The effect of different water types commonly applied during laser diffraction measurement on the particle size distribution of soils

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Abstract

It is expected in the future that soil particle size distribution (PSD) measurements by laser diffraction method (LDM) may replace sieve-pipette sedimentation methods (SPM) as they are faster, require less sample, and are accurate and reproducible. LDM measurement result is a continuous function of PSD, which can facilitate the conversion between the various limits (by countries, by scientific field) of the calculated particle size fractions (PSF – e.g. clay, silt, sand). Currently, there is no standard method for LDM PSD measurement. Many different types of instruments and preparation devices are currently used in laboratories, with various sample preparation, pre-treatment and measurement methods (duration, chemical and/or mechanical dispersion, settings, etc.). In soil LDM PSD measurements, researchers put relatively little emphasis on the choice of the type of aqueous media used. Thus, it is still questionable to what extent the results of the LDM measurement depend on the selection of the dispersion method and the aqueous media. For our research, eight soil samples with various physical and chemical properties were collected in Hungary. The particle size fractions (clay, silt, sand) determined with LDM (Malvern Mastersizer 3000) measured in three types of aqueous media (distilled, deionized and tap water), in different combinations of two dispersion methods (no treatment, ultrasonic or chemical dispersion with Calgon and their combination) were compared. For the comparison, PSF results of the conventional sieve pipette method (SPM) were used as a reference. Our results showed that LDM measurement can achieve various degrees of dispersion with different preparations, in many cases only partial dispersion, disaggregation, sometimes re-aggregation, and flocculation of soil particles were observed as compared to full preparation (in SPM). The "disaggregation pattern" of the soil samples also depended on the quality of the aqueous media and the properties of the soil investigated, because several types and degrees of interactions could occur in the various soil-liquid-dispersant/disaggregation effect systems.

Keywords: pre-treatment, ultrasound, soil texture classification, aqueous media

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Introduction

The particle size distribution (PSD) of soils can be considered one of the most important properties of soils. It may influence many other soil properties (e.g. texture, structure, porosity, ion, molecule adsorption/desorption and nutrient cycling, microbiological activity, hydrological properties, etc.) and is also an important diagnostic criterion in soil classification.

Determining the PSD is an important research field in various industrial applications (pharmacy, ceramic industry, etc.) and also for the earth and environmental sciences (Blott, S.J. and Pye, K. 2008; Varga Gy. *et al.* 2019; Gresina, F. 2020; Polakowski, C. *et al.* 2023).

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Many methods are used by various fields of soil science and a wide range of related sciences for the determination of PSD (such as, for example, sieve-pipette method, aerometer method, PARIO or dynamic image analysis in geography, geochemistry, soil mechanics, hydrology (Gee, G.W. and Bauder, J.W. 1986; Miller, M.P. *et al.* 1988; Allen, T.A. 1990; Clifton, J. *et al.* 1999; Loveland, P.J. and Whalley, W.R. 2001; Nemes, A. *et al.* 2002). These methods differ in that they propose various sample preparation and pre-treatment steps (e.g. removal of organic matter, possibly carbonates and sesquioxides; applying ultrasound and/or dispersing agents) while using different instruments, measurement procedures. The so-called sedimentation methods (e.g. pipette and hydrometer methods) commonly require a lot of manual labour. To eliminate the human error factor and for automation, various instrumental measurement techniques such as the laser diffraction method (LDM) or particle counting, optical sensing were developed at the end of last century (Syvitski, J.P.M. 1991). LDM is now increasingly used to determine the PSD of soils (Pye, K. and Blott, S.J. 2004; Kovács, J. 2008; Ryżak, M. and Bieganowski, A. 2011; Sochan, A. *et al.* 2012; Yang, X. *et al.* 2015).

The advantage of LDM is that it is fast (3–10 min/sample on average), accurate and provides high reproducible measurement results. It gives the result as a continuous function compared to sedimentation methods (e.g. sieve-pipette, hydrometer) (e.g. Konert, M. and Vandenberghe, J. 1997; Buurman, P. *et al.* 2001; Gresina, F. 2020; Igaz, D. *et al.* 2020). However, we do not yet have sufficient experience in comparability and convertibility of the results obtained with various commonly used sedimentation and LDM measurement methods (Ryżak, M. and Bieganowski, A. 2011; Makó, A. *et al.* 2019; Svensson, D.N. *et al.* 2022).

The currently known major drawback of LDM is that it is limited in its ability to take into account the irregular shape or various optical parameters of sediment or soil particles, which may affect LDM results (Varga, Gy. *et al.* 2015; Bieganowski, A. *et al.* 2018;

Varga, Gy. *et al.* 2022). The disadvantage is also that it cannot distinguish between the light diffraction of elementary particles and soil aggregates. This is the basis, for example, for LDM measurements of soil aggregate size distribution (ASD), mainly without pre-treatment (e.g. Kubínová, R. *et al.* 2021; Polakowski, C. *et al.* 2021a). Furthermore, the stability of aggregates can also be determined by LDM methods from the ratio of these size fractions or its changes during treatment with time (with the amount of disaggregation effects/forces (by dispersants and/or ultrasound – e.g. Rengasamy, P. *et al.* 1984; Field, D.J. and Minasny, B. 1999; Mason, A. *et al.* 2011), with applying fluids with various polarity (e.g. Mamedov, A.I. *et al.* 2007) or other statistical indices calculated based on the change of PSD curves (Bieganowski, A. *et al.* 2018).

According to the literature, deviations might be observed between results of various LDM PSD measurements. The results of LDM can be influenced by a number of factors. These can be grouped into several large categories: method of sample pre-treatment and preparation; the type of device used; the type of optical model chosen; the operation of the data processing software; operator-dependent settings; the suitability of the sample to be examined in terms of LDM (Makó, A. *et al.* 2017a; Bieganowski, A. *et al.* 2018; Varga, Gy. *et al.* 2019).

Based on a summary of Bieganowski, A. *et al.* (2018) on earth and soil science literature, preparations (removal of binding agents, such as organic matter, lime, iron oxides) or pre-treatments, chemical (adding NaHMP, Calgon, alkaline salts etc.) or physical dispersion methods (slaking or only stirring and circulation and/or use of ultrasound of different intensities) all influence the accuracy and repeatability of the measurement. In practice, the preparation methods traditionally used in sedimentation measurements (total or partial removal of adhesive materials) are omitted or inconsistently used in LDM PSD measurements due to their lengthy nature. It is assumed that disaggregation of soil samples also occurs

during physical and/or chemical dispersion (e.g. Bartminski, P. *et al.* 2011). Ultrasonic dispersion, as a mechanical dispersion method, is widely used for LDM to achieve aggregate disintegration without damaging the primary particles (Raine, S.R and So, H.B. 1994). In most cases, mechanical and chemical dispersion are used simultaneously in LDM PSD measurements (Buurman, P. *et al.* 1997; Chappel, A. 1998; Ryżak, M. and Bieganowski, A. 2011; Virto, I. *et al.* 2011; Madarász, B. *et al.* 2012; Makó. A. *et al.* 2019; Polakowski, C. *et al.* 2023).

Some literature concerning ASD and aggregate stability measurement with LDM (or other methods) shows that the effect of soil properties on aggregate stability is complex, the resistance of soil samples to disaggregation effects may also differ in PSD measurements. Various effects of different soil properties can be dominant in the formation of aggregates, thus, also in the dispersion and disaggregation of soil samples if the investigated soils are rich in organic matter (e.g. Tyugai, Z. *et al.* 2010; Virto, I. *et al.* 2011; Schulte, P. *et al.* 2016), or has high carbonate (e.g. Virto, I. *et al.* 2011), Fe/Al-(oxi)-hydroxide (Zhao, J. *et al.* 2017), gypsum content (e.g. Pearson, M.J. *et al.* 2015), are hard clay soils (Rengasamy, P. *et al.* 1984) or soils of arid, semiarid regions (e.g. Amézketa, E. *et al.* 2003; Shein, E.V. *et al.* 2013; Goosens, D. *et al.* 2014). Since soils are very heterogeneous and complex materials, we do not have sufficient knowledge on how certain pre-treatments can affect the results of LDM PSD measurement with a specific combination of chemical and physical properties.

A simple parameter, such as the quality of aqueous media used in the measurement, may have an impact on the results. Dispersing media with different ion compositions can be used to examine, for example, the effect of the quality of irrigation water on the aggregate stability of dispersive soils and/or soil with high Na^+ and total salt content soils (e.g. Amézketa, E. *et al.* 2003; Almajmaie, A. *et al.* 2017). Manufacturers of laser diffractometers (e.g. Beckman-Coulter, Bettersizer, Fritsch, Horiba, Malvern) provide

various general recommendations regarding the choice of water type for LDM measurements. Malvern, for example, does not recommend connecting a laser diffractometer to a tap water network because the high pressure of water and the sudden temperature change can cause bubbles to form, which the instrument can identify as particles during measurement (Malvern User's Manual). According to the Fritsch laser diffractometer manual, "normal" tap water is perfectly suitable for general purpose measurements (no mention is made of water chemistry or mechanical purity parameters), but it already points out that in some cases, it may be necessary to use distilled water (depending on the properties of the samples to be tested) (Fritsch User's Manual). No manufacturer provides specific recommendations on the water quality required for testing soil samples.

Several LDM experiments published in the literature have used distilled or deionized water to determine the PSD of soils (Ryżak, M. and Bieganowski, A. 2011; Sochan, A. *et al.* 2012; Kovács, J. *et al.* 2013; Varga, Gy. *et al.* 2016). However, there are also numerous publications in which authors have used tap water for their LDM PSD measurements in the case of sandy samples (Chappell, A. 1998; Storti, F. and Balsamo, F. 2010; Messing, I. *et al.* 2024) or heterogeneous textured soil or sediment samples (Ferro, V. and Mirabile, S. 2009; Özer, M. and Orhan, M. 2015; Abdulkarim, M. *et al.* 2021; Parent, E.J. *et al.* 2021). However, it is still questionable how the results of LDM PSD measurements can be affected by the chemical and physical properties of the soil samples, the types of pre-treatment, their combinations and the properties of the aqueous media used, and how these interact with each other.

Thus, the purpose of our research was to investigate to what extent influence the method of dispersion (chemical dispersion with Calgon or mechanical dispersion by ultrasound) and the quality of the chosen aqueous media (distilled, deionized and tap water) the clay/silt/sand contents of soil samples with different physical, chemical and mineralogical characteristics, calculated from LDM PSD measurements.

Materials and methods

Soil samples were collected from eight sites in Hungary (*Figure 1, Table 1)*. The samples differed in their physical, chemical and mineralogical properties and were representative of the genetic horizons of the most characteristic Hungarian soil types.

Sample 1 (S1) and 8 (S8) contained predominantly smectite-type swelling clay minerals with high adsorption capacity. Sample 6 (S6) had high goethite content (10%) and their swelling clay content is dominated by vermiculite.

Samples 2 (S2), 3 (S3), 4 (S4), 5 (S5) were characterized by chlorite/vermiculite intercalation; they had lower adsorption capacity. Soil sample 7 (S7) contained few swelling clay minerals (dominated by illite and chlorite) and had the highest calcite content (10%).

Basic soil properties were measured according to the appropriate Hungarian standards (see detailed in Barna, Gy. *et al.* 2015). Macro-aggregate stability (WSA) was determined with an Eijkelkamp wet sieving device (Kemper, W.D. and Rosenau, R.C. 1986). LDM measurements were conducted on air-dried soil samples, that were sieved

(< 2 mm) and cleaned from macroscopic plant debris (Barna, Gy. *et al.* 2015). Since in practice, in most cases, the removal of binding agents (carbonates, organic matter, and/or Fe-hidroxides/oxyhidroxides that hold aggregates together) was neglected, or inconsistently applied, typically for the purpose of "speed advantage" of LDM PSD measurement, we conducted our measurements without this kind of preparation steps.

LDM PSD determination was performed by Malvern Mastersizer 3000 device with Hydro LV dispersion unit. The effects of the type of aqueous media and treatments on the clay, silt and sand content of soils with various chemical and physical properties were investigated in matrix type treatment combinations using the method of Makó, A. *et al.* (2017b) and Polakowski, C. *et al.* (2021b). Distilled water (DW), high purity commercially available deionized water (DIW) or tap water (TW) was used as aqueous media *(Table 2)*. Four types of physical and/or chemical dispersion were applied as pre-treatment: no treatment (T1); Calgon only (T2) or ultrasound only (T3) and their combination (T4). The Calgon solution was prepared according to ISO 11277:2009(E)
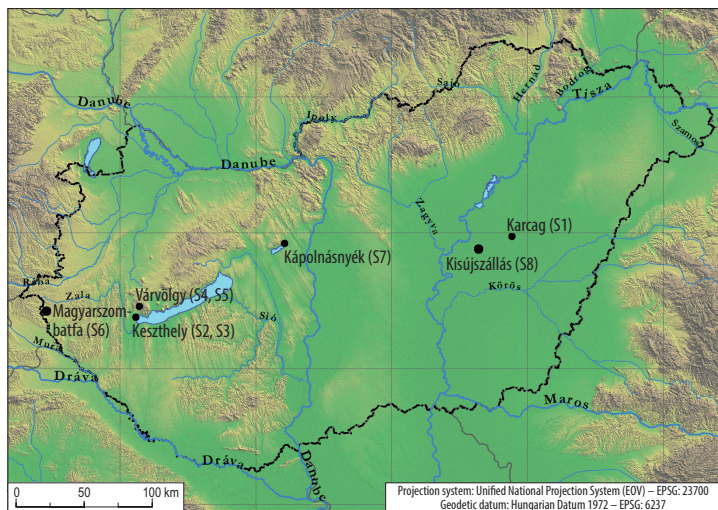

Fig. 1. Geographical positon of the eight sample sites (S1–S8) with the name of nearest settlement.

Table 1. Important physical and chemical characteristics of the soils included in the study

Sample code	WRB soil classification	Symbol and depth of genetic horizons, cm	Clay + Fe-oxihydrates, % (< 0.002 mm)	Silt, % (0.002–0.05 mm)	Sand, % (> 0.05 mm)	SOM, %	CaCO$_3$ %	CEC, mgEq/100 g	Exchangeable Na$^+$, mgEq/100 g	Water stable aggregates, WSA %
				SPM PSD,[*] solid part of the soil (100%)						
Sample sites **S1–S8**: see *Figure 1*										
S1	Vertic Stagnic Solonetz (Clayic) *Karcag***	**B** 5–30	51.09	45.90	0.88	2.00	0.13	40.85	20.63	20.84
S2	Hortic Terric Cambisol (Dystric, Siltic) *Keszthely***	**A** 0–30	21.09	33.13	44.28	1.45	0.05	11.84	0.14	53.40
S3	Hortic Terric Cambisol ** (Dystric, Siltic) *Keszthely*	**B** 30–50	22.90	33.87	42.29	0.93	0.00	12.38	0.13	38.47
S4	Cutanic Luvisol (Siltic) *Várvölgy***	**A** 0–20	15.27	29.35	54.05	1.33	0.00	10.36	0.12	87.57
S5	Cutanic Luvisol (Siltic) *Várvölgy***	**B** 20–50	22.30	26.56	50.49	0.65	0.00	12.78	0.15	38.38
S6	Vertic Gleyic Luvisol (Manganiferric, Siltic) *Magyarszombatfa***	**B** 20–50	38.96	25.93	34.61	0.49	0.00	16.78	0.17	44.41
S7	Vermic Calcic Chernozem (Anthric, Siltic) *Kápolnásnyék***	**A** 0–30	27.60	51.68	7.50	3.70	9.52	30.25	0.25	64.56
S8	Gleyic Vertisol (Clayic) *Kisújszállás***	**A** 0–30	53.88	41.19	1.05	3.89	0.00	35.69	0.29	59.14

[*]SPM PSD: Particle size distribution measured according to the conventional standardized sieve-pipette method ISO 11277: 2009 (E). ** The name of the *nearest settlement*.

(mixture of 33 g of Na-hexametaphosphate and 7 g of anhydrous Na-carbonate L^{-1}). Based on our own previous experience, the pumping and stirring speed during the measurements was 2750 rpm (Makó, A. *et al.* 2022). Prior to all the measurements, the system was degassed. The mass of soil samples added to the dispersing unit varied between 0.5 and 1 g, with obscuration ranging between 5 and 20 per cent. In T2 and T4, 2 cm^3 of Calgon solution was added as a dispersant to the air-dried samples on a watch glass, mixed gently with a glass rod, washed into the dispersion unit without residue, than another 25 cm^3 of Calgon solution was added to the soil suspension in the dispersion unit. The ultrasound was operated at maximum

(100%) power, which was 40 W (frequency: 40 kHz), in the T3 and the T4. The ultrasound time was 240 seconds. During the measurements, the tank was operating with 100 percent volumetric efficiency (V = 600 cm^3) based on the level sensor setting.

The PSD of each soil sample was determined in three sample repetitions and in 3–5 replicates per sample. The light scattering data measured by the detectors were converted with Malvern software into PSD results based on the Mie theory. During the measurement, the following optical settings were used: absorption index (AI) = 0.1; solid phase refractive index (RI) = 1.52; and water refractive index (RI) = 1.33. Particle size fractions (PSF) were determined from the results

Table 2. Properties of the aqueous environments used during LDM measurement

Aqueous medium	pH	EC, μS/cm	Na$^+$	K+	Ca^{2+}	Mg^{2+}	Total hardness, mg/l CaO
			ppm				
Distilled water	6.4	2.2	0	0	0	0	0
Deionized water	6.1	1.5	0	0	0	0	0
Tap water	7.7	530.0	26	2.6	67	17	136

of PSD functions. The size limits 7 μm and 50 μm were applied as thresholds between clay-silt and silt-sand fractions based on the results of previous research by Makó, A. *et al.* (2019). The PSD of the soil samples had previously been determined by the conventional SPM sedimentation method according to the international standard (all adhesives removed) (ISO 11277:2009(E)) (see *Table 1*).

Statistical methods

Univariate (GLM/UNIANOVA) and One-Way ANOVA (Post-Hoc/Duncan test or Tamhane's test depending on the homogeneity of variances) were performed to examine the combined effect of the factors studied (pre-treatments, quality of the aqueous media) on the LDM measured particle size fractions (PSF – clay, silt and sand content) using SPSS ver. 20.0 software.

First, the amount of each particle size fraction (the results measured on all soil samples considered as a common group) per pre-treatment and per fluid were compared. The results were plotted on boxplot charts, in which the PSF values determined by conventional sieve-pipette measurements (SPM) were also displayed. As an additional step, we also examined how these effects were expressed in PSFs of soils with various physical and chemical properties. For visual and basic statistical comparison of PSF results were also displayed on boxplot diagrams and in tables per soil samples and per treatment (the former also included PSF values determined by SPM). Thus, our results might provide information not only on the effect of different types of treatments on the amount of clay (< 7 μm),

silt (7–50 μm) and sand (> 50 μm) fractions in each sample but also the influence of the quality of the liquid phase used, the various types of preparation and the measurement methods used, respectively.

Results

Effects of treatments and the types of liquid media on the PSFs (clay, sand and silt content) of all the soil samples

The significant effect of the factors (pre-treatments, aqueous media quality, soil variation) and their combinations (P < 0.001 in all cases) on the measured clay, silt and sand content was proven by the results of GLM Univariate Analysis (UNIANOVA).

Comparisons using One-Way ANOVA tests showed that, for all three aqueous media, T1 resulted the significantly lowest clay content in all soil samples. If DW and DIW were used, the clay contents were not significantly different for T2 to T4, while for TW, T4 resulted in significantly the highest clay content *(Figure 2)*.

Deviations in the quality of the aqueous media caused much larger differences in silt content. In the case of measurements in DW medium, the same trends as for clay content were observed: significantly lower silt content was measured in T1 than in T2-T4, but no verifiable differences between the results of T2-T4 were observed. For DIW, T1 resulted the lowest silt content, but the highest verifiable silt contents were obtained in T2. However, the highest amount of silt fraction in the TW was measured when the samples were treated with US and Calgon (T3).
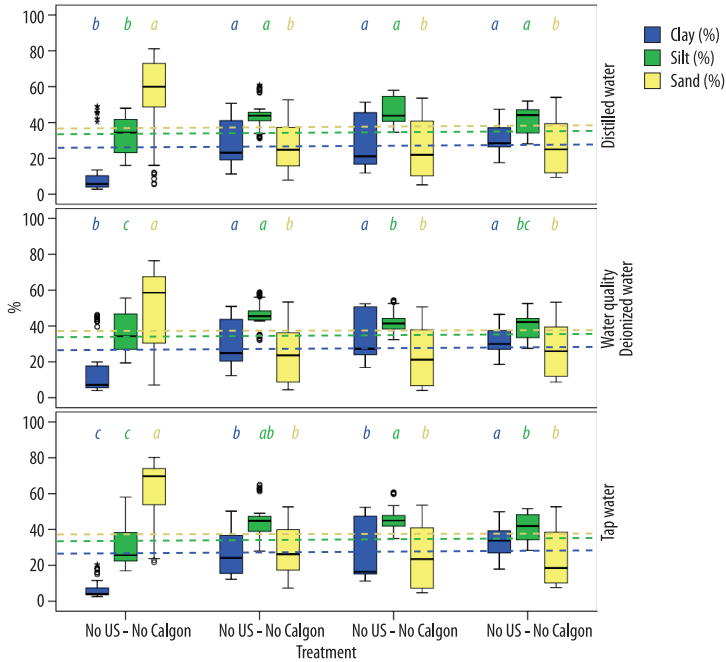
Fig. 2. Changes in LDM clay content in different treatments and aqueous media (all soil samples considered together). Means denoted the same letter did not significantly differ at p < 0.05 (One-way ANOVA). The dashed colour lines indicate the median values of SPM PSD results of the samples. *Source*: Elaborated by the authors.

For sand contents, a similar trend was experienced in all three aqueous media: the amount of particles in the sand fraction was significantly highest in T1, while no difference was experienced between the sand contents of the other treatments.

It is also worth comparing the first and third quartile values (Q1, Q3), the minimum and maximum values and the outliers in the boxplot diagrams in these figures. In *Figure 2*, the outliers showed the clay content of the high sodium content soil samples (S1), which were also dispersed by T1. Otherwise, it could be seen that the distances between Q1 and Q3 were narrow for all three aqueous media for T1, indicating that relatively little amount of clay was released from the aggregates.

In T2 and T3, the clay contents, in addition to being larger, were in a wide range (large interquartile ranges between Q1 and Q3). The clay content measured in T4 is, if not always

significant, was the more and the interquartile range between Q1 and Q3 similarly narrow.

When examining the boxplot diagrams of the silt contents, the first striking feature was the larger variance of measurement results for T1, than other treatments. Outliers were observed for T2 and in some cases for T3, and then they disappeared in T4. Samples containing an outlier sand fraction could be mainly distinguished only from untreated samples if distilled water was applied.

There was no significant difference between the PSF values determined with DW, DIW or TW, but the boxplot diagrams showed that the quality of the aqueous media might have affected the results of the measurement. Without pretreatment (T1), for example, the standard deviation of the PSFs measured in deionised water was the largest for all three. In addition, the number of outliers and their values relative to the minimum and maximum PSF values were different.

Effects of preparation and measurement type on PSFs of all the soil samples

Using LDM, lower clay content, with enormous standard deviation was determined than with sieve-pipette method after total preparation of samples (SPM) (see *Figure 2)*. There were exceptions to this: the LDM median values of clay content measured with T4 in DW, T2 and T3 in DIW were almost equal to the SPM median values, while in DIW and TW the T4 LDM measured higher clay contents than SPM. A similar or higher silt fraction was measured using the LDM method. A higher sand fraction of LDM than SPM measurement was experienced only in the case of T1 treatment.

Results of LDM analysis for soils with different properties

The dispersing effect of aqueous media for each treatment was displayed in four figures, where signs of One Way ANOVA ("a"–"c") presented the significant difference between mean PSF per various types of aqueous media (in abc order the mean values decreased). These figures provide the opportunity to visually investigate the effect of the quality of the liquid media.

Three tables contain the results of One Way ANOVA tests, where the effect of the treatments in various aqueous media was compared for each soil. In these tables, soils with the same dispersion patterns (showing similar levels of disaggregation response to various treatments) were marked with similar colours. First, we investigated the PSF determined by the LDM PSD measurement results of the samples with the highest clay content (S1: Karcag [*Solonetz*], and S8: Kisújszállás [*Vertisol*]) (*Figure 3)*. In general, there was a significant deviation between the dispersibility of S1 and S8 soil samples in each aqueous medium. The clay content and CEC values of the two samples were very high (52–56% – measured according to the ISO standard; CEC: 40.85 and 35.69 mgEq/100 g soil – see *Table 1*), but only S1 had a higher adsorbed Na^+ content compared to S8 soil (20.63 and 0.29 Na^+ mgEq/100 g soil).

Rapid spontaneous dispersion of S1 samples was observed in T1 (stirring and flowing only) in DW and DIW water (due to the inherent sodium content of the samples), whereas this phenomenon did not occur in TW. (The clay contents measured in TW were significantly lower, and the sand contents were higher.) The repeatability of the measurement was also reduced at T1 in the case of all liquid media applied.

In the combined case (T4), we measured higher silt content and lower clay content than in any other treatment, apparently irrespective of the type of aqueous media. This phenomenon was not observed in the case of other soils.

The S8 sample contained very small amounts of adsorbed Na^+, and therefore, when it was placed alone in any aqueous media without ultrasonic treatment or addition of Calgon solution, the aggregates did not disperse significantly. The particle size fractions of S8 sample in the various aqueous media for each treatment were different (see *Figure 3)*. In the case of samples treated without Calgon (T1 and T3), measurements in DIW media showed the highest dispersing effect (significantly the highest clay content was observed here). Either no significant deviations in dispersion were observed in the presence of Calgon (T2) or the results of measurement in TW showed the significantly highest clay release (T4). The difference between the repetitions of individual measurements (the distance between Q1 and Q3 on the boxplot diagrams) was the smallest, regardless of the type of aqueous medium when ultrasound and Calgon were applied in combination (T4).

At Cambisol A, B horizons (S2: Keszthely A [*Cambisol*] and S3: Keszthely B [*Cambisol*]) and Luvisol A, B horizons (S4: Várvölgy A [*Luvisol*] and S5: Várvölgy B [*Luvisol*]) (*Figures 4* and *5)* DIW released most of the clay content in T1, and there was no significant difference in the dispersing effect in T2 (except for S5, where TW dispersed the most).

In T3, only DIW had the significantly highest dispersing effect, while in T4 the results were mixed (significantly or non-significantly, TW medium released the most clay or TW and DIW clay contents were almost the same).
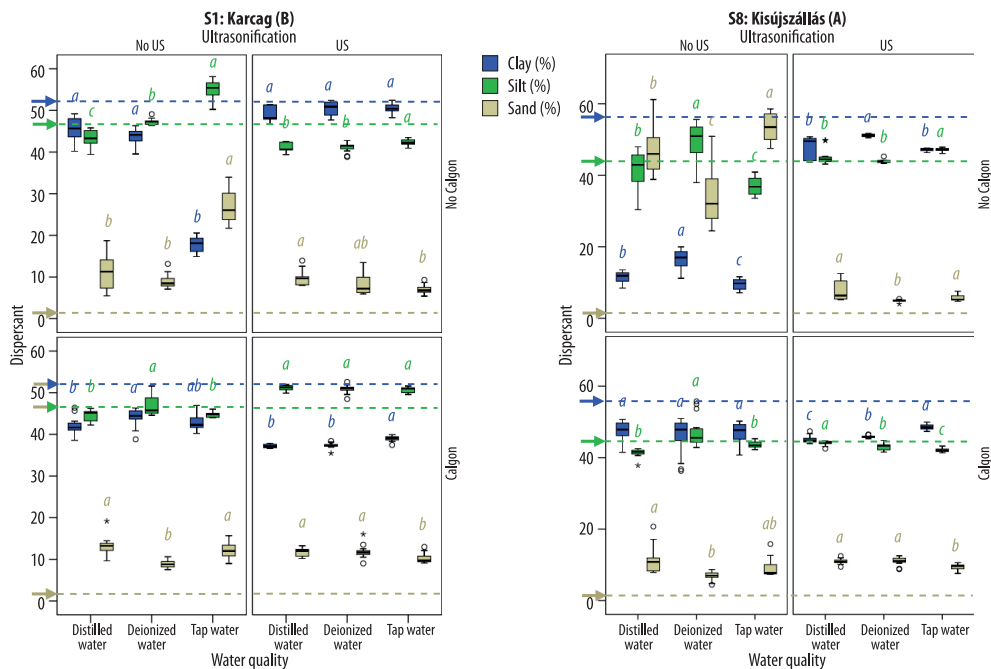
Fig. 3. Changes in clay, silt and sand content (particle size fractions – PSF) measured by laser diffraction method (LDM) in various aqueous media, grouped by soil sample tested – comparison of S1: Karcag (*Solonetz*) and S8: Kisújszállás (*Vertisol*) samples. Letters (a–c) indicate significantly different (p < 0.05; One-way ANOVA) mean PSF values (in ABC order the mean values decrease). The coloured arrows indicate the PSF determined by sieve-pipette method (where the colours correspond to the colours of the PSF determined with LDM). *Source*: Elaborated by the authors.

For the *Cambisol* samples (S2; S3), we can expect a small textural difference between the A and B horizons (with a higher clay content in the B horizon). In the case of the *Luvisol* samples (S4; S5), this difference was significant between the A and B horizons (as observed in the SPM measurements according to the ISO standard (see *Table 1*)). However, this texture differentiation was not always detectable in the results of the LDM PSD tests.

In the *Cambisol* A and B horizon samples (S2; S3), the highest total clay content was measured in TW with the use of US and Calgon (T4) (A horizon: 33.48% clay; B horizon: 30.07% clay) (*Table 3*, *4* and *5*). Dispersion proved to be the most effective in this case. However, the LDM determined a significantly higher clay content than the ISO SPM (~ 21–23%) (see *Table 1*).

Differences in PSF were experienced at all soil profiles only in the DW media with the T2 treatment (and to some extent with T3), in the DIW media with T2 and T3, and in the TW media with T2 (and to a lesser extent with T3 and T4) in the *Luvisol* soil (S4; S5) profile.

Figure 6 compared the particle size fractions determined from LDM PSD results of the two soil samples in various aqueous media (S6: Magyarszombatfa [*gleyic Luvisol*]; S7: Kápolnásnyék [*calcic Chernozem*]). It could be seen that for S6, in the pre-treatments without Calgon (T1 and T3), the order of clay dispersion was DIW > TW > DW, while with the addition of Calgon (T2 and T4), it was TW > DIW = DW. For S7, the deviation between Calgon and non-Calgon treatments was not as clear. In the case of T1 (neither US nor Calgon), the dispersion order was DW = DIW > TW.
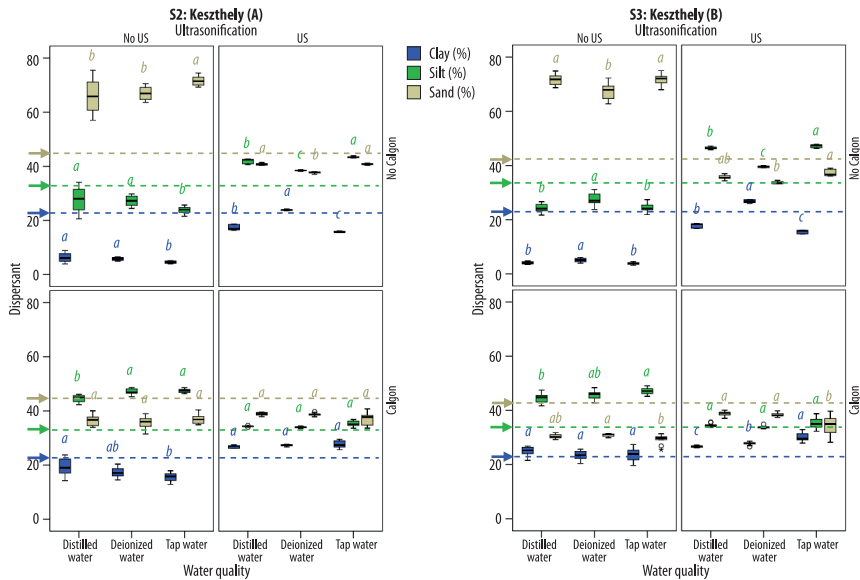
Fig. 4. Changes in clay, silt and sand content (particle size fractions – PSF) measured by laser diffraction method (LDM) in various aqueous media, grouped by soil sample tested – comparison of S2: Keszthely [*Cambisol*], and S3: Keszthely [*Cambisol*] samples. Letters (a–c) indicate significantly different (p < 0.05; One-way ANOVA) mean PSF values (in ABC order the mean values decrease). The coloured arrows indicate the PSF determined by sieve-pipette method (where the colours correspond to the colours of the PSF determined with LDM). *Source*: Elaborated by the authors.
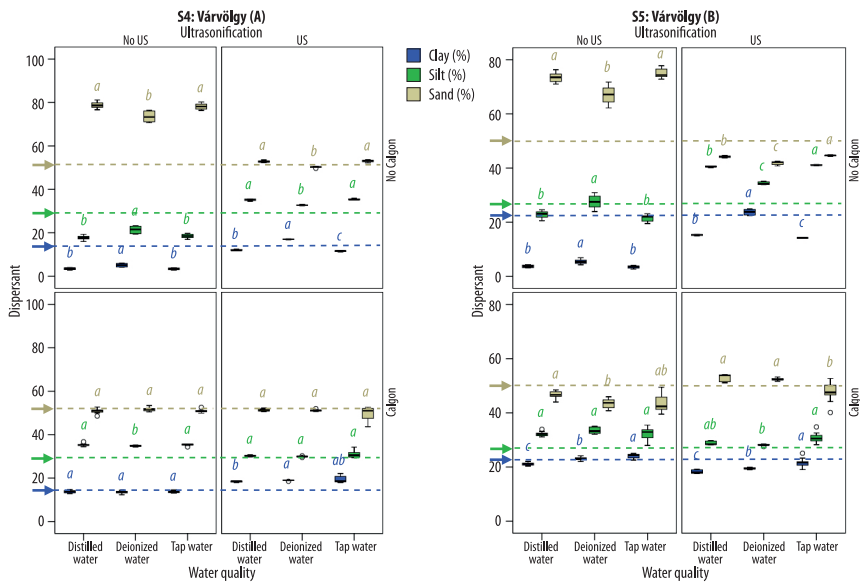

Fig. 5. Changes in clay, silt and sand content (particle size fractions – PSF) measured by laser diffraction method (LDM) in various aqueous media, grouped by soil sample tested – comparison of S4: Várvölgy [*Luvisol*], and S5: Várvölgy B [*Luvisol*] samples. Letters (a–c) indicate significantly different (p < 0.05; One-way ANOVA) mean PSF values (in ABC order the mean values decrease). The coloured arrows indicate the PSF determined by sieve-pipette method (where the colours correspond to the colours of the PSF determined with LDM). *Source*: Elaborated by the authors.
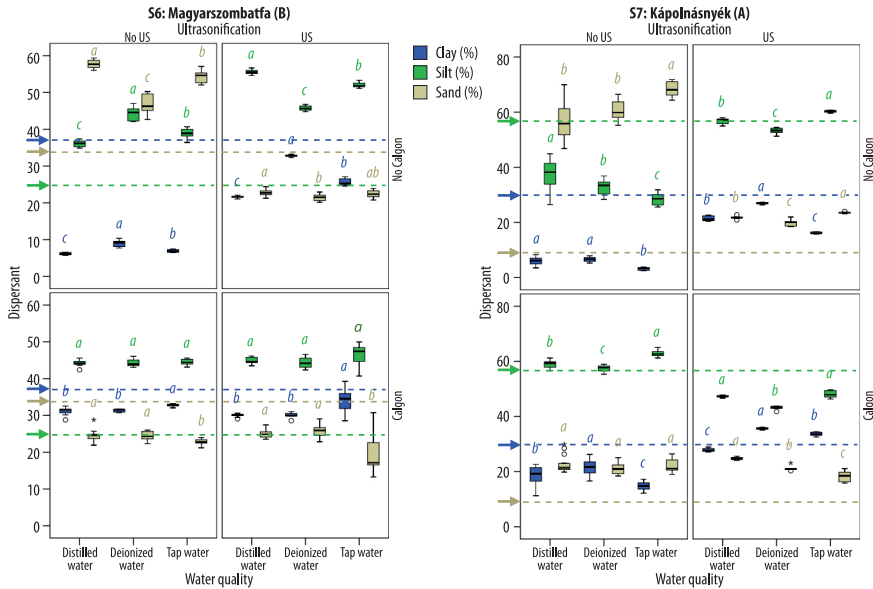
Fig. 6. Changes in clay, silt and sand content (particle size fractions – PSF) measured by laser diffraction method (LDM) in various aqueous media, grouped by soil sample tested – comparison of S6: Magyarszombatfa [*gleyic Luvisol*], and S7: Kápolnásnyék [*calcic Chernozem*] samples. Letters (a–c) indicate significantly different (p < 0.05; One-way ANOVA) mean PSF values (in ABC order the mean values decrease). The coloured arrows indicate the PSF determined by sieve-pipette method (where the colours correspond to the colours of the PSF determined with LDM). *Source*: Elaborated by the authors.

When only one of the pre-treatments was applied (US or Calgon), the dispersion order was as follows: DIW> DW > TW. For the combined T4 (US+Calgon), this order changed slightly to DIW > TW > DW.

In the case of TW, the variation of measured silt and clay contents in the S7 soil also increased in the combined treatment. In contrast, for S6, the silt fraction was higher for the US treatment than for the other treatments, while the clay content remained similarly higher for all three treatments than for no treatment. The slightly diverse behaviour of S5 compared to the previous three samples could be explained by a presumably higher Fe-oxide/hydroxide content (3% compared to 0–2% goethite content in the previous three) and/or by a various clay mineral composition (in this sample the amount of swelling clay mineral was 5% compared to only 2% in the previous three).

The mineral and chemical compositions of the S6 and S7 soils differed from the other samples tested. The S6 contained a significant amount of goethite mineral (10%), presumably with a higher iron content, while the S7 sample contained calcite mineral (10%) with a medium carbonate content (9.52%). The organic matter content of S7 was also high, with the calcium humates forming stable aggregates (crumbs).

When comparing the various treatments (see *Tables 3, 4* and *5*), it could be said that for both soils in all three aqueous media, the lowest clay content was measured in T1, and in general the highest was measured in T4. There were exceptions to the latter, e.g. in the case of S1 sample, where the lowest clay content was measured in the combined treatment of any of the liquid media used.

Furthermore, at S5 samples in all liquid media, the chemical dispersion with Calgon proved to be the most successful, and at S8 US treatment resulted higher clay content in DIW and DW and a similar rate of dispersion was obtained using any of the treatments (T2-T3) in TW.

Table 3. Comparison of LDM clay, silt and sand contents measured in distilled water (DW) by treatment – within a soil sample*

Distilled water								
Soil sample	1	2	3	4	5	6	7	8
Clay (%)								
No US - No Calgon	45.53 a	6.26 c	4.10 c	3.47 d	3.70 d	6.18 c	6.02 c	11.48 c
No US - Calgon	42.13 b	18.96 b	24.92 a	13.81 b	21.18 a	31.14 a	18.38 b	47.39 ab
US - No Calgon	45.95 ab	17.32 b	17.87 b	12.07 c	15.29 c	21.63 b	21.39 b	47.49 a
US - Calgon	36.15 c	26.83 a	24.22 a	18.41 a	18.26 b	30.05 a	27.87 a	45.03 b
Silt (%)								
No US - No Calgon	43.29 b	27.74 d	24.26 c	17.74 c	22.82 d	36.09 c	37.38 d	41.68 ab
No US - Calgon	44.51 b	44.54 a	44.58 a	35.31 a	32.19 b	44.20 b	58.95 a	41.28 b
US - No Calgon	44.15 b	41.90 b	46.47 a	35.13 a	40.52 a	55.61 a	56.84 b	45.15 a
US - Calgon	49.40 a	34.31 c	33.21 b	30.23 b	28.77 c	44.85 b	47.32 c	44.04 a
Sand (%)								
No US - No Calgon	11.18 ab	66.00 a	71.64 a	78.79 a	73.49 a	57.72 a	56.60 a	46.84 a
No US - Calgon	13.37 a	36.50 c	30.50 d	50.87 c	46.63 c	24.66 b	22.67 bc	11.33 b
US - No Calgon	9.90 b	40.78 b	35.65 c	52.80 b	44.19 d	22.75 c	21.77 c	7.35 c
US - Calgon	14.45 ab	38.86 c	42.57 b	51.36 c	52.97 b	25.11 b	24.80 b	10.92 b

*Means marked with the same letter did not significantly differ at p < 0.05 (One-way ANOVA). Within a fraction, soils with similar "patterns" (where the proportions of fractions measured in each treatment were similar) were marked with similar colours.

In general, the Calgon treatment resulted the highest amount of silt fraction in forest soils, almost independent of the quality of the liquid media. The higher sand content of the untreated samples (T1) was only observed for DW and DIW in S1, but not in TW.

The variation in particle size fractions determined by different combinations of chemical and dispersion effects varied from soil to soil (colours of *Tables 3, 4* and *5*). The effect of the liquid phase was less pronounced, but modified the results.

Discussion

Variation in particle size fractions by preparation and measurement methods

It is common practical experience that various preparation and pre-treatment procedures are lengthy, and therefore, in many cases, these are reduced in LDM PSD measurements (e.g. aggregate adhesives are not removed before measurements, disaggregation is considered to be taken care of by

Table 4. Comparison of LDM clay, silt and sand contents measured in deionized water (DIW) by treatment – within a soil sample*

Deionized water								
Soil sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Clay (%)								
No US - No Calgon	43.71 b	5.82 d	5.06 d	5.08 d	5.47 c	9.00 d	6.61 d	16.34 c
No US - Calgon	44.12 b	17.28 c	23.32 c	13.49 c	23.06 a	31.33 b	21.57 c	45.98 b
US - No Calgon	50.41 a	23.87 b	26.77 b	16.99 b	23.75 a	32.79 a	26.97 b	51.16 a
US - Calgon	37.32 c	27.30 a	27.80 a	18.94 a	19.47 b	30.05 c	34.27 a	45.88 b
Silt (%)								
No US - No Calgon	47.24 b	27.28 d	27.47 d	21.45 d	27.48 b	44.18 ab	32.80 d	48.98 a
No US - Calgon	47.05 b	47.04 a	45.80 a	34.81 a	33.47 a	44.33 b	57.43 a	47.18 ab
US - No Calgon	41.12 c	38.44 b	39.53 b	32.75 b	34.37 a	45.79 a	53.27 b	43.93 b
US - Calgon	50.90 a	33.99 c	33.81 c	29.89 c	28.14 b	44.34 ab	45.03 c	43.08 c
Sand (%)								
No US - No Calgon	9.04 b	66.90 a	67.47 a	73.47 a	67.04 a	46.83 a	60.59 a	34.68 a
No US - Calgon	8.83 b	35.68 c	30.88 d	51.70 b	43.47 c	24.33 b	21.00 b	6.84 c
US - No Calgon	8.47 b	37.69 c	33.71 c	50.26 c	41.88 c	21.42 c	19.75 b	4.91 d
US - Calgon	11.78 a	38.72 b	38.38 b	51.17 b	52.40 b	25.61 b	20.70 b	11.04 b

*For footnote see *Table 3.*

ultrasonic treatment and the addition of dispersant to the suspension is either regarded as important or not). However, our results proved that all treatments caused breakdown of aggregates, but to different degrees. It could also be seen from the interquartile range values of particle size fractions (PSF – *figures 3–6*), and the comparison of the results of SPM (with full preparation) and LDM methods that the disaggregation of unprepared and untreated samples was insufficient for accurate, reproducible quantification of elemental particle size fractions. (Assuming that the ISO standard SPM method was suc-

cessful in breaking down the aggregates to a high degree and releasing the whole clay fraction.)

LDM tests usually largely "underestimated" the clay content compared to the SPM method, even for treated samples. In many cases, the difference in silt fractions between LDM and SPM was significant, which also reflected that the degree of disaggregation of samples that could be achieved by different treatments could vary considerably.

In general, the silt contents were "overestimated" for all except the T1 measurements, while the sand contents were "underesti-

Table 5. Comparison of LDM clay, silt and sand contents measured in tap water (TW) by treatment – within a soil sample*

Tap water								
Treatment	1	2	3	4	5	6	7	8
Clay (%)								
No US - No Calgon	17.94 *d*	4.58 *c*	3.94 *d*	3.39 *d*	3.46 *d*	6.89 *c*	3.22 *c*	9.49 *b*
No US - Calgon	42.97 *b*	15.57 *b*	23.47 *b*	13.82 *b*	24.03 *a*	32.74 *a*	14.75 *b*	46.88 *a*
US - No Calgon	50.53 *a*	15.76 *b*	15.59 *c*	11.56 *c*	14.21 *c*	25.66 *b*	16.20 *b*	46.18 *a*
US - Calgon	39.00 *c*	33.48 *a*	30.07 *a*	19.34 *a*	21.68 *b*	34.04 *a*	37.10 *a*	47.73 *a*
Silt (%)								
No US - No Calgon	55.00 *a*	23.82 *d*	24.41 *c*	18.40 *c*	21.55 *c*	38.94 *c*	28.53 *d*	37.17 *d*
No US - Calgon	44.76 *c*	47.41 *a*	47.22 *a*	35.31 *a*	32.37 *b*	44.46 *b*	62.93 *a*	43.63 *b*
US - No Calgon	42.38 *d*	43.42 *b*	47.16 *a*	35.40 *a*	41.14 *a*	52.05 *a*	60.28 *b*	47.97 *a*
US - Calgon	50.72 *b*	36.87 *c*	35.37 *b*	31.00 *b*	30.75 *b*	46.47 *b*	50.67 *c*	41.38 *c*
Sand (%)								
No US - No Calgon	27.06 *a*	71.59 *a*	71.73 *a*	78.21 *a*	74.99 *a*	54.18 *a*	68.24 *a*	53.33 *a*
No US - Calgon	12.26 *b*	37.02 *c*	29.31 *c*	50.86 *c*	43.61 *b*	22.80 *b*	22.30 *bc*	9.50 *b*
US - No Calgon	7.09 *c*	40.81 *b*	37.25 *b*	53.04 *b*	44.65 *b*	22.29 *b*	23.52 *b*	5.85 *c*
US - Calgon	10.29 *b*	29.65 *bc*	34.55 *b*	49.66 *bc*	47.57 *b*	19.50 *b*	12.23 *c*	10.89 *b*

*For footnote see Table 3.

mated" for all except the T1 LDM measurements. It can be seen from *Figure 2* that the LDM measurements were least successful in achieving total disaggregation at T1, and were the most effective at T4 for DW and T2 and T3 for DIW. From *Figure 2* it was likely that the micro- and macro-aggregates in the sand fraction were disintegrated into elementary particles (clay, silt and sand) and micro-aggregates (mainly to sizes corresponding to the silt fraction) by the T2-T4 treatments but that complete disintegration of aggregates did not generally occur.

It was also puzzling why the median values of LDM sand contents in the boxplots were systematically smaller than the medians of the measured SPM values (see *Figure 2*). Where did part of the sand fraction go? It is possible that with maximum ultrasonic action and mixing, the entire sand fraction could disintegrate due to "too strong" dispersion effects. However, it was not consistently followed by a more significant disintegration of the silt fraction. Further research in this direction should be needed, as some literature has reported that sand fractions measured by conventional methods are often "under- or "overestimated" by LDM methods depending on the type of apparatus and even the design of the preparation units (Sochan, A. *et al.* 2012; Polakowski, C. *et al.* 2015; Mattheus, C.R. 2020; Stevenson, A. *et al.* 2023).

Effect of the physical and chemical dispersion

Dispersion rates were lowest for all aqueous media in T1, because elementary particles could not be separated during the measurement without pre-treatment even if a slight spontaneous disaggregation effect of the aqueous medium may be prevailed (e.g. in dispersive soils). Generally, higher clay content and the smaller standard deviation between the individual measurement repeats suggested that the disaggregation was the most complete in the T4 among each LMD measurement.

If some pre-treatment was applied, the greatest variation was in the silt fraction. The measurable amount of this fraction was significantly affected by the quality of the liquid media. The high interquartile range also showed that the amount of partial disaggregation varied with soil properties and the type of liquid media. The outliers under treatments were also more significant in the silt fraction, indicating that the effect of pre-treatment was most critical in determining this fraction (which obviously affected the measurement results of other fractions). The uncertainty of the measurement results was also reflected in the large variation in the clay content that could be determined by different treatments.

It is questionable, however, how to explain the small, although not significant, increase in the median of sand content at T4 treatment compared to T3 and T2 treatments, despite the lack of parallel statistically verifiable changes in the other fractions in this comparison. It is also interesting to note that the resulting clay content followed a similar "disaggregation pattern", being much higher in the T2-T4 treatment than in the T1 treatment (untreated samples).

It was also the case that disaggregation and dispersion of well-structured soils with high aggregate stability (such as the S6 [*gleyic Luvisol*], and S7 [*calcic Chernozem*]) occurred in several stages, and it is a question of which stage we "caught" and measured the particle size distribution during our measurements. Thus, the "particles" measured by the LDM method can in many cases, be a mixture of ag-gregates and elementary particles of different sizes (e.g. Buurman, P. *et al.* 1997). It is very difficult to separate elementary particles from small micro-aggregates and even from "composite building units" of elementary particles in lower size ranges (Totsche, K.U. *et al.* 2018).

In the sequence of pre-treatments, the macro-aggregates in the sand fraction were increasingly transformed into micro-aggregates in the silt fraction, and then, towards T4, a larger part of these micro-aggregates were also broken up and some of the particles were measured in the clay fraction. This trend was broadly supported by the boxplot diagrams of sand contents in the three aqueous media (see *Figure 2*). Similar, "intermediate" dispersion states in various aqueous media had been reported by the authors in other research investigating PSD or ASD of soils and sediments (e.g. Goosens, D. *et al.* 2014; Abdulkarim, M. *et al.* 2021). The variable number of outliers for treatments may indicate that different dispersion states could be achieved with various degrees of disaggregation depending on soil properties.

Sometimes, not only the partial disaggregation but flocculation, precipitation and the formation of artefacts were occurred (e.g. in high colloid and/or sodium content soils), as noted by also Shein, E.V. *et al.* (2006), Tan, X. *et al.* (2017), and Abdulkarim, M. *et al.* (2021). These were due to soil/aqueous media, soil/dispersant or aqueous media/dispersant interactions and varied depending on the treatment applied and soil properties. Calgon is used as a dispersing agent to saturate the negatively charged surfaces of soil with Na$^+$.

The required Calgon concentration might depend on the soil's CEC value and the quality of the adsorbed cations. When comparing the effect of each treatment on the S1, it was seen that T1 and T3 released the most clay particles from the aggregates in DW, and T3 in DIW and TW. With the addition of Calgon solution (T2 and T4), the salinity of the fluid increased significantly, thus, enhancing flocculation effects in the system. Therefore, the rate of clay dispersion was lower with Calgon only (T2) than with sonification only (T3).

Presumably, therefore, this was not primarily a function of the properties of the liquid but rather the result of the interaction between the solid and liquid phases and the dispersant, which could be enhanced or weakened by the effect of the US (re-aggregation or flocculation of the components). A similar result was obtained, e.g. by Abdulkarim, M. *et al.* (2021), who explained this by complex simultaneous or specific processes. In addition, the dispersing effect of Calgon (repulsive effect of increasing Na⁺ content; complexing ability of NaHMP; increasing the pH of the suspension and reducing positively charged surfaces) depended on the proportion of Na⁺ present and able to act in the suspension. The CEC value (which, for example, is twice as high in *Chernozem* soil compared to *Luvisol* soil) related to the amount and quality of the adsorption sites, colloid surfaces, e.g. the clay content, the mineral composition (including Fe-(oxi)hydroxides content), and the presence and quality of organic matter. Calgon should form calcium-phosphate and calcium-carbonate precipitates in soil when the measurement occurs in tap water or hard water (Buurman, P. *et al*. 1997).

Insufficient or excessive addition of Calgon (both dependent on soil properties) could also lead to erroneous measurements (Murray, M.R. 2002; Kaur, A. and Fanourakis, G. 2018). It is likely that the amount of Calgon solution to be added could be optimized even by soil type (Kaiser, M. *et al.* 2012). However, since this complicates the standardization of measurements, the most widely used concentrations are those specified in the ISO 11277:2009(E) standard.

Effect of soil properties

To summarise, *figures 3–6*, and *tables 3–5* showed that all soils gave different "responses" to various levels and types of disaggregation and dispersion (chemical and/or physical). The extent of these responses, therefore varied with the type of treatments applied, and potentially different size ranges

of aggregates/non-elementary particles were affected, according to a "pattern of disaggregation" (disaggregation and/or dispersion processes).

For example, while in the case of DW measurements, the clay content of the S1: Karcag (*Solonetz*) untreated sample (T1) showed both positive and negative variation between treatments, the clay content of the S7: Kápolnásnyék [*calcic Chernozem*] sample increased monotonically in T1-T4.

The sample with high sodium content (S1) exhibited a significantly distinct disaggregation pattern from the other samples due to major dispersion, even with low disaggregation effects/forces. This may indicate that, in addition to spontaneous disintegration on contact with the liquid phase (Amézketa, E. *et al.* 2003), there was rapid disintegration of weakly bound particles/aggregates. All three treatments showed similar effectiveness to the applied full preparation at the SPM. In general, the "dispersion pattern" of the tested S1 sample pre-treatment showed huge deviations compared to the other tested soil samples for all measurements in various aqueous media (see *Table 3, 4* and *5* – purple colouring).

The particle size fractions of the tested S2: Keszthely (A) [*Cambisol*]; S3: Keszthely (B) [*Cambisol*] and S4: Várvölgy (A) [*Luvisol*]; S5 Várvölgy (B) [*Luvisol*] (see *Figure 4* and *5*) showed that the dispersing, disaggregating effect of the aqueous media varied depending on the pre-treatment.

Comparing the dispersing effect of each pre-treatment on different soils, we measured the significantly highest clay content with the T4 (combined treatment) in all aqueous media for the S2 and S4 samples. For the S3 sample, we observed similar results. However, for S5, the T2 resulted the best dispersing effect in DW and TW media, while for DIW, T3 released the highest amount of clay (see *Table 3, 4* and *5*). These soil samples are more similar to each other than to the soils of the first group (upper genetic horizons of forest soils with various organic matter contents), which may explain their similar disaggregation and dispersion behaviour.

When TW was used in the LDM measurement, at S7: Kápolnásnyék (*Chernozem)* soils silt and clay contents also increased in the combined treatment (the distance between Q1 and Q3 increased in the boxplot plots), suggesting the formation of possible Ca phosphate precipitates from Calgon and calcium carbonate in TW (possibly soil) (see Buurman, P. *et al*. 1997) (see *Figure 6*). In some cases, for example at S6: Magyarszombatfa [*Luvisol*], the more effective treatment (at least the more silt content) was experienced by the use of only US. The chemical and physical dispersion (and sometimes the degree of flocculation) can also vary depending on the mineral composition of the soil (e.g. Goossens, D. *et al*. 2014; Tan, X. *et al*. 2017; Polakowski, C. *et al*. 2023). In this sample, the role of iron hydroxides/oxihydroxides, which hold aggregates together, may be the most crucial in dispersion. Fe-oxides/hydroxides can play an important role in the formation of not only micro- but also macro-aggregates due to their high specific surface (Totsche, K.U. *et al*. 2018; Kirsten, M. *et al*. 2021). If some of these transform into goethite, a crystalline form with a smaller specific surface area and lower reactivity, the active (mainly non-crystalline) iron content may decrease, altering the stability of aggregates (e.g. Kögel-Knabner, I. *et al*. 2008; Kaiser, M.K. *et al*. 2011; Regelink, I. *et al*. 2013). This may also determine the applicability of LDM PSD analysis for samples with higher iron content.

With increasing soil organic matter content, aggregate stability increases only within certain limits. The stability of aggregates depends on the quality of organic matter (Amézketa, E. 1999; Tyugay, Z. *et al*. 2010; Schulte, P. *et al*. 2016; Li, S. *et al*. 2023). This is also reflected in the characteristic disaggregation patterns of the S1: Karcag (*Solonetz)* sample with higher sodium humate content and the S7: Kápolnásnyék (*Chernozem)* sample containing mainly calcium-humates. We had less opportunity to investigate the effect of carbonates as components responsible for the stability of the aggregates, as the soils studied are mostly carbonate-free. Moreover,

the colloidal effects responsible for aggregation may not only be cumulative but may also be mutually reinforcing (Amézketa, E. 1999; Shein, E.V. *et al*. 2006; Schulte, P. *et al*. 2016; Totsche, K.U. *et al*. 2018). This may be the reason why some, either chemical or mechanical, or various intensities treatments may cause different degrees of disaggregation or even re-aggregation.

The dispersion pattern and behaviour of soils against disaggregation and dispersion forces also depend on their aggregate stability. More dispersible soils and even soil with higher aggregate stability might show similar PSF results at a given soil for any of the treatments (T2-T4), without preparation. But the reasons for this may be different. In the former case, a rapid high amount of dispersion and in the latter case, the fact that treatments are insufficient may lead to this (see *Table 3, 4* and *5*). Soils differ in their aggregate stability (Bronick, C.J. and Lal, R. 2005), and different soil constituents and binding forces form aggregates in various aggregate size ranges (Amézketa, E. 1999). Thus, different preparation and pre-treatment methods may be recommended prior to PSD measurements depending on the physical, chemical and mineral properties of the soils (Schulte, P. *et al*. 2016; Fisher, P. *et al*. 2017). These relationships might reveal high complexity, a dynamic "equilibrium aggregation" (Totsche, K.U. *et al*. 2018) is formed in the soil for each combination of soil properties, which might be affected by the changes of "external" conditions (e.g. dispersing forces or quality, hardness or pH of the liquid media) or "internal" conditions (e.g. rate of adsorbed cations, flocculation/dispersion of colloids). This may be the reason why, according to the experiences, the role of soil properties (e.g. organic matter content and quality, clay content and quality, carbonate content, etc.) varies enormously and may even have opposite effects on stability (e.g. Amézketa, E., 1999; Mamedov, A.I. *et al*. 2016; Virto, I. *et al*. 2011; Balázs, R. *et al*. 2011; Kaiser, M. *et al*. 2012; Kögel-Knabner, I. *et al*. 2008; Almajmaie, A. *et al*. 2017; Totsche, K.U. *et al*. 2018). In the case of

well-structured aggregated soils, a combination of physical and chemical disaggregation and dispersion techniques is therefore recommended (Shein, E.V. *et al.* 2006; Tyugay Z. *et al.* 2010; Abdulkarim, M. *et al.* 2021).

Effect of the aqueous media quality

Neither manuals for LDM measurement instruments nor practice gave precise instructions on the quality of the liquid phase to be used in the measurement. If all soils were considered together, the effect of liquid quality on the effectiveness of disaggregation processes was really less noticeable (it was not always significant).

However, when the differences in PSF values for soils with different soil properties were examined separately (*figures 3–6, tables 3–5*), it was found that the quality of the liquid phase (e.g. ionic composition, hardness, interaction with soil, dispersant, etc.) might have influenced the results. In the case of the sample with high Na^+ content, a significantly higher clay content was measured with tap water (TW) in the combined treatment (T4) than in other aqueous media. Parallel to this, particularly the sand content was lower. This might have been due to the addition of Calgon solution with high salt content to the tap water with also high salt content. This system was subjected to prolonged (240 seconds), high-energy (40 kHz) ultrasonic treatment, which facilitated ion dissociation and an increase in pH, thereby making dispersion more effective (Matouq, M. 2008). TW is a medium-hard water in the sense that its pH is moderately alkaline and contains higher amounts of salts (mainly Ca/Mg carbonates and bicarbonates, etc. – *Table 2*). This means that depending on the ionic composition of the solution, there are different possibilities for structural changes resulting from solid-liquid phase interactions (e.g. ion exchange, dissolution of salts).

Regarding the effect of aqueous media DW was applied, the clay content pattern of the more clayey B horizons of forest soils (S3, S5,

S6) was similar, but the higher organic matter content A horizons of forest soils (S2 and S4) were in a different group. Similar experiences were described for the behaviour of the soils in DIW treatments, but similar pattern for clay content was observed for S2-S4 soils and other pattern for soils with higher clay content S5, S6 and S8. Soils with characteristic soil properties could have been better distinguished in distilled water, which might have referred to lower interaction between solid-liquid phases than in measurement with tap water (see *Table 3, 4* and *5*).

Among the aqueous media, the most distinct dispersion of the soils in sand and clay content was observed in the TW measurement, which might have been due to the varying degrees of dispersion, probably incomplete, in this medium. The "patterns" of disaggregation in the TW measurements were significantly different from the previous two aqueous media. It could be assumed that these results reflected, for example, the differences in CEC between soils, as higher CEC allowed for a higher degree of ion exchange during PSD measurement (Mason, A. *et al.* 2011; Tan, X. *et al.* 2017), which affected the aggregate stability variously up to other soil properties. Depending on the liquid phase and the quality of the original soil solution, even precipitates might have formed (Shein, A.V. *et al.* 2006). However, fluids affected the measurement results differently, relying on the dispersing effect and the dispersant used in the LDM measurement and the amount of dispersant used.

Conclusions

The aim of our research was to elucidate the effect of the quality of the aqueous media on the amount of particle size fractions (PSF) determined from LDM PSD measurements in the case of soils with certain extreme properties (e.g. high clay or exchangeable Na^+ content, variable organic matter content).

We also investigated how the two most commonly used treatments (Calgon dispersant/ultrasonic dispersing) and their combina-

tions affected the PSFs when various aqueous media were applied. Also, how did these PSF results change depending on the above compared to the SPM ISO standard PSD results?

To summarise, all soils gave different "responses" to various levels and types of disaggregation and dispersion with no pre-treatment or applying Calgon and/or ultrasound. The extent of these responses will therefore vary with the type of treatments applied, and potentially different size ranges of aggregates/non-elementary particles may be affected, according to a "pattern of disaggregation/dispersion".

– The primary reason is that, according to the methodology used for LDM PSD measurements, soil aggregates were generally *only partially disaggregated* by ultrasonic and chemical dispersion or a combination of both. The degree of disaggregation can vary depending on the micro- and macro-aggregate stability determined by the physical, chemical and mineralogical properties of the soils (e.g. soils with high Na^+ content and low aggregate stability were well disaggregated by the dispersion methods used, whereas samples with high organic matter content or iron oxide content and high aggregate stability were poorly disaggregated). Thus, although of varying importance depending on the aggregate stability, it is considered necessary to assist the disaggregation of samples by removing aggregate-stabilising adhesives prior to LDM PSD measurements.

– The degree of disaggregation depends not only on soil properties but also on the *choice of dispersion methods*. In some of the soils studied, the combination of ultrasonic and chemical dispersion resulted in the highest degree of disaggregation, with clay content being released from macro- and micro-aggregates. For other soils, better results were obtained when only one type of dispersion method was used. The use of a chemical dispersant (Calgon) may, for example, result in the formation of artificial products (Ca-phosphates) in the presence of Ca^{2+} ions, or the combination of chemi-

cal dispersion and ultrasound may have a flocculating effect in the suspension.

– Another important finding was that the *choice of the aqueous media* used also affected the reliability and accuracy of LDM PSD results. The chemistry, ion content and soluble salt content of the aqueous media can influence the degree of disaggregation, the degree of dispersion and flocculation, the ion exchange processes and the formation of artificial products. The optimum aqueous media for more complete disaggregation varied depending on the soil properties and the dispersion methods used.

Our results point to the need for standardisation of the LDM PSD measurement methodology since only in this way can the results of the different laboratories be compared. They also call attention to the need to find a solution to ensure a more accurate determination of the particle size distribution of soil samples with extreme properties (by complete disaggregation where possible, complete dispersion of elementary particles, and elimination of artificial product formation) while maintaining the speed advantages of the LDM PSD methodology.

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