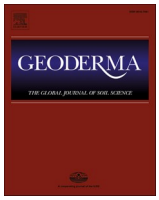
Geoderma 441 (2024) 116756

Contents lists available at ScienceDirect

Geoderma

journal homepage: www.elsevier.com/locate/geoderma



A laser diffractometry technique for determining the soil water stable aggregates index

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ARTICLE INFO

Handling Editor: L. Morgan Cristine

Keywords:
Water stable aggregates index
Soil aggregates
Laser diffractometry
Wet sieving

ABSTRACT

This study introduced and optimized the Sonication and Laser Diffractometry technique (SLD) for evaluating the stability of soil aggregates, drawing comparisons with the conventional Wet Sieving (WS) method. The SLD technique uses ultrasound and particle motion, and is shown to be suitable for fine soils where the WS method struggles with problems such as sieve screen clogging. Results show that the water aggregates stability index (WSAi) determined using SLD is consistent with values obtained from WS and has good reproducibility. Comparisons show that SLD offers advantages in speed, simplicity, and lower potential for human error, providing results in only a few minutes compared to the days required for WS. Challenges such as the adaptability of the methodology to different soil types and equipment remain areas for further research. Nevertheless, this study highlights the potential of SLD as an efficient and reliable method for assessing aggregate stability in soils.

1. Introduction

Healthy soils with stable aggregates experience lower soil erosion rates, and higher sequestration of carbon, nitrogen and phosphorus (Kasper et al., 2009). Stable soil aggregates also improve soil biological activity and crops productivity by facilitating the movement of air and water (Amézketa, 1999; Karami et al., 2012; Gyawali and Stewart, 2019). The stability of soil aggregates depends strongly on the amount and the composition of organic matter in the soil (Tisdall and Oades, 1982; Chaney and Swift, 1984; Haynes and Swift, 1990) and the content of clay particles and their complexes with humus (Six et al., 2002). Larger aggregates are generally found to be more fragile than small aggregates (Dexter, 1988; Sparling et al., 1994; Six et al., 2004; An et al., 2010; Gyawali and Stewart, 2019). Soil aggregates are often classified into microaggregates and macroaggregates according to their size, with a threshold diameter of 0.25 mm used for the division into two groups (Tisdall and Oades, 1982; Amezketa et al., 2003; Fristensky and Grismer, 2008). Most studies on the stability of wet aggregates have focused primarily on the stability of macroaggregates, as larger aggregates usually have the greatest impact on the soil structure (Robinson and Page, 1951; Kemper and Rosenau, 1986; Haynes and Swift, 1990; Almajmaie et al., 2017).

There are several methods for measuring aggregate stability (Kemper and Rosenau, 1986; Amézketa, 1999; Franzluebbers et al., 2000; Bieganowski et al., 2010; Fajardo et al., 2016; Almajmaie et al., 2017; Flynn et al., 2020; Rieke et al., 2022). It has already been shown that the different methods for determining water aggregate stability are not strongly correlated (Morgan, 2020). To determine water aggregate stability, aggregates are usually immersed in water for a certain time (Le Bissonnais, 1996) or are subjected to a defined kinetic energy, whether in the form of falling water drops (Moebius-Clune, 2017), mechanical sieving (Kemper and Rosenau, 1986) or ultrasound (Bieganowski et al., 2010), and then the degradation of the aggregates is monitored.

The wet sieving technique (Kemper and Koch, 1966) is the most commonly used method for estimating the stability of soil aggregates. The collapse of aggregates during wet sieving is caused largely by slacking in the water, and to a lesser extent by the physical effects of water movement and abrasion on the surface of the sieve during sieve movement (Kemper and Koch, 1966; Emerson, 1967; Kemper and Rosenau, 1986). Laser diffraction measurements detect a detailed distribution of aggregate sizes, although the distribution function is typically summarised by the median size of the soil particles (d50). Aggregate stability can then be characterised by analysing the d50 changes as the aggregates are disturbed (Bieganowski et al., 2010; Virto et al., 2011; Gyawali and Stewart, 2019). The d50 value has the advantage of being a single number, therefore making the analysis simple. However, d50 does not capture the textural differences between soils. Selection of the optimum method or procedure for determining the

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https://doi.org/10.1016/j.geoderma.2023.116756
Received 22 August 2023; Received in revised form 8 November 2023; Accepted 13 December 2023
Available online 25 December 2023

stability of soil aggregates should be based on the purpose of the analysis and the soil type (Herrick et al., 2001). The method must also be easily repeatable (Almajmaie et al., 2017).

The aim of this study is to introduce a rapid, simple and standardised method to evaluate the stability of soil aggregates in water by ultrasonically crushing the aggregates and determining the size distribution by laser diffraction. We present an uncertainty analysis of the technique and compare our results with those of the standard wet sieving technique of Kemper and Rosenau (1986).

2. Materials and methods

Disturbed soil samples were collected from six different localities in Czechia. All samples were taken from the top 10 cm of cultivated agricultural fields. The sampling localities are in different morphological and climatic conditions and represent the most common soil types in the Czech Republic. Brief information on the localities and soils can be found in Tables 1 and 2. The soil classification was made according to the IUSS Working Group (2015). Soil samples were collected in September 2022 and August 2023 when seedbed conditions prevailed in the fields. A total of five disturbed samples were taken at each of the six localities (see Table 1: NUC, ROH, RIS, ZEH, VRA, CIC), each with an approximate volume of 100 cm^3. The sampling sites within each location were randomly selected over an area of approximately 100 m^2. The soil samples from each site were then mixed into a composite sample, sealed in a plastic bag and transported to a laboratory.

In the laboratory, the samples were spread out in a thin layer, air-dried and freed from stones and organic residues. The samples were then sieved to obtain macroaggregates in a size range of 1 to 2 mm. For each sample, the pH was determined according to Blakemore et al. (1987). The pH of the soil was measured in a 0.01 M $CaCl_2$ solution. 10 g of soil was placed in a 50 ml beaker along with 25 ml of $CaCl_2$. The pH solution was stirred and was allowed to stand for at least 2 h. The pH was then measured using an Hach IntelliCAL pH PHC101 three-point calibrated electrode (Hach Company, USA). The procedure for measuring soil pH was carried out in three replicates for the six soils (IDs). The particle size distribution (PSD) was determined by laser diffraction, using a Mastersizer 3000 instrument (Malvern Panalytical, UK).

2.1. Wet-sieving technique (WS)

Wet sieving, as a standard technique for WSAi (water-stable aggregates index) determination (Kemper and Rosenau, 1986), was used to obtain the reference values. We used a wet sieving apparatus (Eijkelkamp Soil and Water, Netherlands) consisting of eight 250 μm sieves and 16 cups (8 cups for measurements in water and 8 cups for measurements in a dispersion solution) to collect broken aggregates. For the analysis, 4 g of soil with aggregate sizes between 1 and 2 mm were placed on 250 μm sieves. At least 24 soil replicates were measured for soils from each locality. The aggregates were pre-wetted with a plant sprayer and were allowed to stand still for 5 min. Slow prewetting was performed to make the conditions comparable to natural soil wetting (Amézketa, 1999).

Table 2
Characteristics of the soils.

ID	Soil type (WRB 2015)	Particle size distribution (%)			Soil texture	pH $CaCl_2$
		Clay < 0.002 (mm)	Silt 0.002–0.05 (mm)	Sand 0.05–2.0 (mm)		
NUC	Cambisols	2	63	35	Silt Loam	5.6 ± 0.1
ROH	Phaeozems	3	36	61	Sandy Loam	5.5 ± 0.2
RIS	Chernozems	9	55	36	Silt Loam	5.3 ± 0.1
ZEH	Fluvisols	38	50	12	Silt Clay Loam	5.4 ± 0.1
VRA	Cambisols	2	50	48	Sandy Loam	5.4 ± 0.1
CIC	Phaeozems	9	70	21	Silt Loam	5.5 ± 0.1

100 ml of distilled water was added to each cup and was repeatedly mechanically lifted and submerged in the wet sieve apparatus for 3 min (a single stroke of 13 mm, frequency approximately 34 cycles – min^{-1}). The sieving process was then repeated with the dispersion solution until the aggregates were completely dissolved. The type of dispersion solution was selected according to the measured pH, as recommended by Blakemore et al. (1987). For pH less than 7, sodium hydroxide was used at a concentration of 2 g l^{-1}. For pH above 7, sodium hexametaphosphate was used at a concentration of 2 g l^{-1}.

After sieving, the undersized soil samples were dried at 105 °C for 24 h and were weighed. For the samples with the dispersion solution, the weight of the reagent (0.2 g) was subtracted from the mass after drying. The water stable aggregate index (WSAi) was calculated as follows:

$$WSAi_{WS} = \frac{m_{ds}}{m_{H_2O} + m_{ds}} \cdot 100 \quad (1)$$

where:

- $WSAi_{ws}$ is the water-stable aggregates index (%) determined by wet sieving
- m_{ds} is the undersized mass of soil (g) in the dispersing solution (stable aggregates)
- m_{H2O} is the undersized mass of soil (g) in distilled water (unstable aggregates)

2.2. Sonication and laser diffractometry (SLD)

The measurements were carried out with the Mastersizer 3000 in combination with the instrument Hydro LV (Malvern Panalytical, United Kingdom). Hydro LV (Fig. 1) is an automated large-volume device in which aggregates are broken down into individual particles by the force of ultrasound radiation and movement through the device.

This configuration allows accurate and fast measurements of the

Table 1
Characteristics of the sampling locations (meteorological data based on VUMOP, 2017; CHMU, 2019).

Locality name	Soil ID	GPS	Elevation (m a. s. l.)	Annual rainfall (mm)	Annual mean air temperature (°C)
Nucice	NUC	14.8617398°E 49.9663870°N	392	550–600	9–10
Rohozec	ROH	15.3522415°E 49.9785102°N	223	500–550	9–10
Risuty	RIS	14.0174110°E 50.2173800°N	312	550–600	8–9
Zehusice	ZEH	15.4290595°E 49.9710047°N	211	500–550	9–10
Vrane nad Vltavou	VRA	14.2370233°E 50.1550328°N	209	500–550	10–11
Cicovice	CIC	14.3833539°E 49.9321797°N	295	500–550	10–11

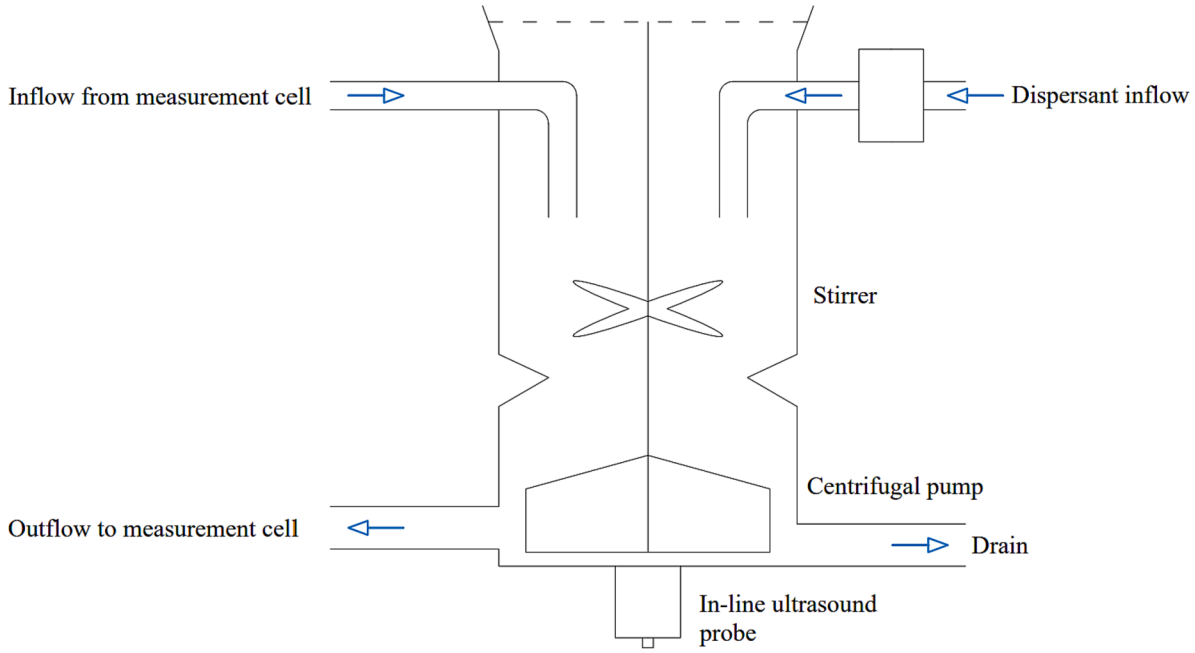


Fig. 1. Scheme of automated Hydro LV (adapted from Malvern, 2013).

particle size distribution (PSD). The Mastersizer 3000 emits red and blue laser beams (red laser: $\lambda = 633$ nm, blue laser: $\lambda = 466$ nm) through a sample dispersed in a liquid and records the light scattering pattern (Malvern, 2013). The light scattering caused by a homogeneous, isotropic sphere is commonlydescribed by the Mie theory (Wriedt, 2012). The full Mie theory algorithm is embedded in the Mastersizer 3000 software, which then allows the equations for the interaction of light with the matter to be solved to calculate the PSD (Malvern, 2013). In reality, soil particles are rarely spherical. Therefore, the measured particle volume is converted to an equivalent sphere of the same volume, for which the apparent diameter is calculated. For more details on Mie theory see the Mastersizer manual or a more detailed overview by Wriedt (2012). The laser diffractometer was calibrated before the experiments with the standards provided by the manufacturer.

A PSD analysis for each soil was carried out in three replicates (see Appendix A). The soil sample preparation was similar to the WS technique: the soil was pre-wetted with a fine plant spray and was left for 5 min before being immersed in the Hydro LV unit. Then approximately 0.5 g of the soil aggregates were dispersed in deionised water and approximately 0.2 g of the aggregates were dispersed in a dispersion solution. The dispersion solution was identical to the solution used in the WS technique. The sample mass is approximate as it significantly affects the degree of obscuration, which is the most important indicator of data quality. Initially, approximately 0.5 g of the aggregates should be added to the deionised water and 0.2 g to the dispersion solution. The mass of aggregates may need to be modified to achieve the ideal obscuration level. All records with obscuration above 35 % were excluded form this study and the measurement was repeated with a smaller sample volume, until satisfactory obscuration was reached. The resulting aggregate stability index is not affected by the sample size since the calculations are done with relative values.

2.2.1. *Mastersizer 3000 settings*

The Mie light scattering theory requires the definition of several parameters related to the soil properties and the settings of the analyser (Malvern, 2013). Table 3 summarizes the Mastersizer 3000 settings that have been found to be optimal through trial and error. To account for the irregularity of the soil particles, it is very important to set the particle type as 'non-spherical', as was shown by e.g. Gabas et al. (1994) and Blott and Pye (2006).

Table 3
Mastersizer 3000 and Hydro LV settings (*mass may vary, according to the measured obscuration).

Parameter		Distilled water	Dispersion solution
Soil properties	Refractive index	1.457	
	Absorption index	0.01	
	Particle density ($g \cdot cm^{-3}$)	2.64	
	Sample amount (g)	0.1–1 g *	
Properties of the solution	Refractive index	1.33	
Hydro LV properties	Sonicating level (%)	0	100
	Sonicating time (s)	0	70
	Stirrer/pump speed (rpm)	2500	
Measurement Properties	Red-laser duration (s)	10	
	Blue-laser duration (s)		
	Repetitions	5	
	Weighted residua (%)	<1	
Total operating time (s)		100	170
Total measurement time (s)		100	

2.2.1.1. Stirrer speed and sonication. The stirrer speed and the centrifugal pump speed are sensitive parameters that influence the disaggregation of soil aggregates. The Hydro LV unit allows stirrer/pump speeds in the range of 0 to 3500 rpm. The sonication probe has a power of 40 W and a frequency of 40 kHz. Since the soil aggregates settle quickly and to avoid foaming and air bubbles, the speed of the stirrer was set to 2500 rpm. At this speed, the aggregates did not settle, and no obvious breakup of the aggregates was observed. Sonication was performed at 100 % power for 70 s before the measurements started. This setting ensures complete disintegration of the aggregates.

2.2.1.2. Duration of the PSD analysis. Accuracy increases with increasing measurement time. Based on trial-and-error tests, the optimal measurement time was set to 10 s for both red and blue light beams. For each soil sample, a total of five repetitions (10 s red light and 10 s blue

light) were performed. The measurement time for a single sample, both in water and in the dispersion solution, is 100 s. The result of the entire measurement comprises two PSD histograms, one for water (stable aggregates) and one for the dispersion solution (individual particles). A detailed description of the settings of Mastersizer 3000 can be found in Appendix B.

2.2.2. Calculation of the water stable aggregates index ($WSAi_{SLD}$)

The resulting histogram of the disturbed particles is divided into 100 size classes. Classes larger than 250 μm that were not disintegrated during the sonication process, are excluded from further analysis (20 % of the total size classes), because particles larger than 250 μm are considered to be either stable aggregates or large grains. The water stable aggregates index ($WSAi_{SLD,n}$) is calculated for each particle size class as follows:

$$WSAi_{SLD,n} = \frac{SR_{sol,n}}{SR_{w,n} + SR_{sol,n}} \quad (2)$$

where:

- $WSAi_{SLD,n}$ is the water stable aggregates index of the soil for each size class (-)
- $SR_{sol,n}$ is the volume residue in the n-th size class in the dispersion solution (%)
- $SR_{w,n}$ is the residue in the n-th size class in water (%)

The $WSAi_{SLD,n}$ for all particle size classes are then calculated as weighted mean:

$$WSAi_{SLD,soil} = \frac{\sum_1^n \frac{SR_{sol,n}^2}{SR_{w,n}+SR_{sol,n}}}{\sum_1^n SR_{sol,n}} \cdot 100 \quad (3)$$

where:

- $WSAi_{SLD,soil}$ is the water stable aggregates index of the soil (%)

Eq. (2) is used to calculate the soil water stability index for each size class, and Eq. (3) gives the weighted arithmetic mean of 80 size classes. With this technique, the WSAi can be calculated for any set of selected particle sizes.

Fig. 2 describes the process steps of wet sieving and laser diffraction and shows the similarities between the two methods. Yellow colour highlights the steps that are identical in both approaches.

3. Results

Fig. 3 shows the histograms of the particle size distribution obtained with the SLD technique. The red dashed line represents the same threshold as the Kamper 250 μm sieve used in the wet sieving technique. The data excluded from the WSA calculation is to the right of the dashed line. It is important to note that the x-axis is on a logarithmic scale to make it easier to see the differences between the histograms. The histograms measured either in distilled water or in the dispersion solution represent the average of all replicate measurements in each case. The NUC sample shows partial agreement between the PSD measured in distilled water and in the dispersion solution, indicating that most of the aggregate decay occurred in the distilled water, and that those aggregates are therefore less stable. From this we can predict that the respective sample has a lower WSAi value (see Table 4 for specific values). All soils show the greatest abundance of aggregates that are approximately 200–250 μm in size. Only the soils from NUC and CIC have similar histograms before and after disturbance, while the other soils differ significantly. The ROH and RIS histograms after disturbance are bimodal in character, indicating a high representation of fine particles that were part of the soil aggregates.

The reliability of the obtained data can be assessed using two criteria: either the extent of obscuration, or the calculated residual values. Trustworthy measurements are characterized by obscuration and

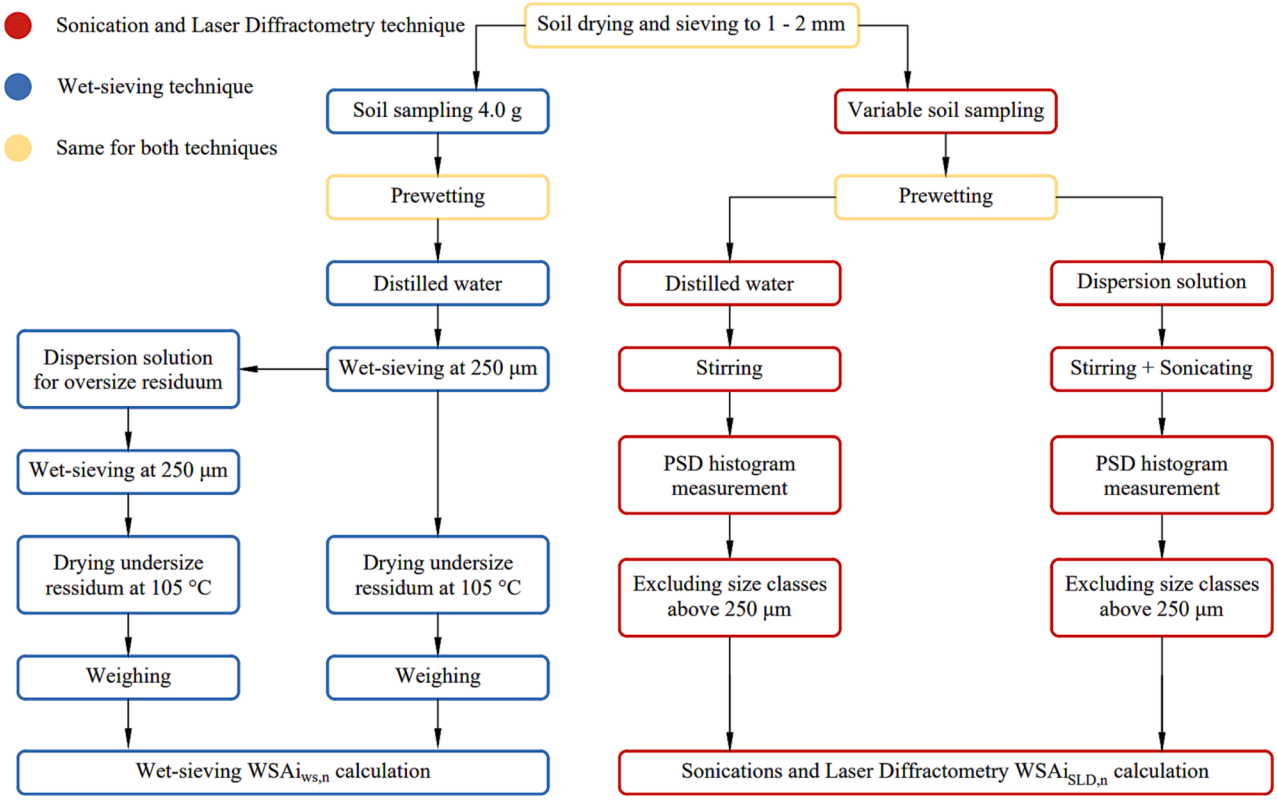


Fig. 2. Aggregate stability flow chart, simultaneous determination of WSAi by Wet-sieving and by the Sonication and Laser Diffractometry approaches.

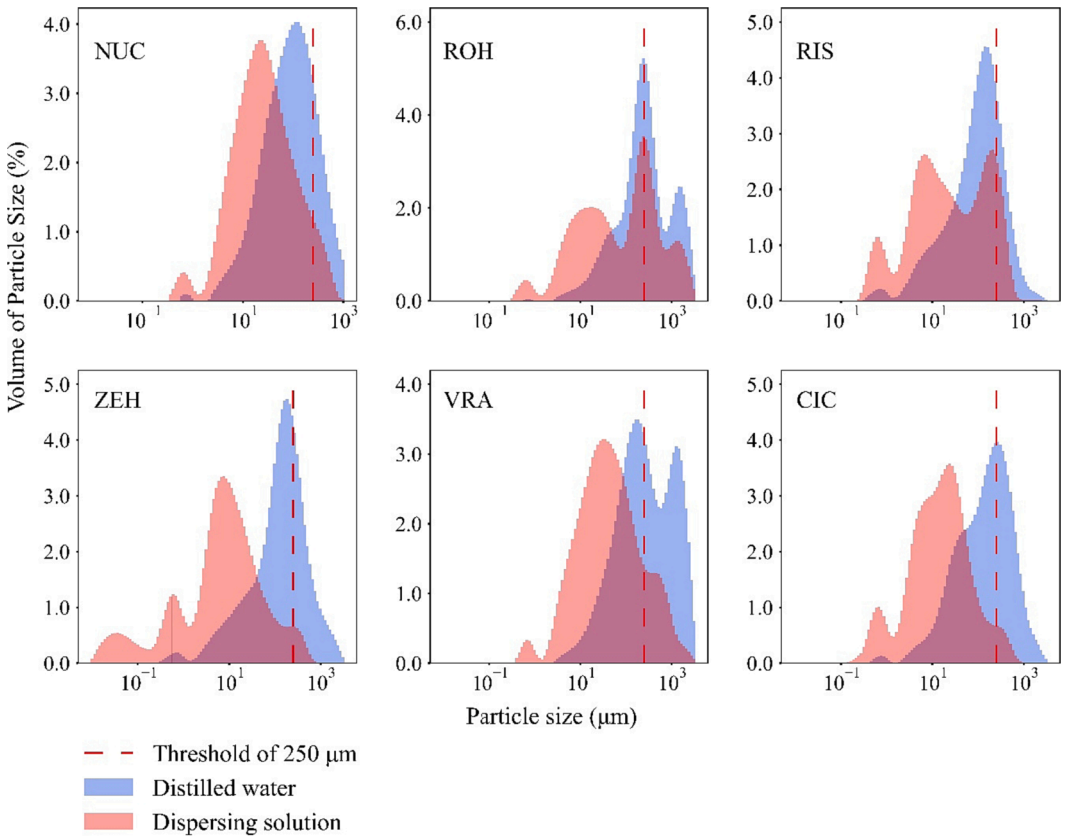


Fig. 3. Histograms of the particle and aggregate size distribution of the tested soils obtained during the sonication and laser diffraction technique, aggregates disrupted in water (blue), aggregates disrupted in the dispersion solution (red). The threshold line separates the particles above 250 μm that are not included in the WSAi calculation.

Table 4
Median soil aggregate water stability indices and standard deviations determined by the laser diffraction technique and by the wet sieving technique, including RMSE and the ANOVA p-value (*RMSE has been calculated for all WSAi values depicted in Fig. 6).

ID	$WSAi_{SLD}$ (%)	$WSAi_{WS}$ (%)
NUC	66.6 ± 0.5	82.5 ± 3
ROH	65.1 ± 0.8	62.9 ± 3.7
RIS	59.7 ± 0.8	48.2 ± 3.7
ZEH	72.9 ± 0.4	67.9 ± 1
VRA	69.2 ± 1	75.5 ± 2.4
CIC	71 ± 0.6	79.7 ± 1.9
RMSE*	1.6	
ANOVA p-value	0.35	

residual values that fall within predefined thresholds. The level of obscuration during the measurement shows quality and reproducibility, while residual values provide insight into the alignment between the data and the model. The level of obscuration during the measurement should be between 10 % and 30 % and the calculated residuals should not exceed 1 %. Fig. 4 shows that the measurements of the stability of the individual aggregates in distilled water and in the dispersion solution show different behaviour. The stability measurements of the aggregates in the dispersion solution show almost no changes in obscuration, due to the perfect decomposition of the aggregates. The particles sizes measurement in distilled water, on the other hand, shows an increase in obscuration. However, this increase in obscuration has no significant influence on the subsequently calculated aggregate stabilities.

Fig. 4 shows that most of the recorded data are within the 10 to 30 % obscuration interval and calculated residuals are below the threshold of 1 %, which means that the measured PSD values are reliable. Measured data that exceeded obscuration threshold of 35 % were excluded from the study.

The whisker plot (Fig. 5) shows the aggregates stability indexes (WSAi) determined using the SLD and WS techniques. The aggregate stabilities calculated with the WS technique have a higher variability than the values calculated with the SLD technique. Fig. 5A shows all WSAi for both methods for the tested soils. Fig. 5B shows a comparison of the ranges of the calculated indexes for all tested soils together. The $WSAi_{SLD}$ calculated from the PSD histograms shows a lower variance between all soils than the $WSAi_{WS}$. This indicates a better reproducibility of the laser diffraction technique, even with a lower number of replicates. The WSAi determined with the WS method has a higher median value (Table 4) than the WSAi determined with SLD.

Statistical analysis of the mean WSAi values was performed using analysis of variance (ANOVA), and the root mean square error (RMSE) between WS and SLD was calculated. The RMSE value of 1.6 % indicates a good match between the WS and SLD. This observation is confirmed by the calculated p-value of 0.35 at a confidence level of 95 %, which indicates that there is not statistically significant difference between the results of the evaluated methods. This statement is also support by the F-test where F-value = 0.90 < Fcrit = 3.95. Scatter diagram (Fig. 6) shows calculated WSAi values by each method, the median values are presented in Table 4.

4. Discussion

The development of the SLD technique was motivated by the ambition to achieve comparability with the WS technique. The results, presented in Table 4 and Fig. 5, show that the median values are similar and there is no significant difference between the mean values of WSA_{iSLD}

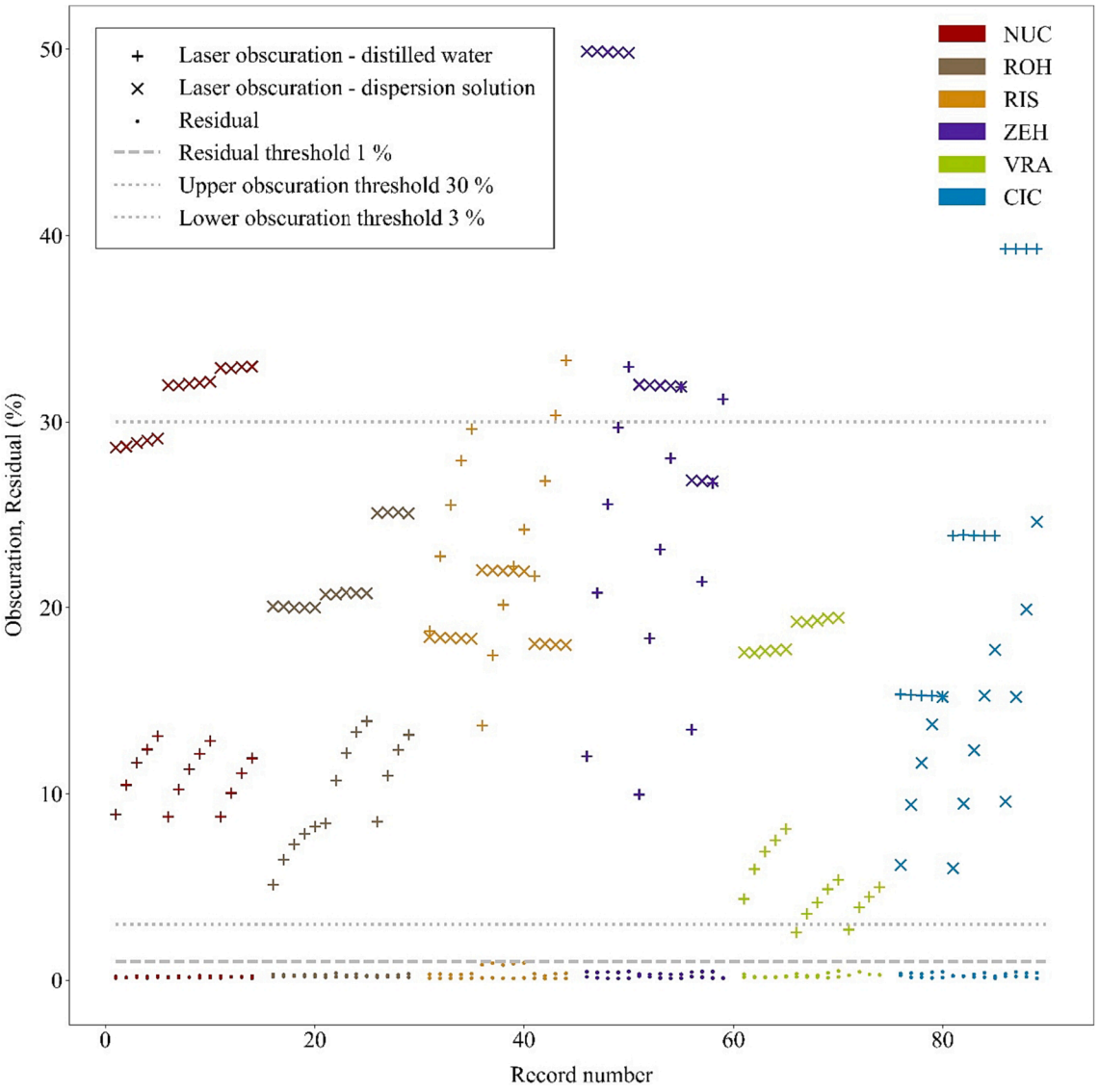


Fig. 4. Adjustment of the measured data, the cross sign stands for the degree of obscuration for the measurements in the dispersion solution, the plus sign stands for the degree of obscuration for the measurements in distilled water, the grey dashed line is the upper limit for the residuals, the grey dotted lines are the lower and upper recommended limits for the obscuration. The soil samples are represented by different colours.

and WSA_{iWS}. In addition, low internal variability of the SLD results (Table 4) has the potential to reduce the number of replicates required. Moreover, the calculated root mean square error (RMSE) of 1.6 % between the aggregate stabilities obtained with SLD or with WS confirms that there is good consistency between the two approaches. Fig. 6 suggests the possibility of applying linear regression to the data. However, doing so might mistakenly indicate a direct relation between SLD and WS, because of the SLD reaches finer resolution. Bieganowski et al. (2018) also investigated aggregate stability of silty loam soils with a use of a laser diffractometer and by wet sieving. Their observations justifies that the linear regression should not be applied, as it could introduce bias.

The obscuration level, a measure of the amount of laser light lost due to the presence of the sample in the analyser beam, is used as a standard quality indicator for measurements with Mastersizer 3000. A general guideline is that the degree of obscuration should be between 3 % and 20 % (Malvern, 2013), although some studies have suggested a more relaxed range of up to 25 % (Sperazza et al., 2004; Ullah et al., 2022). As the mass of the soil sample in 600 ml of water was very high, the obscuration level often reached values as high as 50 %, which is well beyond the recommended upper limit. Further investigation of the SLD technique will include implementation of Hydro EV unit (Malvern Panalytical, United Kingdom) into the protocol. The Hydro EV is a manual unit with a variable volume of water, its use may lead to improvements in the methodology because it allows better control over the level of obscuration.

Wet sieving (WS) is a technique that requires several steps, including mechanical sieving, drying and weighing, which contributes to a somewhat higher variability of results for a single sample. In addition, it is difficult to standardise the exact procedure due to the variety of instruments and approaches used by different laboratories. Measured results may therefore vary from institution to institution. The WS technique is based on the sieve method, where fine soils with a high clay and silt content may lead to the 250 μm sieves clogging. In extreme cases, the sieves are completely blocked, making it impossible to determine the WSAi of the soil.

The new laser diffraction technique (SLD) for determining WSAi has several advantages over conventional techniques. The most important advantages are speed, reproducibility and simplicity. Unlike the wet sieving technique (WS), the SLD technique minimises the number of

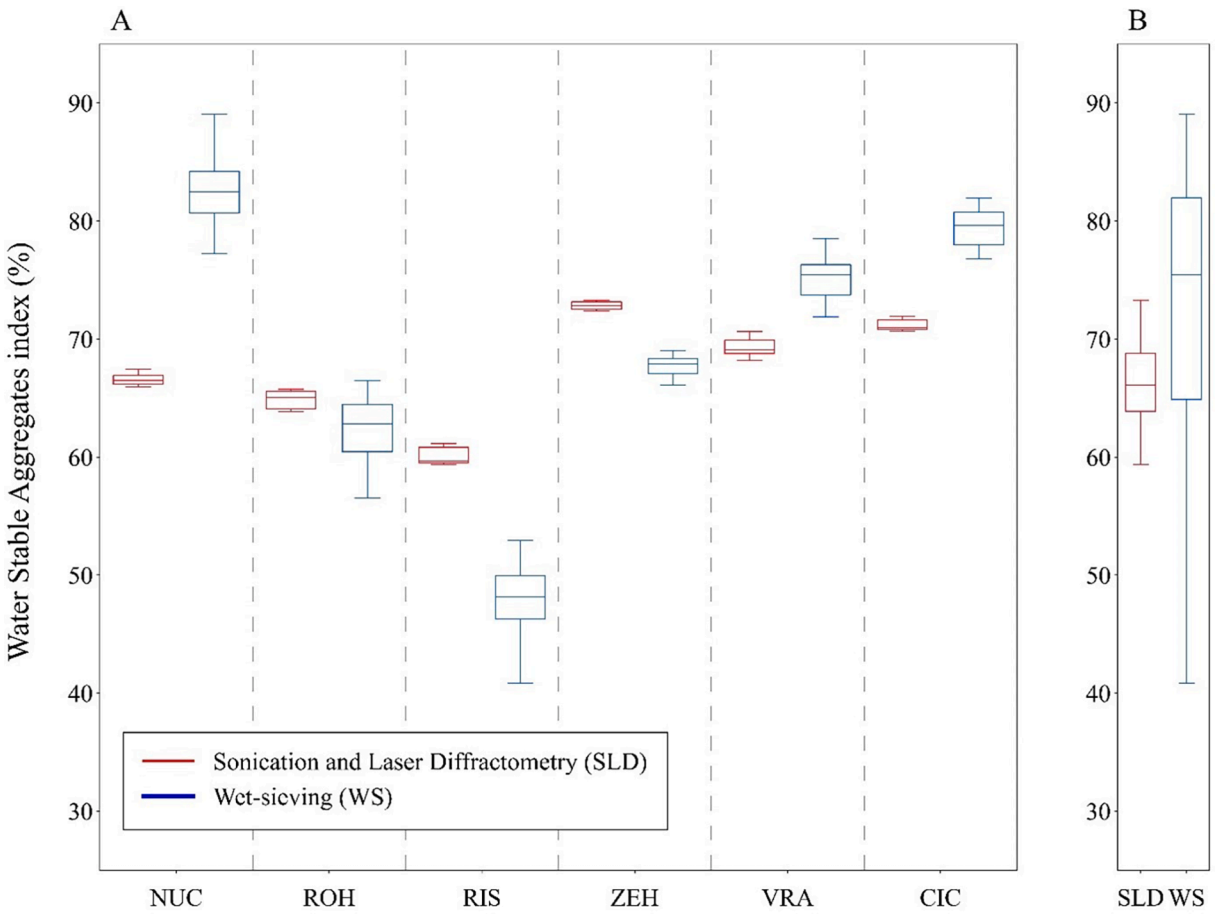


Fig. 5. Distribution of water stable aggregates indexes obtained by SLD (red) and by WS (blue) for different soils. Fig. A shows the WSAi for each soil obtained either by wet-sieving or by laser diffraction, Fig. B shows all WSAi if combined (points – outliers, bottom line – Q1, middle line – median, top line – Q3, interquartile range IQR = Q3 – Q1, upper whiskers = Q3 + 1.5(IQR), lower whiskers = Q1 – 1.5(IQR).

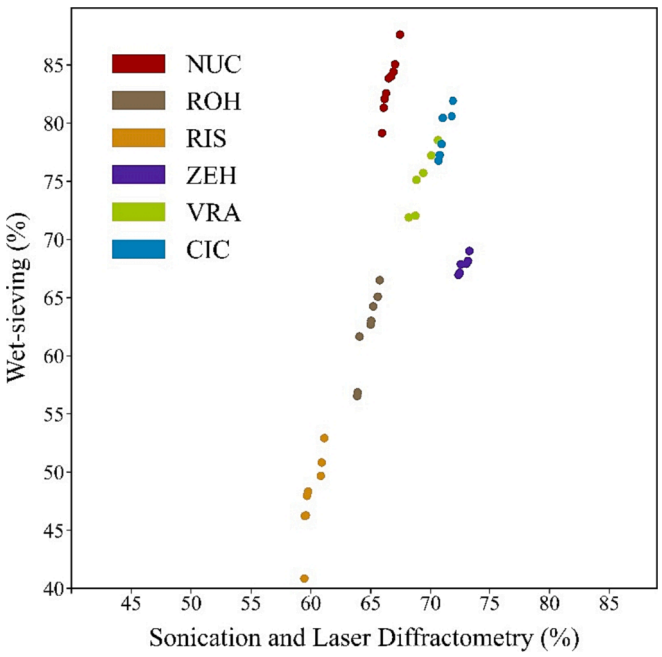


Fig. 6. Scatter diagram of WSAi measured by Sonication and Laser Diffractometry (SLD) and Wet-sieving (WS) methods. Datapoints represent the calculated WSAi.

steps that potentially leads to uncertainties due to human errors. This is mainly because in the SLD analysis the original sample is not manipulated after pre-wetting and insertion into the analyser. The SLD technique is able to provide results within a few minutes, while the WS technique takes up to several days. The SLD technique utilizes 80 % of the measured size classes (below 250 μm) to calculate the aggregate stability. Since the aggregate stabilities for each size class are solved in this way, this technique is less error-prone than the method for calculating stability using the d50 parameter shift (Gyawali and Stewart, 2019). In this study, the 250 μm separation was primarily used to compare SLD and WS results. By excluding this specific feature of the SLD method, it might be possible to explore varied levels of WSAi. This could offer deeper insights into the aggregate stability index, enhancing its relevance as a soil health indicator.

It is also appropriate to point out disadvantages of the SLD technique, and limitations of our study. One drawback is the relatively small number of soil types has been analysed. Soils are very heterogeneous, but the SLD technique was developed and optimized only based on six agricultural soils, all collected in the Czech Republic. Despite the good measurement results, the obscuration in some cases reaches excessively high. The aggregate stability of such soils will therefore not be possible to be measured with the SLD technique, unless necessary adjustments will be made. Finally, it needs to be pointed out that we have only used one manufacturer's diffractometer, while diffractometers from other manufacturers may behave differently and may require different settings.

Determining the stability of soil aggregates requires initial air drying of the soil sample. Until now, no consensus has been reached on the

method for the drying process. The methods vary. Kemper and Rosenau, (1986) suggest leaving the soil in a thin layer with good air circulation for 24 h. (Haynes and Swift, 1990) used a drying oven set at 22 °C for 48 h, (Almajmaie et al., 2017) dried the sample at 40 °C for 24 h, and (Utomo and Dexter, 1982) left the aggregates exposed at the room temperature and humidity for 10 days. The duration of the drying process has a significant impact on the stability of the aggregates. Studies by Churchman and Tate (1987), Kemper and Rosenau (1986) and Murer et al. (1993) have shown that air-dried aggregates have higher stability than wet aggregates, and that stability increases with the drying duration.. In our study many analyses have been performed on samples of different ages. Although we do not report different drying tests in this study, based on our unpublished results we suggest storing and drying the aggregates in a thin layer at 20 to 30 °C for at least one day, but no longer than 10 days, to ensure accurate measurements. To ensure reliable and comparable results, it is recommended to perform the analysis immediately after the drying.

5. Conclusions

This study has scientifically tested a new technique to quantify soil aggregate stability. In order to harmonize the procedure with the standard Wet Sieving technique (WS) the Sonication and Laser Diffractometry technique (SLD) utilizes the same limit of 250 μm for separation of grains and stable aggregates. However, it is possible to work with an alternative boundary in the SLD technique and thus investigate the stability of aggregates of various sizes. Since the process of disintegration of soil aggregates during SLD measurements operates on the principle of ultrasound and particle movement through the instrument, SLD is also suitable for fine soils, where the WS technique cannot be applied, e.g. due to clogging of the sieves.

The water stable aggregates index (WSAi) obtained with the SLD technique is calculated by dividing the particle size distribution of the soil aggregates in distilled water and in a dispersion solution. Since both techniques are based on the measurement of physical properties, they are comparable.

In summary the SLD technique produced comparable results with WS. However, SLD based aggregate stability measurement is a novel technique, it requires to be further tested especially to evaluate its usability on other soil types, laser diffractometers. Future studies may explore improvements to the methodology, such as testing with a manual unit with variable water volume. To ensure accurate and comparable results across laboratories, it is important to standardize the methods and procedures used to determine WSAi.

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.

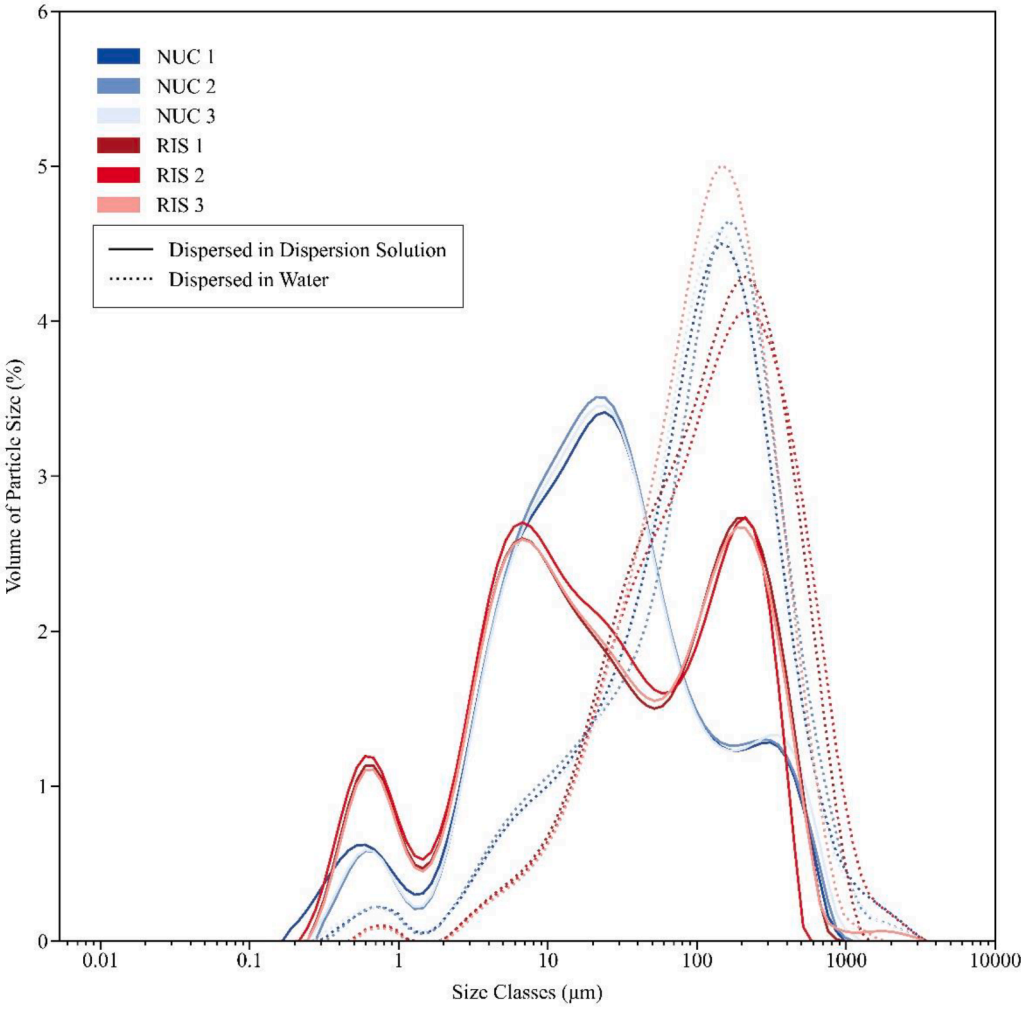
Data availability

Data will be made available on request.

Acknowledgement

This study has been supported by the Grant Agency of the Czech Technical University in Prague grant No. SGS23/155/OHK1/3T/11, European Commission Horizon 2020 grant No. 101000224 (TUdi) and Ministry of Education, Youth and Sports of the Czech Republic grant No. LTAUSA19019.

Appendix A – Repeatability of Mastersizer 3000 measurements for two selected samples RIS (shades of red) and NUC (shades of blue)



Appendix B – Mastersizer 3000 settings for measurements in distilled water and dispersion solution

Setting	Distilled Water	Dispersion Solution
Particle Type		
Non-spherical particle mode	Yes	Yes
Fraunhofer type	No	No
Material properties		
Material name	Silica SiO2	Silica SiO3
Refractive index	1.457	1.457
Absorption index	0.01	0.01
Particle density	2.64 g/cm^3	2.64 g/cm^3
Different optical properties in blue light	No	No
Dispersant properties		
Dispersant name	Water	Water
Refractive index	1.33	1.33
Level sensor threshold	100	100
Measurement duration		
Background measurement duration (red)	20.00 s	20.00 s
Sample measurement duration (red)	10.00 s	10.00 s
Perform blue light measurement?	Yes	Yes
Background measurement duration (blue)	20.00 s	20.00 s
Sample measurement duration (blue)	10.00 s	10.00 s

(*continued on next page*)

(*continued*)

Setting	Distilled Water	Dispersion Solution
Assess light background stability	No	No
Measurement sequence		
Aliquots	1	1
Automatic number of measurements	No	No
Pre-alignment delay	0.00 s	0.00 s
Number of measurements	5	5
Delay between measurements	0.00 s	0.00 s
Pre-measurement delay	0.00 s	0.00 s
Close measurement window after measurement	No	No
Measurement obscuration settings		
Auto start measurement	Yes	Yes
Obscuration low limit	3.00 %	3.00 %
Obscuration high limit	30.00 %	30.00 %
Enable obscuration filtering	No	No
Measurement alarms		
Use Background Check	No	No
Background Check Limits	[1;200];[20;60]	[1;200];[20;60]
Accessory control settings		
Accessory name	Hydro LV	Hydro LV
Is accessory dry?	No	No
Stirrer speed	2500 rpm	2500 rpm
Ultrasound percentage	0 %	100 %
Fill Dispersant Source Identifier	Auto	Auto
Manual tank fill?	No	No
Degas after tank and cell fill	Yes	Yes
Sonicate to stability?	No	No
Ultrasound mode	None	Pre-Measurement
Degas after pre-measurement ultrasound	None	No
Align after pre-measurement ultrasound	None	No
Ultrasonication duration	None	120.00 s
Clean sequence settings		
Clean sequence type	Quick	Quick
Sonicate during clean?	No	No
Manually Fill Tank During Clean?	No	No
Clean Dispersant Source Identifier	Auto	Auto
Clean Dispersant Level Sensor Threshold	0	0
Degas After Clean?	No	No
Analysis settings		
Analysis model	General Purpose	General Purpose
Single result mode	No	No
Number of killed inner detectors	0	0
Blue light detectors killed	No	No
Fine powder mode	No	No
Analysis sensitivity	Normal	Normal
Analysed as Mastersizer 3000E?	No	No
Result Settings		
Result range is limited	No	No
Result Units	Volume	Volume
Extend Result	No	No
Result Emulation	No	No
User sizes for histograms and tables		
Use user sizes	No	No
Data export output		
Enabled?	No	No
Averaging		
Averaging enabled?	No	No
Printing options		
Printing enabled?	No	No

References

Almajmaie, A., Hardie, M., Acuna, T., Birch, C., 2017. Evaluation of methods for determining soil aggregate stability. Soil Tillage Res. 167, 39–45.

Amézketa, E., 1999. Soil aggregate stability: A review. J. Sustain. Agric. 14, 83–151.

Amezketa, E., Aragües, R., Carranza, R. Urgel, B., 2003. Macro- and micro-aggregate stability of soils determined by a combination of wet-sieving and laser-ray diffraction. Spanish J. Agric. Res., 1, 83, (At: https://www.researchgate.net/publication/267366197_Macro-and_micro-aggregate_stability_of_soils_determined_by_a_combination_of_wet-sieving_and_laser-ray_diffraction. Accessed: 7/10/2022).

An, S., Mentler, A., Mayer, H., Blum, W.E.H., 2010. Soil aggregation, aggregate stability, organic carbon and nitrogen in different soil aggregate fractions under forest and shrub vegetation on the Loess Plateau, China. Catena 81, 226–233.

Bieganowski, A., Zaleski, T., Kajdas, B., Sochan, A., Józefowska, A., Beczek, M., Lipiec, J., Turski, M., Ryżak, M., 2018. An improved method for determination of aggregate stability using laser diffraction. Land Degrad. Dev. 29, 1376–1384, (At: https://onlinelibrary.wiley.com/doi/full/10.1002/ldr.2941. Accessed: 7/10/2022).

Bieganowski, A., Ryżak, M., Witkowska-Walczak, B., 2010. Determination of soil aggregate disintegration dynamics using laser diffraction. Clay Miner. 45, 23–34.

Blakemore, L.C., Searle, P.L., Daly, B.K., 1987. Methods for chemical analysis of soils. NZ Soil Bureau Scientific Report.

Blott, S.J., Pye, K., 2006. Particle size distribution analysis of sand-sized particles by laser diffraction: An experimental investigation of instrument sensitivity and the effects of particle shape. Sedimentology 53, 671–685.

Chaney, K., Swift, R.S., 1984. The influence of organic matter on aggregate stability in some British soils. J. Soil Sci., 35, 223–230, (At: https://www.academia.edu/31408139/The_influence_of_organic_matter_on_aggregate_stability_in_some_British_soils. Accessed: 7/10/2022).

CHMU, 2019. I v Česku se mění klima. (At: https://storymaps.arcgis.com/stories/623ff16d5dd54607a1a272539aa5dda2. Accessed: 20/2/2023).

Churchman, G.J., Tate, K.R., 1987. Stability of aggregates of different size grades in allophanic soils from volcanic ash in New Zealand. J. Soil Sci. 38, 19–27.

Dexter, A.R., 1988. Advances in characterization of soil structure. Soil Tillage Res. 11, 199–238.

Emerson, W.W., 1967. A classification of soil aggregates based on their coherence in water. Soil Res., 5, 47–57, (At: https://www.publish.csiro.au/sr/sr9670047. Accessed: 7/10/2022).

Fajardo, M., McBratney, A.B., Field, D.J., Minasny, B., 2016. Soil slaking assessment using image recognition. Soil Tillage Res. 163, 119–129.

Flynn, K.D., Bagnall, D.K., Morgan, C.L.S., 2020. Evaluation of SLAKES, a smartphone application for quantifying aggregate stability, in high-clay soils. Soil Sci. Soc. Am. J. 84, 345–353.

Franzluebbers, A.J., Wright, S.F., Stuedemann, J.A., 2000. Soil Aggregation and Glomalin under Pastures in the Southern Piedmont USA. Soil Sci. Soc. Am. J. 64, 1018–1026.

Fristensky, A., Grismer, M.E., 2008. A simultaneous model for ultrasonic aggregate stability assessment. Catena 74, 153–164.

Gabas, N., Hiquily, N., Lagucrie, C., 1994. Response of Laser Diffraction Particle Sizer to Anisometric Particles.

Gyawali, A.J., Stewart, R.D. 2019. An improved method for quantifying soil aggregate stability. Soil Sci. Soc. Am. J., 83, 27–36, (At: https://onlinelibrary.wiley.com/doi/full/10.2136/sssaj2018.06.0235. Accessed: 7/10/2022).

Haynes, R.J., Swift, R.S., 1990. Stability of soil aggregates in relation to organic constituents and soil water content. J. Soil Sci. 41, 73–83, (At: https://onlinelibrary.wiley.com/doi/full/10.1111/j.1365-2389.1990.tb00046.x. Accessed: 7/10/2022).

Herrick, J.E., Whitford, W.G., de Soyza, A.G., Van Zee, J.W., Havstad, K.M., Seybold, C. A., Walton, M., 2001. Field soil aggregate stability kit for soil quality and rangeland health evaluations. CATENA, 44, 27–35, (At: https://linkinghub.elsevier.com/retrieve/pii/S0341816200001739. Accessed: 29/11/2022).

IUSS Working Group WRB, 2015. World reference base for soil resources 2014, update 2015 International soil classification system for naming soils and creating legends for soil maps. FAO.

Karami, A., Homaee, M., Afzalinia, S., Ruhipour, H., Basirat, S., 2012. Organic resource management: Impacts on soil aggregate stability and other soil physico-chemical properties. Agr Ecosyst Environ 148, 22–28.

Kasper, M., Buchan, G.D., Mentler, A., Blum, W.E.H., 2009. Influence of soil tillage systems on aggregate stability and the distribution of C and N in different aggregate fractions. Soil Tillage Res. 105, 192–199.

Kemper, W.D., Koch, E.J. 1966. Aggregate Stability of Soils from Western United States and Canada. Tech. Bull., (At: https://ideas.repec.org/p/ags/uerstb/171386.html. Accessed: 7/10/2022).

Kemper, W.D., Rosenau, R.C., 1986. 7 Aggregate Stability and Size Dlstributlon'.

Le Bissonnais, Y., 1996. Aggregate stability and assessment of soil crustability and erodibility: I. Theory and methodology.

Malvern. 2013. Manual: Mastersizer 3000 User Manual (English) MAN0474. (At: www.malvern.com).

Moebius-Clune, B.N. 2017. Comprehensive assessment of soil health: The Cornell framework manual.

Morgan, C.L.S., 2020. Assessing Soil Health: Soil Water Cycling. Crops & Soils, 53, 35–41, (At: https://onlinelibrary.wiley.com/doi/full/10.1002/crso.20064. Accessed: 30/1/2023).

Murer, E.J., Baumgarten, A., Eder, G., Gerzabek, M.H., Kandeler, E., Rampazzo, N., 1993. An improved sieving machine for estimation of soil aggregate stability (SAS). Geoderma 56, 539–547.

Rieke, E.L., Bagnall, D.K., Morgan, C.L.S., Flynn, K.D., Howe, J.A., et al., 2022. Evaluation of aggregate stability methods for soil health. Geoderma 428, 116156.

Robinson, D.O., Page, J.B. 1951. Soil aggregate stability. Soil Sci. Soc. Am. J. 15, 25–29, (At: https://onlinelibrary.wiley.com/doi/full/10.2136/sssaj1951.036159950015000C0005x. Accessed: 25/9/2023).

Six, J., Feller, C., Denef, K., Ogle, S.M., De Moraes Sa, J.C., Albrecht, A., 2002. Soil organic matter, biota and aggregation in temperate and tropical soils – Effects of no-tillage. Agronomie 22, 755–775. https://doi.org/10.1051/agro:2002043. Accessed: 7/10/2022.

Six, J., Bossuyt, H., Degryze, S., Denef, K., 2004. A history of research on the link between (micro)aggregates, soil biota, and soil organic matter dynamics. Soil Tillage Res. 79, 7–31.

Sparling, G.P., Hart, P.B.S., August, J.A., Leslie, D.M., 1994. A comparison of soil and microbial carbon, nitrogen, and phosphorus contents, and macro-aggregate stability of a soil under native forest and after clearance for pastures and plantation forest. Biol. Fertility Soils 1994 17:2, 17, 91–100, (At: https://link.springer.com/article/10.1007/BF00337739. Accessed: 7/10/2022).

Sperazza, M., Moore, J.N., Hendrix, M.S., 2004. High-resolution particle size analysis of naturally occurring very fine-grained sediment through laser diffractometry. SEPM (Society for Sedimentary Geology.

Tisdall, J.M., Oades, J.M., 1982. Organic matter and water-stable aggregates in soils, J. Soil Sci., 33, 141–163, (At: https://onlinelibrary.wiley.com/doi/full/10.1111/j.1365-2389.1982.tb01755.x. Accessed: 7/10/2022).

Ullah, R., Abdullah, R.A., Kassim, A., Yunus, N.Z.M., Ullah, A., Junaid, M., Roshan, M.J., 2022. Effectiveness of laser diffraction method for particle size evaluation of residual soil. Indian Geotech. J. https://link.springer.com/10.1007/s40098-022-00615-1.

Utomo, W.H., Dexter, A.R., 1982. Changes in soil aggregate water stability induced by wetting and drying cycles in non-saturated soil.

Virto, I., Gartzia-Bengoetxea, N., Fernández-Ugalde, O., 2011. Role of organic matter and carbonates in soil aggregation estimated using laser diffractometry. Pedosphere 21, 566–572.

VUMOP, 2017. Protierozní kalkulačka. (At: https://kalkulacka.vumop.cz/?core=account. Accessed: 20/2/2023).

Wriedt, T., 2012. Mie theory: A review. Springer Ser. Opt. Sci. 169, 53–71.