total number of shifts: A) 50; B) 30.

The above information on the shifts shown in Fig. 3 should be considered rather qualitative. The distribution of shift values, for LDM and LDMadj as compared with SPM, calculated from Eq. (3) gives a quantitative measure. Thus, in Fig. 4 (all 116 soils) it can be seen that both the mean shift and its standard deviation were smaller for LDMadj. The difference was statistically significant (Kruskal-Wallis H test, $p < 0.0001$).

3.2. Influence of sample pretreatment on soil texture classification (2nd stage)

The soils selected for the second stage represent a relatively wide range of TOC and IC contents (Table 1). The influence of soil preparation and pretreatment on texture classification is presented in Table 3. It can be seen in this table that the texture classification was the same when PSD was measured by SPM in only 6 soil samples (out of 16) and by all variants of soil preparations and pretreatments measured by LDMadj. Of the remaining cases, the least number of differences relative to SPM were observed when fresh soils dispersed by ultrasound were tested (5 cases). A slightly worse result was observed for air dried soil with ultrasounds (6 cases). For the other soil pretreatments, the number of differences was bigger (from 7 to 9). Concerning shift distance on the Ferret's triangle in relation to sample pretreatment (Fig. 5), there were no significant differences between tested variants (one-way ANOVA, $p > 0.05$). However, differences in standard deviation of the shifts are clearly visible. The air dried with ultrasound dispersion had the lowest standard deviation, while the highest dispersion was for fresh soil dispersed with ultrasound (Fig. 5).

In order to obtain the answer to which factors (drying, dispersion, or their combination) influenced these observed differences, sample drying and dispersion impact on clay content was tested with two-way ANOVA. Both factors had a significant influence on clay content, i.e., 12/14/13 samples out of 16 for drying/dispersion/drying + dispersion, respectively, differed significantly ($p < 0.05$) by clay content.

A confusion matrix of the differences in classification depending on the soil pretreatment is presented in Table 4. It can be seen that the best match between SPM-based and LDMadj-based texture classification was obtained when fresh soil after ultrasound disaggregation was measured (the highest value of OA = 69 % and $\kappa = 0.63$) and slightly worse results were seen for air dried soil, also with ultrasound (OA = 63 %, $\kappa = 0.55$). For all pretreatment cases where chemical dispersion (calgon) was used, the differences in classification were large (lowest OA and κ values). The

accuracy of the texture classification of the LDMadj measurements (what percentage of the texture classes defined by the LDMadj method matched the SPM-based texture classes) was highest for sandy loam, silty loam, and sandy clay loam classes (UA reached 100 % for most treatments). For ultrasound treatments, this accuracy was also generally higher for other texture classes (e.g., fresh and air dried soils at clay texture class: UA = 100 %; at loam texture class: UA = 67 %). The appropriateness of the classification into texture classes measured by the SPM method (i.e., the proportion of members of each texture class defined by the SPM method that could be classified into a similar class based on LDMadj measurements), however, was highest for sandy loam and loam and silty loam texture classes (PA = 50–100 %). In these classes, there is little to distinguish between the effectiveness of ultrasound and calgon pretreatment. However, for heavier textured soils (clay loam, clay), the agreement is clearly better for ultrasound (PA = 33–100 %) than for calgon pretreatment (PA = 0–33 %).

According to Fig. 1, the clay fraction content is the most sensitive for underestimation in measurements by LDM – which is shown by kernel density on Fig. 1A and B. Therefore, the comparison of clay fraction content for all soils used in the 2nd stage, depending on the soil preparation and pretreatment, related to the results from SPM (dotted lines) was performed (Fig. 6).

The analysis of Fig. 6 leads to the conclusion that the correction of LDM (LDMadj) eliminates the problem of clay fraction underestimation. In 4 cases (1,5,6,9), the content of clay fraction measured by LDMadj was higher than for SPM for all pretreatments. These samples are soils with low organic carbon content (Table 1) and thereby poorer aggregate stability, where the LDM method has relatively easily released the clay fraction from the aggregates. In a further 4 cases (8,13,14,16), LDMadj generally showed lower clay fraction content than SPM for all pretreatments. The remaining samples showed approximately similar clay content by LDMadj and SPM, with variation depending on the preparation and pretreatment.

In general, in most cases, ultrasound treatment was the most effective in disrupting soil aggregates and releasing most of the clay fractions. Also, in most cases, fresh soils had the highest number of clay particles that could be released and measured by the LDMadj, with the lowest proportions of clay particles measured from 105 °C dried soils.

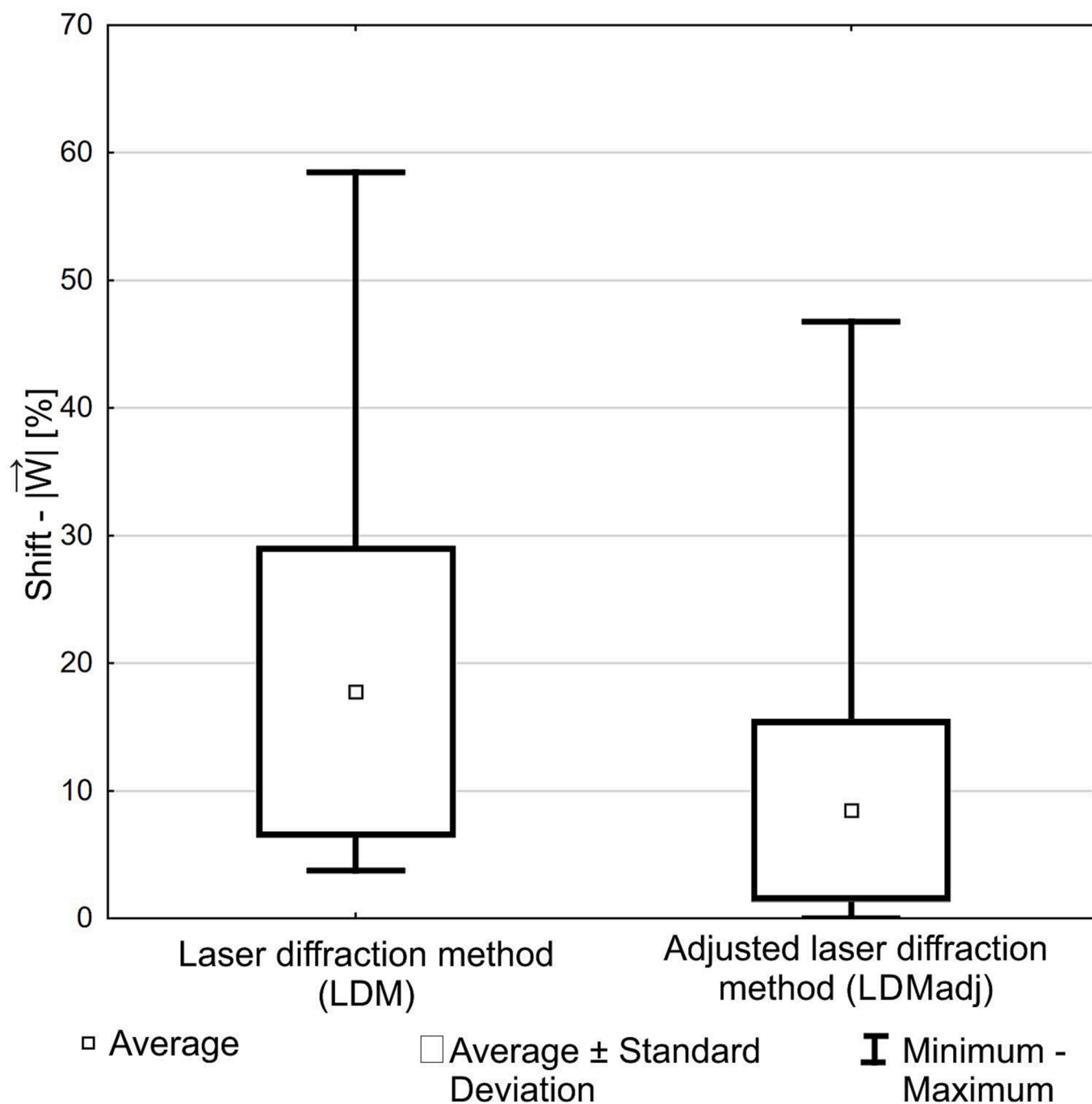


Fig. 4. Comparison of shift length $|\vec{W}|$ on the Ferret's Triangle according to different LDM results calculation methods. LDM – laser diffraction; LDMadj – adjusted laser diffraction method. H; p – Kruskal-Wallis H test results.

4. Discussion

4.1. Validation of pedotransfer function in the context of soil texture classification

In the first step, the effectiveness of the pedotransfer function (including the size range modification of clay fraction) proposed by Makó et al. (2017) in the harmonization between LDM and SPM in soil texture classification was tested. Although many pedotransfer functions have been developed in the past, they are rarely tested on independent databases. Based on the 116 soil samples representative of Poland, it can be confirmed that the pedotransfer function suggested by Makó et al.

(2017) performs well in the harmonization of LDM data to SPM results. The distribution of points on the soil texture triangle obtained using the SPM and the LDMadj results were very similar (Fig. 1A and C), and even better than in the original Makó et al. (2017) work (74 % vs ~56 % agreement). Kernel density contours, in both cases, show that the majority of the samples are found in the sand, loamy sand, and sandy loam fields, which corresponds to the soil conditions in Poland, while the group of silt loam textured soils probably represent soils formed on a loess parent material under the influence of water.

When looking for the reasons for the differences in badly classified cases (26 % of all soil samples), first of all, the problem of the clay fraction underestimation should be analyzed, as was reported in the

Table 3

Comparison of the texture classification obtained after different combinations of sample preparation and pretreatment in relation to results obtained by standard SPM. The cases where at least one classification result was different are marked in gray.

Soil No.	SPM (Texture group of soils [‡] according to IUSS - WRB 2015)	LDMadj (Texture group of soils [‡] according to IUSS - WRB 2015)					
		Fresh + C*	Fresh + U*	Air dried + C*	Air dried + U*	105° + C*	105° + U*
1	SiLo	SiLo	SiLo	SiLo	SiLo	SiLo	SiLo
2	SiLo	SiLo	SiLo	SiLo	SiLo	SiLo	SiLo
3	Lo	Lo	Lo	Lo	Lo	Lo	Lo
4	SaLo	SaLo	SaLo	SaLo	SaLo	SaLo	SaLo
5	SaLo	Lo	Lo	SaLo	SaLo	Lo	Lo
6	SiLo	SiLo	SiLo	SiLo	SiLo	SiLo	SiLo
7	SiLo	SiLo	SiClLo	SiLo	SiLo	SiLo	SiLo
8	ClLo	Lo	ClLo	Lo	Lo	Lo	Lo
9	SiLo	SiClLo	SiClLo	Lo	SiClLo	SiClLo	SiClLo
10	SaClLo	SaLo	SaClLo	Lo	ClLo	Lo	SaClLo
11	SaClLo	SaClLo	ClLo	Lo	ClLo	ClLo	ClLo
12	ClLo	ClLo	ClLo	SiLo	SiClLo	SiLo	SiClLo
13	Cl	ClLo	ClLo	Lo	ClLo	Lo	ClLo
14	Cl	ClLo	Cl	ClLo	Cl	ClLo	SiClLo
15	ClLo	Lo	ClLo	Lo	ClLo	Lo	ClLo
16	Lo	Lo	Lo	Lo	Lo	Lo	Lo

* +C – chemical dispersion; +U – ultrasound dispersion.

[‡] Sa, sand; LoSa, loamy sand; SaLo, sandy loam; Lo, loam; SiLo, silt loam; Si, silt; SaClLo, sandy clay loam; ClLo, clay loam; SiClLo, silty clay loam; SaCl, sandy clay; SiCl, silty clay; Cl, clay.

literature (Eshel et al., 2004; Konert and Vandenberghe, 1997; Nimblad Svensson et al., 2022). Based on the data presented in Figs. 2 and 4, it seems reasonable to conclude that the application of the pedotransfer function (Eq's. (1) and (2)) proposed by Makó et al. (2017) solves this problem to a large degree. The differences between the clay fraction content measured by SPM and LDMadj, however, are rather random (Fig. 1C and 3B). In other words, there is no trend similar to the case

when the results from SPM and LDM are compared, and the clay content is clearly underestimated (Fig. 1B and 3A). The reasons for these differences between SPM and LDM can be caused by the uncertainty of both methods (i.e., unfulfilled base method assumptions such as the same particles specific density or spherical shape) (Bieganowski et al., 2018). Also, the equipment model may have an impact on the final SPM and LDM comparison, as results acquired on different equipment models can

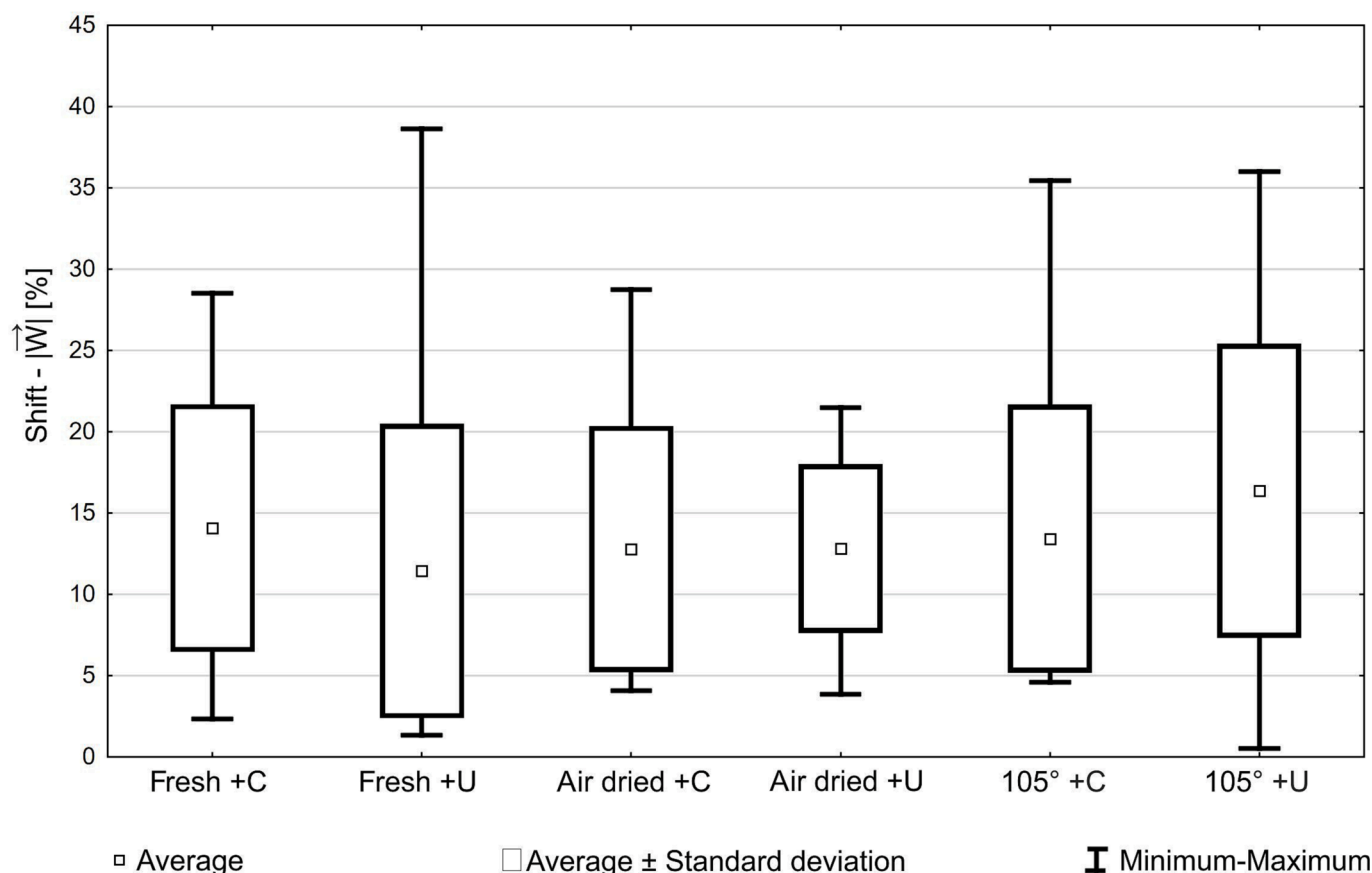


Fig. 5. The comparison of shift length $|W|$ on the Ferret's Triangle according to drying and dispersion variant (all results recalculated according to Makó et al., 2017). +C – chemical dispersion; +U – ultrasound dispersion. F; p – one-way ANOVA F test results.

Table 4

Confusion matrix results of comparative studies to show the individual soil texture class accuracy of LDMadj determined textures with SPM method derived textures.

Pretreatments	N [†]	Soil Texture Classes [‡] Accuracy (UA/PA) * (%)														κ ^{***}
		Sa	LoSa	SaLo	Lo	SiLo	Si	SaClLo	ClLo	SiClLo	SaCl	SiCl	Cl	HCl	OA ^{**}	
Fresh + C x	16	–	–	50/50	40/100	100/80	–	100/50	33/33	–	–	–	-/0	–	56	0.47
Fresh + U x	16	–	–	100/50	67/100	100/60	–	100/50	60/100	–	–	–	100/50	–	69	0.63
Air dried + C x	16	–	–	100/100	25/100	80/80	–	-/0	0/0	–	–	–	-/0	–	50	0.38
Air dried + U x	16	–	–	100/100	67/100	100/80	–	-/0	25/33	–	–	–	100/50	–	63	0.55
105 °C + C x	16	–	–	100/50	29/100	80/80	–	-/0	0/0	–	–	–	-/0	–	44	0.31
105 °C + U x	16	–	–	100/50	50/100	100/80	–	100/50	33/33	–	–	–	-/0	–	56	0.48

[†] Number of soil samples analyzed.

[‡] Sa, sand; LoSa, loamy sand; SaLo, sandy loam; Lo, loam; SiLo, silt loam; Si, silt; SaClLo, sandy clay loam; ClLo, clay loam; SiClLo, silty clay loam; SaCl, sandy clay; SiCl, silty clay; Cl, clay; HCl, heavy clay.

* UA/PA, Users and Producers Accuracy.

** OA, Overall Accuracy is the percent correctly classified.

*** κ, Kappa index.

× +C – chemical dispersion; +U – ultrasound dispersion.

differ (Goossens, 2008) and, therefore, the clay-silt limit may vary between equipment models. More research is, therefore, needed to find out which of these methods has a greater influence on these differences.

The fact that errors in soil texture classification with the use of LDMadj are also random is confirmed in Fig. 3B. The amount of shifting in all directions in the Ferret's Triangle when LDMadj was used is generally equal, while trends were more visible when LDM was used (Fig. 3A).

Our results concerning soil texture classification accuracy on an independent sample base (74 % match) are comparable to most studies on the original datasets. One work that achieved better agreement was

done by Yang et al. (2015). They reported 91 % accuracy in soil texture classification (USDA triangle) after using a pedotransfer function to convert LDM results to SPM-like data developed on 235 soil samples in China. Their method seems to be very effective, but as in every other case, this accuracy was measured on the original dataset, not on an independent database, so the comparison cannot be complete. Validating their procedure would be crucial to find out how universal and applicable the pedotransfer function used in their analysis really is.

Other work has shown lower classification effectiveness, although to a comparable extent. Taubner et al. (2009) classified LDM data into WRB textural classes and compared it to the standard SPM-based texture

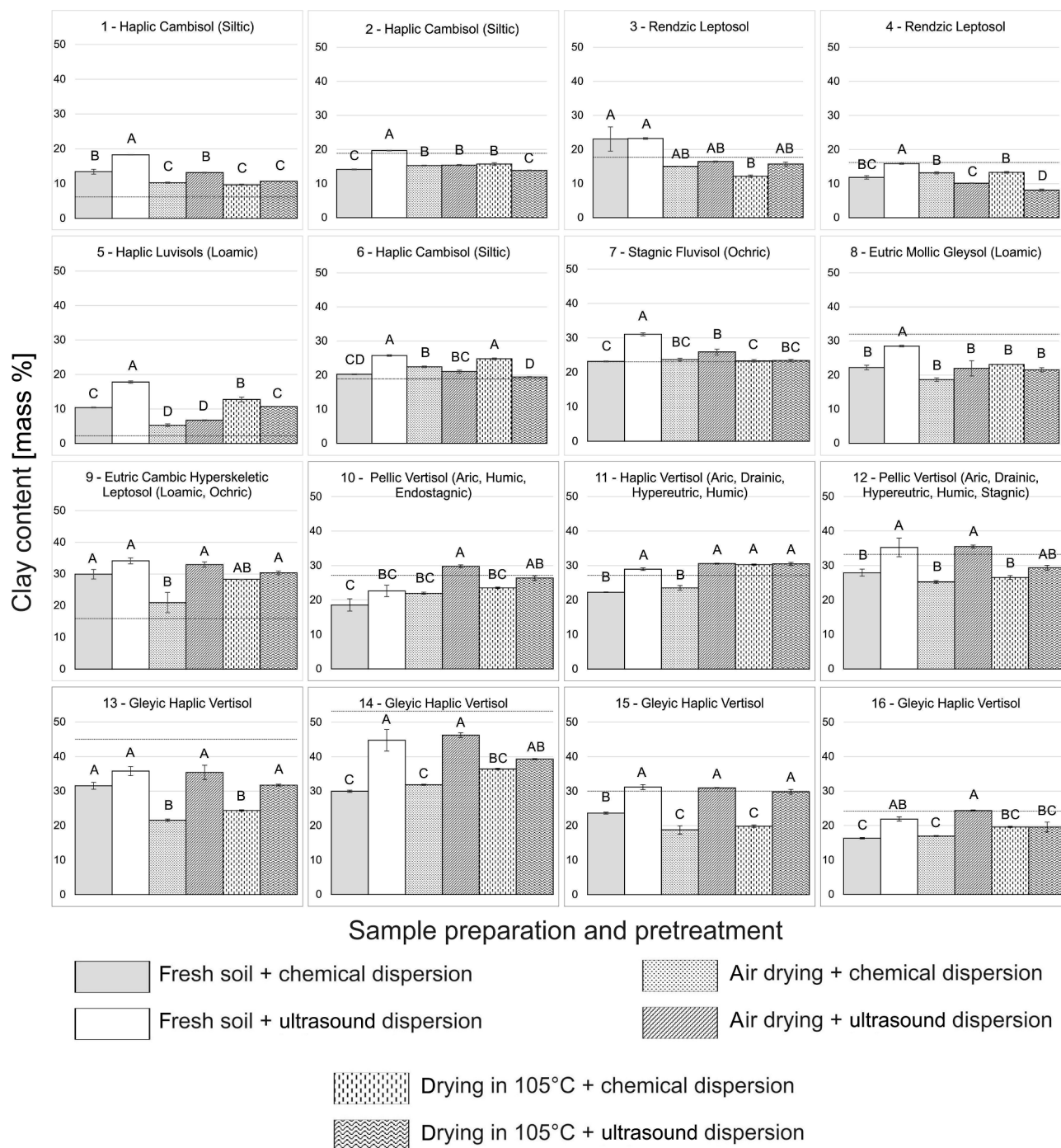


Fig. 6. Clay content in soil samples depending on the soil preparation and pretreatment method, measured by LDMadj (bars) and SPM (dotted line – mass %). The letters denote statistically homogenous groups – the same letters that tested variants did not differ significantly (Tukey HSD test, $p < 0.05$).

classification results of 16 soil samples from Germany. They found growing discrepancies with increasing clay content when comparing the standard texture classes to LDM-based texture classes. Therefore, they also modified the LDM results by transforming the clay content into an “SPM-like” clay content by means of a linear-regression correlation. In the outcome 10 out of 16 samples (62 %) were classified correctly after their modification, which is a little weaker than the results in our study. Based on the extensive study by [Yudina et al. \(2020\)](#) on 315 soil samples from Russia, the most effective way (without using additional soil properties) to use the LDM data to determine the texture classes of the USDA classification was to recalculate the clay content and use the original LDM sand content data. In their study, a logistic function was

applied to approximate the clay content. The original sand content determined by the LDM was used to determine the texture classes. The silt content was calculated as the difference between 100 % and the clay and sand contents. Their approach achieved maximum 63.6 % accuracy in determining correct texture class, and 67.6 % when total carbon content was taken into account.

4.2. Soil sample preparation and pretreatment

It might seem that sample preparation has the same effect on both methods. However, there are two arguments that this is not the case: i) the sensitivity of the LDM is much higher than that of the SPM

(Dobrowolski et al., 2012); ii) the amount of soil taken in the LDM measurement is many times smaller than in SPM. If so – the lack of reproducibility in soil disaggregation may result in greater uncertainty in the result. Therefore, this study wanted to find the preparation and pretreatment methods that gives the most similar result of LDMadj to the standard SPM in texture classification.

4.2.1. Drying

The least number (5 cases out of 16) of differences between LDMadj and standard SPM-based texture classifications were observed when fresh soils were dispersed with ultrasound (Table 3). The possible explanation is that this method gives the highest clay content (Fig. 6) in most samples, decreasing the clay fraction differences between the results of the two methods. Another confirmation of this observation can be seen in Table 4 where the same conclusion can be drawn based on the confusion matrix, and partly in Fig. 5 where, for this pretreatment, the average value of shifting of the points in Ferret's Triangle was the smallest – the latter, however, not significantly and with relatively high standard deviation.

Similar observations were reported in Thompson et al. (1985), claiming that drying causes a decrease in clay porosity, which may hinder the penetration of the dispersant into the aggregates. Hall et al. (2013) claimed that during the oven-drying process, salt crystallization and local changes in hydrophobicity, as well as material transport inside aggregate capillaries, can occur in the pores and on the surfaces of the particles. Therefore, drying soil affects its properties and should be avoided. The opposite claim concerning marine sediments was stated by Jaijel et al. (2021), where oven drying along with peroxide treatment was suggested.

So, the question arises of whether fresh sample analysis should be recommended as a standard procedure. There are, however, several reasons for not recommending this. First of all, it should be stated that the difference in correct (assuming that SPM gives correct results) soil texture classification shift (Eq. (3)) between fresh + ultrasound and air dried + ultrasound dispersion was not statistically significant ($p = 0.37$). This is further confirmed in Table 3 showing a similar number of errors in classification for fresh soil + ultrasound (5) and air dry soil + ultrasound (6). Also, Table 4 shows similar OA for fresh soil + ultrasound (69) and air dry soil + ultrasound (63). Moreover, the scattering (standard deviation in Fig. 5) of the results for fresh + ultrasounds samples was much bigger than for air dry soil + ultrasounds. Therefore, the use of air dry soil + ultrasound would be an acceptable pretreatment procedure despite a slightly larger measurement error than fresh + ultrasound. This is further supported by some practical considerations. If fresh soil is measured, the problem with interlaboratory comparisons would appear and as a result, the error would be bigger. For example, fresh soil means soil with water content at the given moment of field sampling. Soil water content may vary between water-saturated soil (after heavy rain or flood) to nearly air dry soil (after a drought period). Therefore, the fresh soil measures introduce a new, difficult-to-estimate source of errors to the measurement resulting from the different disaggregation efficiency of samples with varying humidity. Moreover, considering the laboratory practice of soil science investigations, it is very difficult to presume that PSD measurements will always be performed immediately after returning from sampling.

4.2.2. Dispersion

Comparing the results of chemical and physical (ultrasound) dispersion in the air dry soil variant, ultrasound dispersion has the lowest shift between LDMadj and SPM (Fig. 5), the highest OA (Table 4), and the highest clay content (Fig. 6). It can also be stated that both TOC and IC content (Table 1) did not influence the differences between the methods.

Analyzing the efficiency of aggregate disaggregation, the factors influencing both physical (ultrasound) and chemical (calgon) procedures should be discussed. Factors influencing disaggregation efficiency

for ultrasound dispersion are: i) differences in the ultrasound power of the probe, ii) differences in the shape and size of the vessel in which the soil suspension is measured, iii) concentration of the suspension, and iv) duration of sonification. These factors influence the amount of energy absorbed by the soil aggregates and, as a result, their disintegration. As these parameters are not standardized, they can be a source of error in PSD measurements using different equipment and laboratories, since the effect of ultrasound power is highly dependent on energy and material. Kaiser et al. (2012) found that above 60 J cm^{-3} , soil organic matter particles can be damaged, and over 700 J cm^{-3} , primary mineral particles of sand or silt size can be damaged. However, this seems not to be the case in commonly used LDM systems, and the problem of different efficiency of disaggregation will persist because of lack of standardization.

In the case of chemical procedures, the factors influencing the disaggregation efficiency are i) different solutions with various dispersants, ii) differences in their manner, and/or iii) duration of application. For instance, some authors use calgon as described by ISO 11277:2020 (2020) (Makó et al., 2017) or sodium pyrophosphate (Tyugai et al., 2010). Moreover, dispersion can be carried out in different ways: in a separate vessel according to ISO 11277:2020 (2020) (Panuska et al., 2008), or calgon can be added to the soil suspension directly in the dispersion unit of the laser diffractometer, as in the present study, and by Makó et al. (2017). An additional important issue is the time interval between the moment of dispersant addition and the start of the measurement.

Abdulkarim et al. (2021) compared different chemical dispersing agents and found that calgon (sodium hexametaphosphate + sodium carbonate) was the most effective of all chemical methods because it resulted in the highest clay content measured. However, based on our test results, physical (ultrasound) dispersion seems to be even more effective than calgon regarding the disaggregation of clay. Abdulkarim et al. (2021) also found that dispersion efficiency is strongly dependent on the soil type, which is confirmed in our data, especially in the case of the Haplic Vertisol (Gleyic) which has high SPM clay content samples, and where it seems that ultrasound dispersion results in significantly higher clay content compared to calgon pretreatment (Fig. 6). However, our study did not find any trend concerning soil type impact on clay content either in different drying or dispersion procedures (Fig. 6).

Tan et al. (2017) showed that clay minerals reacted differently to both chemical and physical dispersion; ultrasound dispersion always yielded the smallest particle sizes, whereas the effect of chemical dispersion varied between the minerals. Shein et al. (2006) and Tyugai et al. (2010) observed the presence of stable cores of microaggregates in chernozem samples; in this case, ultrasound dispersion was insufficient and sodium pyrophosphate treatment was necessary for dispersion. On the other hand, Tan et al. (2017) also found that for some clay minerals, chemical dispersion was not sufficient to change the charge of the particles, concluding that ultrasound dispersion was better suited for such samples. Moreover, they found that Ca^{2+} -containing minerals can become temporarily larger when treated with Na^+ -based dispersants and finally exfoliate into smaller particles because of cation exchange with both the dispersant and the solvent (e.g., water). Shein (2009) observed an even stronger effect – Ca^{2+} ions formed persistent, insoluble crystals. Finally, dispersion with pyrophosphate can be temporary and subsequent flocculation may occur (Gorączko and Topoliński, 2020).

Based on the results in our study, ultrasound dispersion along with air drying should be recommended as a compromise between theoretical assumptions, laboratory practice, and SPM comparison issues. Obviously, such a procedure is biased with an error that is difficult to estimate. However, if this procedure is consistently followed, all measurements would be biased by this systematic error and, therefore, the results would be comparable.

5. Conclusions

The differences in the particle size distributions obtained by sieve-pipette method and laser diffractometry influence the differences in soil classification. One way to solve this problem is to use pedotransfer functions.

Using the pedotransfer functions proposed by Makó et al. (2017), which were based on a modified clay-silt limit, this study achieved 74 % accuracy. To the best of the authors' knowledge, it is the only study that reflects the performance of a pedotransfer function developed on an independent database.

In light of the results, it seems that all drying and dispersion methods pose methodological problems and choosing which one to use is rather a matter of SPM comparison efficiency and balancing between theory and practice. Therefore, the recommendation of this study is to use air dry soil with ultrasound as the sample pretreatment in LDM measurements of PSD because it is a reasonable compromise between theoretical and practical requirements.

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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