Experiences of soil physical measurements with laser diffractometer and their application possibilities in soil water management research

András Makó[1,2*], Magdalena Ryżak[3], Gyöngyi Barna[1], Cezary Polakowski[3], Kálmán Rajkai[1], Zsófia Bakacsi[1], Hilda Hernádi[2], Michał Beczek[3], Agata Sochan[3], Rafał Mazur[3], Mihály Kocsis[2], Andrzej Bieganowski[3]

[1]Department of Soil Physics and Water Management, Institute for Soil Sciences,
Centre for Agricultural Research, Budapest, Hungary
[2] Department of Environmental Sustainability (Georgikon Campus), Institute of Environmental Sciences,
Hungarian University of Agriculture and Life Sciences, Keszthely, Hungary
[3]Institute of Agrophysics Polish Academy of Sciences, Lublin, Poland

Summary

In this study, we presented the experience of two high-speed laser diffractometry methods for measuring particle size distribution (PSD) and microaggregate stability (MiAS%) of soils, which parameters have a significant influence on the soil water management properties. PSD results obtained with sieve-pipette and laser diffractometry method were compared on a continental (LUCAS), a national (HunSSD) and a regional (TOKAJ) database. We found significant differences between the results of the two methods at all three scales. When the clay/silt boundary was modified to 7 μm for the LDM, significantly better results were obtained. The LDM was also suitable for the determination of the MiAS% of soils, which was influenced mainly by organic matter, pH and exchangeable Na^+ content of soils.

Keywords: laser diffractometry, particle size distribution, aggregate stability

A lézerdiffraktometriás talajfizikai mérések tapasztalatai és ezek alkalmazása a talaj vízgazdálkodási kutatásban

Makó András[1,2*], Magdalena Ryżak[3], Barna Gyöngyi[1], Cezary Polakowski[3], Rajkai Kálmán[1], Bakacsi Zsófia[1], Hernádi Hilda[2], Michał Beczek[3], Agata Sochan[3], Rafał Mazur[3], Kocsis Mihály[2], Andrzej Bieganowski[3]

[1]ELKH ATK TAKI Talajfizikai és Vízgazdálkodási Osztály, Budapest, Magyarország
[2]MATE KÖTI Környezeti Fenntarthatósági Tanszék, Keszthely, Magyarország
[3]Lengyel Tudományos Akadémia Agrofizikai Kutatóintézet, Lublin, Lengyelország

Összefoglaló

Tanulmányunkban a lézerdiffraktométerrel végzett talajfizikai mérések tapasztalatait és alkalmazási lehetőségeit vizsgáltuk a vízgazdálkodási kutatásokban. A talajok mechanikai összetétele, azaz az elemi talajrészecskék méret szerinti százalékos eloszlása, az egyik legfontosabb talajfizikai paraméter, mely számos egyéb tulajdonságot, így a talajok szerkezetét, vízgazdálkodását befolyásolja. Meghatározása több módon történhet: pl. a hagyományos szitás-pipettás ülepítéses módszerrel (SZPM), vagy az egyik legmodernebbnek számító lézerdiffraktométeres (LDM) eljárással. Kutatásunk során e kétféle módszerrel kapott mechanikai összetétel eredményeket három nagyobb adatbázison hasonlítottuk össze: egy kontinentális (LUCAS), egy hazai (HunSSD) és egy regionális (TOKAJ) adatállományon.

Azt tapasztaltuk mindhárom adatbázis esetében, hogy a lézerdiffrakciós vizsgálatok az agyagtartalmat alulbecslik a pipettás módszerrel kapott eredményekhez képest, míg a portartalmat felülbecslik (az adatsorok eltérését jellemző RMSE értékek az agyagfrakciókra: 16,30; 19,29 és 24,97; a porfrakciókra: 15,68; 19,82 és 26,95. A homoktartalmak közt lényegesen kisebb eltéréseket tapasztaltunk (RMSE: 7,26; 9,25 és 5,25 a három adatbázis esetében). Ha azonban az LDM vizsgálati eredményeknél módosítottuk az agyag és a por frakció mérethatárát 2 µm-ről 7 µm-re, szignifikánsan jobb eredményeket kaptunk az összehasonlítás során mind az agyagtartalom (RMSE: 8,99; 6,77 és 6,54), mind a portartalom esetében (RMSE: 8,87; 7,46 és 5,74). A különböző módszerekkel mért és számított PSD eredményeket textúra háromszög diagramokon is ábrázoltunk.

A lézerdiffrakciós eljárás alkalmas a talajok mikroaggregátum stabilitásának (MiAS%) meghatározására is, melyet a HunSSD hazai adatbázis talajain mutattunk be. Megállapítottuk, hogy erős, szignifikánsan pozitív kapcsolat van a MiAS% és a talaj szervesanyag-tartalma között; és erős, szignifikánsan negatív kapcsolat a mikroaggregátumok stabilitása és a pH, a mésztartalom, a sótartalom és a kicserélhető nátriumtartalom között.

A tanulmányban vizsgált talajfizikai tulajdonságok szorosan összefüggnek a talajok vízgazdálkodási tulajdonságaival. A talajok vízgazdálkodási tulajdonságait jellemző hidrofizikai paraméterek (víztartó képesség, vízvezető képesség) becslése általában a mechanikai összetétel adatok felhasználásával történik. A becslési módszerek (pedotranszfer függvények) pontosítására egyre gyakrabban figyelembe veszik a talaj szerkezeti tulajdonságait is. Amennyiben gyorsan és megbízhatóan tudjuk mérni a talajok mechanikai összetételét és aggregátum-stabilitását lézerdiffrakciós módszerekkel, akkor a hidrofizikai tulajdonságok becsléséhez szükséges input adatokat is gyorsan, nagyobb számban tudjuk előállítani, így a talajok vízgazdálkodását is több minta alapján, nagyobb részletességgel, megbízhatóbban tudjuk jellemezni.

Kulcsszavak: lézer diffraktometria, mechanikai összetétel, aggregátum stabilitás

Introduction

The main objective of the new EU water policy is to achieve good status of surface and groundwater resources in the Member States, as far as possible, based on available scientific knowledge. The general objectives of Directive 2000/60/EC on Community action in the field of water protection (the Water Framework Directive or WFD) include, in addition to the protection of freshwater resources, the protection and improvement of terrestrial ecosystems directly dependent on and linked to water *(EC 2000; Rekolainen–Kämäri–Hiltunen 2003)*. Accordingly, the WFD provides, inter alia, for the assessment and conservation of exploitable water resources, the discharge of pollutants affecting water resources, the progressive reduction of pollution and the mitigation of the adverse effects of floods, waterlogging and droughts. It aims to regulate the quantity and quality of fresh water together and to plan for the achievement of sustainable 'good status' in the long term (drawing up river basin management plans; developing indicators of water scarcity and drought, risk maps, etc.). The WFD does not directly address the management of water resources in specific economic sectors, such as agriculture (e.g. mitigation of drought damage, problems of quantity and quality of irrigation water, impact of waterlogging, floods, etc.) *(Kaika 2003; Grygoruk–Okruszko 2015; Giakoumis–Voulvoulis 2019)*.

From the point of view of agricultural water management, it is important to note that the WFD only discusses and regulates the concepts of 'surface and groundwater bodies' and 'heavily modified water bodies' in relation to water resources, and does not address the properties of variable water saturation layers of soil in close contact with surface and groundwater, nor the quantity and quality of the water stored there. However, the hydrophysical properties of soils (water absorption, water conductivity and water holding capacity), which depend on the structure and stratification of the soil and on the chemical, physical, mineralogical and biological properties of the individual layers, are decisive factors both for the water resources available for agricultural production and for the soil degradation processes associated with extreme water supply situations (floods, waterlogging, drought) *(Volk–Liersch –Schmidt 2009; Rajkai et al. 2018)*.

In agricultural aspects, one of the most important consequences of climate change linked to greenhouse gases and land-use change is the increasing frequency of these extreme water situations. The most important input parameters for water management models using different 'climate scenarios' for the future are measured or estimated hydrophysical parameters. The results of the model runs also support the empirical observations that the same weather conditions on different soils and with different crops grown on them can have various consequences depending on the soil properties ('sensitivity to drought or waterlogging') *(Kocsis et al. 2020; Steinfeld et al. 2020)*. In the field of agriculture and the environment, the combined environmental effects of, for example, physically and biologically degraded, nutrient-poor soils during drought, or soils saturated with water and contaminated with some pollutant at the same time, are of particular importance *(Makó–Hernádi 2012)*. *It is clear from the above that the knowledge of soil hydrophysical properties (SHP) and the development of related measurement or estimation methods are becoming increasingly important for the planning and conservation of agricultural water resources.*

SHP can be influenced by a number of soil properties, such as the particle size distribution (PSD) of the soils (or soil texture), bulk density, organic matter content or other routinely measured properties such as soil lime content, pH, exchangeable Na⁺ content and total salt content, which may hydrolyse differently, depending on the chemical and the mineral composition of soils or their water repellency (hydrophobic character) *(Rajkai et al. 2018)*. Soil structure is one of the most important soil factors affecting SHP, and all the soil parameters mentioned above are in some way related to soil structure, so that these soil properties may influence *SHP* not only directly but also indirectly through their effects on aggregate formation and stabilisation/destabilisation *(Kögel-Knaber et al. 2008; Józefaciuk–Czachor 2014)*.

In addition to the soil properties listed above, a number of natural (abiotic and biotic) and anthropogenic factors can also affect SHP. Natural factors include soil-forming rocks, structural changes due to climate (variability), periodic soil water cover, forest fires. Among anthropogenic factors, the cropping system applied to the soil, in particular the tillage regime, is the most important, but hydrophysical properties can be affected by the use of high organic matter additives in crop production, fertilizers applied during crop production, organic compounds that reduce the surface tension of water, surfactants, soil conditioning agents, chemical soil amendments (e.g. lime or gypsum) or irrigation with irrigation water of different origin and quality *(Rajkai et al. 2018)*.

In general, it can be concluded that the hydrophysical properties of soils are mainly related to the pore system (total porosity, pore size distribution), the development and change of which depends mainly on the PSD and structural conditions (particle morphology, aggregate size distribution and aggregate stability) of the soils. The other factors affecting SHP are also largely influenced through changes in soil structure.

Soil hydrophysical properties can be determined by various measurement methods, but these are time-consuming and expensive. Therefore, in practice, they are often estimated from more easily measured and/or routinely surveyed soil data, such as PSD, organic carbon content and bulk density *(Wösten–Pachepsky–Rawls 2001)*. The estimator functions are called pedotransfer functions (PTFs) in the literature *(Bouma 1989)*. Today, the development of PTFs estimating soil hydrophysical properties has become one of the most important branches of soil physics research. Over the last three decades, several soil water management and soil physics databases have been established worldwide that are suitable for the development of pedotransfer functions. The most significant of these are GRIZZLY, UNSODA, NRCS-NSSC, IGBT-DIS, WISE, HYPRES, EU-HYDI *(Weynants et al. 2013; Tóth et al. 2015)*, and the largest Hungarian soil physical database is called MARTHA (acronym of 'Detailed Hungarian Soil Physical and Hydrological Database') *(Makó et al. 2010)*. The most commonly used independent variables in the PTFs developed from these databases are from PSD data.

Most of the commonly used PSD measurements are based on Stokes' law and they are called sedimentation methods, such as the pipette and hydrometer methods. The pipette and hydrometer methods give comparable results when similar pre-treatment techniques are used *(Liu et al. 1966)*. The sedimentation-based methods require relatively large amount of samples (20 to 40 grams) and their capacity for resolution to subgroups of sizes is limited. A new and increasingly common test method for measuring PSD is the laser diffractometry method (LDM) *(Bieganowski et al. 2018a)*. There are several papers describing comparisons of the LDM with the sieve-pipette method (SPM) *(Loizeau et al. 1994; Konert–Vandenberghe 1997; Beuselinck et al. 1998; Eshel et al. 2004; Arriga–Lowery–Mays 2006; Taubner–Roth–Tippkötter 2009; Igaz et al. 2020; Bai et al. 2021)* and hydrometer method (HM) *(Ryżak–Walczak–Niewczas 2004; Ryżak–Bieganowski 2010; Jedari et al. 2020; Sedláčková–Ševelová 2021)*. Literature review has led to the conclusion that LDM tends to underestimate the amount of clay fraction. This underestimation is attributed mainly to the shape of particles being not spherical *(Matsuyama–Yamamoto 2004; Blott–Pye 2006; Fedotov et al. 2007; Polakowski et al. 2014)*. These studies stated that the different results might be related to the selected optical parameters or to the measurement limits in case of older apparatus. Apart from these reasons, the lack of comparability may also come from the uncertainty of the used LDM apparatus, the applied pre-treatments and settings *(Ryżak–Bieganowski 2011; Madarász et al. 2012; Sochan et al. 2012; Makó et al. 2014; Koza et al. 2021)*. Antinoro et al. (*2012*) tested the effects of PSD measurement method (sedimentation and LDM) on the prediction of the water retention capacity. LDM-measured PSD may be directly used for PTF developments *(Lamorski et al. 2014)*, or for water balance modelling *(Szecsődi et al. 2021)*. The comparability of the results, obtained by different measurement methods, and data harmonisation are also an important issue, because it is not always the same which PSD data in the databases on which PTFs are based, and derived from which measurements, and how uniform they are. The reliability, accuracy and usability of PTFs may depend on all of these.

Based on the foregoing, the structural characteristics of soils can also determine to a large extent the hydrophysical properties of soils. Such soil structural characteristics include not only the shape and size distribution of aggregates but also their stability.

There are several methods for testing the stability of aggregates. These can be grouped, for example, according to the size of the aggregates to be tested. Macroaggregates are generally defined as aggregates larger than 250 microns. Their stability is most often determined by

the so-called wet sieving method *(Kemper–Rosenau 1986; Le Bissonnais 1996a)*.

When measuring the stability of micro-aggregates (aggregates smaller than 250 microns), the fate (dispersion/flocculation) of the clay fraction of the soils is measured primarily. The measurement methods focus mainly on the quantification of the clay particle size or on the total PSD curves of the soils. The choice of appropriate preparations (wetting in water only, wetting and shaking, sonication or chemical dispersion, wetting in liquids of different polarity) can be used to vary the decomposition forces *(Amézketa 1999)*. Since the last century, sedimentation-type PSD methods have been used to measure the stability of these microaggregates *(Kachinsky 1965; Abu-Sharar–Bingham–Rhoades 1987)*. More recently, the laser diffraction measurement technique has been proposed to characterize the structural state of micro-aggregates, since LDM PSD measurements are significantly faster, require less sample amount and give a complete grain size distribution, than sedimentation techniques *(Levy et al. 1993; Bieganowski–Ryżak–Witkowska-Walczak 2010; Bieganowski et al. 2018b)*. Various indicators are known to characterise the stability of microaggregates. The dispersion indices express the ratio of the amount of clay content measured under different dispersion conditions. Some authors define the ratio of the geometric mean diameters of non-dispersed and dispersed soil particles in the suspensions. The decomposition dynamics of microaggregates can be informed by expressing the change in the clay, silt and sand fraction of the soils or median or geometric mean diameter of the PSD as a function of decomposition time *(Slattery–Burt 1997; Beuselinck–Govers–Poesen 1999; Mason et al. 2003; Fristensky–Grismer 2009; Bieganowski–Ryżak–Witkowska-Walczak 2010; Bortoluzzi et al. 2010; Mason–Greene–Joeckel 2011; Virto-Gartzia-Bengoetxea–Fernández-Ugalde 2011)*.

In our research we were looking for answers to the following questions: (1) To what extent does the particle size distribution (PSD) of soils, the soil property most often considered in the estimation of soil hydrophysical parameters (water retention and conductivity), depend on the measurement methodology used? (2) How can results from different measurement methods be compared and how can PSD data from different data sources be harmonised within databases to be used for estimating hydrophysical properties? (3) How can soil structure, which has a significant influence on soil hydrophysical parameters, be characterised using novel measurement methods?
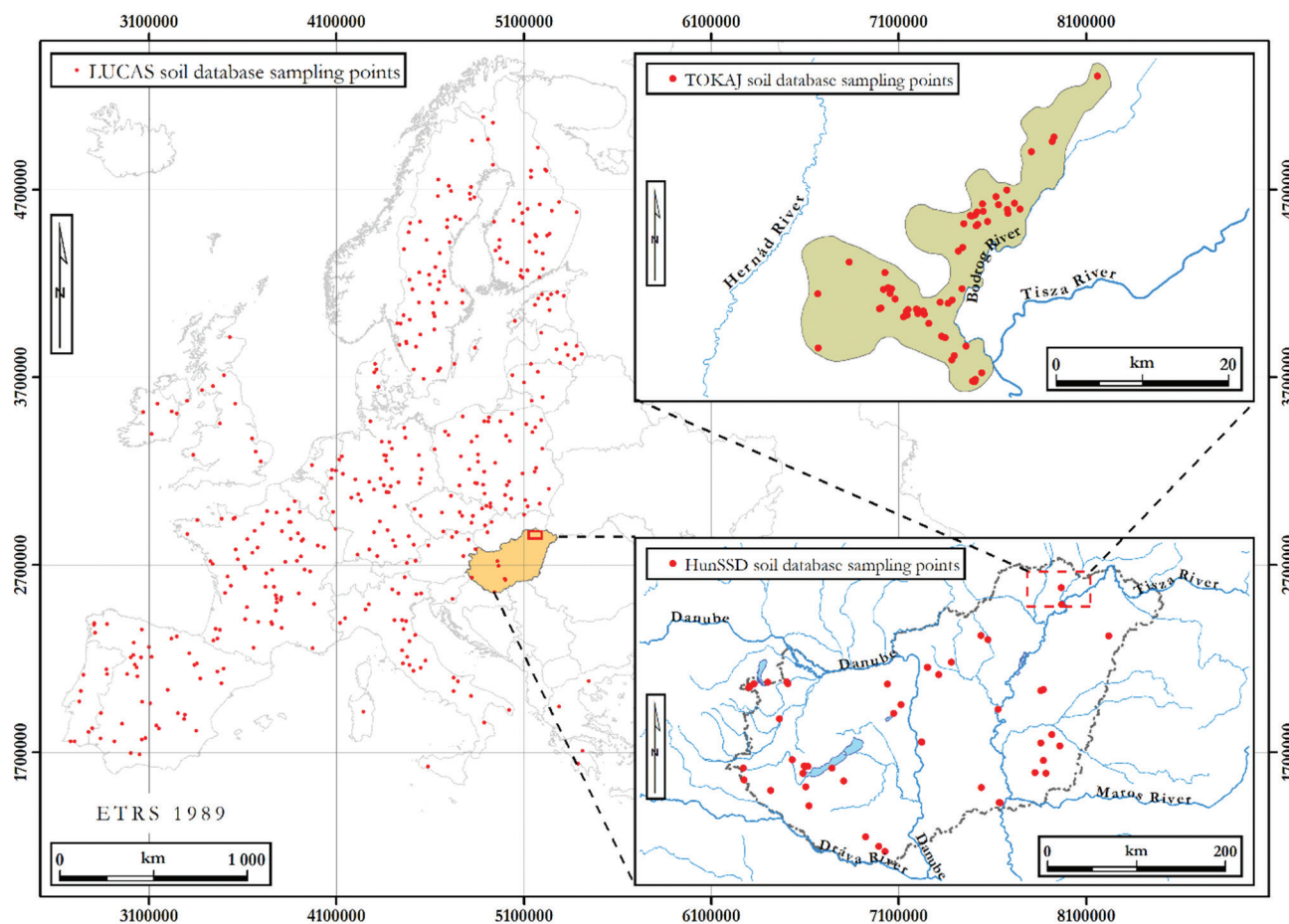

Figure 1 | Soil sampling points for the three databases at different scales

Source: creation by the authors

Materials and methods

Our studies were carried out on three databases. The first is a representative continental soil database. The LUCAS (Land Use/Land Cover Area Frame Survey) soil database is the first consistent spatial database to characterise Europe's topsoils. It was compiled by first collecting nearly 22 000 soil samples using a uniform sampling procedure and then testing the soil samples in a single certified laboratory using a uniform standard methodology (*Tóth–Jones–Montanarella 2013*). A random sampling (R software srswor program; *Tillé–Matei 2015*) of 400 LUCAS topsoil samples were used to select samples that were representative of the variability of the soil cover (countries, climatic zones, plant cover, texture, organic carbon, CaCO$_3$ content, etc.) *(Figure 1)*.

The second one is a representative national soil database, namely HunSSD (Hungarian Soil Structural Database). For the characterisation of the most important soil types in Hungary, representative soil profiles were excavated and described at 55 sites, from which disturbed and undisturbed soil samples of cc. 250 soil horizons were collected *(Figure 1)*. In addition to the basic soil properties, the hydrophysical properties (water retention and water conductivity) of the samples were determined and the soil structure of the samples was investigated using different methods. On the basis of these analyses, we compiled the first soil physics database in Hungary including soil structural properties. The tests presented here were carried out on soil samples from these databases.

The third soil database (TOKAJ) is a regional one. Soil samples were collected in 2014 and 2015 as part of a comprehensive soil survey in the Tokaj wine region of Northern Hungary *(Figure 1)*. This is one of the most famous wine regions in Hungary and Europe, with a spatially diverse and unique terroir, both in terms of soil geology and climate, as a result of natural and man-made landscape evolution. The main parent materials are here rhyolite tuff and loess. The area is characterised by a variety of soil sequences. At high altitudes, where erosion is well marked, fresh, fragmented rhyolite tuff is present at shallow depths below the surface. Here the soils are generally Cambic, Lithic or Mollic Leptosols. On the lower slopes, volcanic 'sands' are found as parent material, the feldspar minerals of which have been transformed into clay minerals by weathering processes in the near-surface soil layers; the surface soil layers therefore have a clayey texture. Calcisols with a thin humic upper layer are found on the surfaces covered with loess. A total of 155 soil samples were collected from 53 representative bored soil profiles in the region *(Makó et al. 2019)* *(Figure 1)*.

The analysed soils of the three databases at different scales show a wide range of variation in several soil properties *(Table 1)*. Soils formed largely on weathered volcanic rocks are the most clayey in the TOKAJ database.

Most of the soils with sand texture are in the LUCAS database. Soils in the widest pH range (pH: 4.2–10.1) are found in the national database, but the most acidic soils are in the European database (pH: 3.8). Organic matter content is also the most variable in the national database (humus%: 0.1–44.1), but the average organic matter content is the highest in the European database (humus %: 2.9). The main reason for this is that the soil samples in the LUCAS database were derived exclusively from the topsoils. The soil sample with the highest lime content is found in the LUCAS database (CaCO$_3$%: 60.7), but the average lime content of the soils is highest in the HunSSD database (CaCO$_3$%: 7.8).

Table 1 | Mean physical and chemical properties of the soil samples used in the PSD measurements

Soil properties	Mean	Standard deviation	Minimum	Maximum
LUCAS				
Clay[a] / m%	22.3	15.0	1.0	76.0
Silt[b] / m%	41.7	18.2	4.0	88.0
Sand[c] / m%	36.0	26.2	1.0	94.0
pH(H$_2$O) / –	6.3	1.2	3.8	8.7
Humus[d] / m%	2.86	2.6	0.3	17.0
CaCO$_3$ / m%	4.7	10.8	0.0	60.7
HunSSD				
Clay[a] / m%	24.5	13.2	0.19	67.8
Silt[b] / m%	42.1	18.9	1.31	72.9
Sand[c] / m%	33.4	28.4	0.5	96.3
pH(H$_2$O) / –	7.6	1.2	4.2	10.1
Humus[d] / gkg^{-1}	1.9	3.6	0.1	44.1
CaCO$_3$ / gkg^{-1}	7.8	10.7	0	53.1
TOKAJ				
Clay[a] / m%	35.3	12.4	7.5	67.9
Silt[b] / m%	45.6	11.4	12.1	65.4
Sand[c] / m%	19.1	12.4	2.7	78.2
pH(H$_2$O) / –	7.2	1.0	5.0	8.9
Humus[d] / gkg^{-1}	1.1	0.6	0.2	3.1
CaCO$_3$[e] / gkg^{-1}	1.8	4.1	0	36.0

[a]Clay, clay content; [b]Silt, silt content; [c]Sand, sand content (all these fractions were determined by the ISO or MSZ sieve–pipette method); [d]Humus, organic matter content; [e]CaCO$_3$, lime content; Laboratory standards are described by *Tóth–Jones–Montanarella (2013)* and *Makó– Tóth (2013)*

Source: creation by the authors

There was a larger diversity of soil types and range in some soil properties in the European LUCAS soil dataset. For other soil properties, however, the Hungarian HunSSD database showed higher diversity, as it included

soil samples from the lower soil layers, not only from topsoils, as the LUCAS database did. The least diverse soils were found in the regional TOKAJ database *(Table 1)*.

Comparative PSD studies were carried out between sieve-pipette sedimentation (SPM) and laser diffraction (LDM) methods. For all PSD measurement methods, soil samples (air-dried, milled, passed through a 2 mm sieve, free from macroscopic plant residues) were prepared in the conventional way. Among the SPM methods, the international standard *(ISO 11277:2020)* was used for the continental database, while the national standard methodology *(MSZ-08.0205-78)* was used for the tests at the national databases. The SPM-ISO method does not clearly describe in which case the removal of lime and iron (oxyhydroxides) in addition to the destruction of organic matters with hydrogen peroxide should be performed, leaving it to the decision of the investigator, which leads to a high degree of uncertainty in the comparability of the measurement results. For this reason, only the removal of organic matter was performed on the whole sample material during sample preparation. The national standard (SPM-MSZ) does not apply organic matter removal during preparation. In the LDM tests, for comparability, organic matter removal with hydrogen peroxide was also applied to soil samples from the continental database. After removal of the organic matter, the organic free soil suspension was evaporated at 40°C, then carefully crushed and sieved again through a 2 mm sieve. This was necessary because, according to the literature and our own experience, sample dosing in suspension form during LDM PSD measurements reduces the repeatability of measurements *(Fisher et al. 2017)*. No such preparation was applied to the soils of the national databases. Calcium carbonate were not removed from any of the soil sample sets.

The SPM-ISO PSD measurements were carried out in the laboratory of SGS Hungary (Kecskemét). Soil suspension was prepared from 30 g of air-dry soil sample. During the measurements, the sand fraction was sieved from the pre-treated and dispersed soil suspension, dried at 105°C, and then determined by sieve analysis and mass determination of the individual sand sub-fractions. The amount of fractions smaller than the sand fractions was determined by sedimentation using the pipette method (taking into account settling times and pipetting depths calculated on the basis of the standard average soil density of 2.65 g·cm^{-3}). Chemical dispersion was carried out using a 'Calgon' solution (33 g Na hexametaphosphate and 7 g anhydrous Na carbonate dissolved in 1 l water), followed by shaking the soil suspension for 18 hours. The results of the SPM-ISO PSD tests were used for further data analysis of the SPM-ISO sand (63–2000 μm), SPM-ISO silt (2–63 μm) and SPM-ISO clay (>2 μm) contents by weight.

In the SPM-MSZ PSD procedure, a soil suspension was prepared from 25 g of air-dry soil sample. No chemical pre-treatment was applied, only chemical and physical dispersion of the samples was performed by adding a standard solution of sodium pyrophosphate (55.8 g·L^{-1}) to the suspension and shaking the suspension for 6–10 hours. The soil suspension was then carefully washed through a 250 μm sieve into a sedimentation cylinder and the coarse sand fraction greater than 250 μm was determined by mass measuring after drying. The particles passed through the sieve were separated into fractions by settling and pipetting. For the calculation of the settling time, it was assumed that the average density of the particles was 2.65 g·cm^{-3}. The percentage (% by weight) of the constituent fractions was determined according to the national standard (SPM-MSZ sand: 2000 μm to 50 μm; SPM-MSZ silt: 50 to 2 μm; SPM-MSZ clay: <2 μm).

The laser diffraction particle size distribution (LDM PSD) studies were also performed in two ways. For the continental and regional scale databases we used a Mastersizer 2000, while for the national scale database we had a Mastersizer 3000 type device.

The Mastersizer 2000 (Malvern Instruments, Malvern, UK) with a measurement range of 0.02–2000 μm (*Malvern Operator's Guide 1999; ISO 13320:2020*) was available at MOTIM Zrt. (Mosonmagyaróvár). A Hydro G dispersion unit was connected to the instrument. The measurements were performed in two or three replicates (from independent measurements), depending on the variance of the results. The weight of the soil samples added to the disperser varied between 0.5 and 1 g, depending on the texture of the soil. The obscuration of the soil suspension was held between 10 and 20%. The air-dry soil sample was moistened with a few drops of Calgon solution on a watch glass and gently mixed with a glass rod. The soil pulp was then washed residue-free into the dispersing unit already containing 25 cm^3 of Calgon solution and about 800 cm^3 of deionised water (*Bieganowski–Ryżak–Witkowska-Walczak 2010*). For complete disaggregation and dispersion of the soil, 240 seconds of 75% power ultrasonic treatment (0.75 × 35 W and 0.75 × 40 kHz, respectively) was used. The Hydro G dispersion unit was set to a pump speed of 1750 rpm, and that of the stirrer at 700 rpm (*Ryżak–Bieganowski 2011; Sochan et al. 2012*). The laser light intensity data measured by the detectors of the instrument were converted into PSD results based on Mie theory, where the following settings were used in the calculations: absorption index (AI) = 0.1; refractive index (RI) of the solid phase = 1.52 and refractive index (RI) of water = 1.33. The duration of the measurements was 60 s (30 s blue and 30 s red laser light) (*Ryżak–Bieganowski 2010*). The algorithms used in the PSD calculations were 'general purpose analysis' and 'irregular shape ratio' (*Malvern Operator's Guide 1999*).

The Mastersizer 3000 (Malvern Instruments, Malvern, UK) with a measurement range of 0.02–3000 μm was available in our institute. It was equipped with a

Hydro LV dispersion unit for the LDM PSD measurements. Based on our preliminary experience, the pumping and stirring speed was adjusted to 2750 rpm, the absorption index (AI) was set to 0.1 and the refractive index (RI) was set to 1.52. The obscuration range of soil suspension varied between 5 and 20%. The size distribution of particles was calculated based on the diffraction image, according to Mie theory (non-spherical particle mode), Mastersizer 2000 emulation was not applied. During ultrasonic dispersion, the ultrasound power was set to 100%. The duration of ultrasonic dispersion was 4 minutes. Similar to the Mastersizer 2000 measurements, the air dry soil sample was first moistened with 3 drops of Calgon solution and then washed into the dispersion unit containing 800 ml deionised water and 25 ml of Calgon solution. All measurements were carried out on three independent samples.

The microaggregate stability (MiAS%) of the soil samples was also determined with the Mastersizer 3000 using laser diffractometry. These tests were carried out on samples from the national scale soil database. The procedure consisted in measuring the clay content of air-dried, crushed soils sieved through a 2 mm sieve in two different ways: with and without dispersion, using the LDM PSD method. The clay content of the dispersed soils was measured as described in the previous paragraph, pretreated in a Hydro LV dispersion unit, using Calgon dispersant and 4 minutes of ultrasonication. For the non-dispersed soil samples, neither a chemical dispersant nor ultrasound was used, only the soil sample was stirred and circulated (pumping and stirring speed: 2750 rpm) in distilled water medium for 4 minutes, and then we did the PSD measurement. The LDM PSD curves were used to determine the two type clay content of soil samples. Microaggregate stability *(MiAS, %)* was calculated according to Vageler's structure factor (Eq. (1)) from the rate of clay fractions determined with dispersion (c_d) and without any dispersion (c_{nd}) *(Vageler 1932)*:

$$MiAS\ (\%) = \frac{(c_d - c_{nd})}{c_d} \times 100 \qquad (1)$$

The results were evaluated as follows. For the PSD studies, since we wanted to compare the LDM-PSD results with the SPM-PSD results, at first LDM-PSD fractions similar to the SPM-PSD clay, silt and sand fractions were calculated from the particle distribution data (fraction boundaries 2 and 63 µm for the ISO standard and 2 and 50 µm for the MSZ standard) *(Makó et al. 2019)*.

In the next phase of data processing, we searched for the optimal clay/silt and silt/sand size boundaries at which the SPM and LDM clay, silt and sand fractions differ the least. Fourteen possible clay fractions were calculated from the LDM PSD results at upper boundary ranging from 1.4 to 10.0 µm (1.4, 1.6, 1.9, 2.2, 2.9, 3.3, 3.8, 4.4, 5.0, 5.8, 6.6, 7.6, 8.7 and 10.0 µm, respectively). In order to optimize the thresholds, close to

the optimal clay/silt boundary (between 6.6 and 7.6 µm) different fraction boundaries were examined using linear interpolation with a resolution of 0.1 µm. The calculated clay, silt and sand fractions with various boundaries were used as dependent variables against the conventional clay, silt and sand fraction measured with SPM-MSZ (<2.0 µm, 2–50 µm and 50–2000 µm, correspondingly). Lin's concordance correlation coefficient (CCC) computation *(Lin 1989)* between the PSD results of SPM and LDM data for each dataset were performed to optimize the clay, silt and sand fraction boundaries of the LDM measurements. Lin's CCC measurements indicate how well a new set of observations (LDM PSD measurements) reproduce an original set (SPM-MSZ PSD), so that it may be used to assess the efficiency of the laser measurement methods *(Fisher et al. 2017)*. The resulting 'modified optimum LDM fraction boundaries' were used to calculate the modified clay, silt and sand fractions. (This study was carried out on the regional national database and the results were extended to all three databases for comparability. In the future, we intend to perform the size limit optimization on all three databases separately.)

The differences between the SPM-ISO or SPM-MSZ and differently calculated LDM particle fractions were visualized in a USDA texture triangle with the Origin-Pro 2021 software (OriginLab Corporation, Northampton, MA, United States). The effectiveness of the size limit change in matching PSD data measured by different methods was investigated based on the evaluation of the coefficients of determination (R^2) and root mean square errors *(RMSE)* (mass %):

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^{N} \left(y_i - \hat{y}_i \right)^2}, \qquad (2)$$

where: y_i stand for the SPM clay, silt or sand values, $\hat{y}_i$ are the differently calculated LDM clay, silt or sand values, N is the total number of samples.

To evaluate the results of microaggregate stability measurements, we examined the relationship between the soil properties and MiAS values. Pearson's correlation coefficients (r) were used to determine the relationships between soil properties. OriginPro 2021 software was used to investigate the correlations, identify significant effects and present them in a correlation matrix.

For the further statistical analyses, we used the 'standard model' version of Regression/Automatic Linear Modelling method of the IBM SPSS 22.0.0.0 software package (IBM Corp. Released 2013. IBM SPSS Statistics for Windows, Version 22.0. Armonk, NY: IBM Corp.). The advantage of this method over the traditional linear regression method (Regression/Linear) is the automatic selection of the most efficient estimating variables and the automatic data preparation. In selecting efficient estimator variables, the method searches
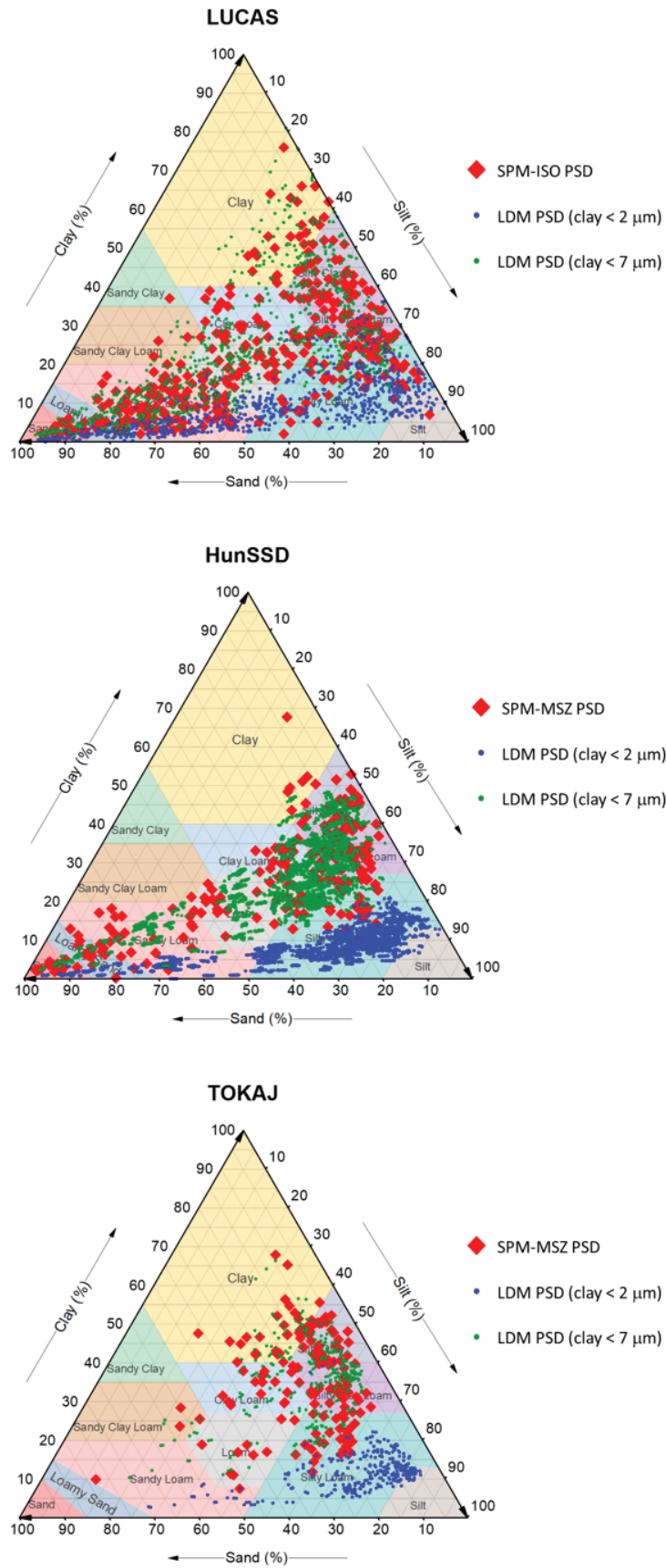
Figure 2 Comparison of LDM and SPM PSD results for three different scale databases on USDA texture triangle plots (two different clay/silt size limits were used for LDM PSD measurements)

Source: creation by the authors

Table 2 | The effect of fraction size limit changes on the accuracy of conversion from LDM to SPM. Summary results for R^2 and RMSE values

Databases		Conventional fraction size boundaries			Modified fraction size boundaries		
		clay (mass %)	silt (mass %)	sand (mass %)	clay (mass %)	silt (mass %)	sand (mass %)
LUCAS	**RMSE**	16.30	15.68	7.26	8.99	8.87	7.26
	R^2	0.71	0.75	0.94	0.74	0.83	0.94
HunSSD	**RMSE**	19.29	19.82	9.25	6.77	7.46	9.25
	R^2	0.75	0.82	0.92	0.74	0.87	0.92
TOKAJ	**RMSE**	24.97	26.95	5.25	6.54	5.74	5.25
	R^2	0.69	0.49	0.84	0.72	0.75	0.84

Source: creation by the authors

only for independent variables that are well related to the dependent variable. We have chosen the 'Forward stepwise' selection method for this purpose. The used optimization statistics method was the corrected Akaike information criterion [AICC]. The optional automatic data preparation was applied, where the 'Automatic Linear Modelling' method performed the missing data substitution (by inserting the mean, mode or median of the variables, depending on the type of variables) and selecting and replacing outliers *(Yang 2013; Bhat 2016; Hall–Caton 2017)*. When selecting outliers for each variable, the procedure sets the thresholds at three times the standard deviation from the mean. The procedure then examines the role of the outliers in the estimation by calculating the Cook's distance. After identifying outliers and outliers that significantly affect the estimation, the decision to exclude these records completely can be made during data processing.

Results

The PSD results of the three databases determined by two different methods (SPM and LDM) are compared and shown in *Figure 2* and *Table 2*. The textural triangles *(Figure 2)* illustrate the distribution of textures in the three databases, compared the texture classes formed in different ways, based on calculated clay, silt and sand fractions. *Table 2* compares the results of correlation tests between clay, silt and sand contents for SPM and LDM measurements and the RMSE values indicating the degree of under- and overestimation of the two types of measurements.

The correlations between the LDM and SPM values of clay fraction in the European (LUCAS), Hungarian (HunSSD) and regional (TOKAJ) sets were very similar, respectively: R^2 = 0.71, 0.75 and 0.69, while maintaining the traditional 2 µm clay/silt LDM size limit. At the same fraction size threshold, there were already larger differences in the correlation of silt and sand contents between the databases (R^2 = 0.75, 0.82 and 0.49 for silt fractions and R^2 = 0.94, 0.92 and 0.84 for sand fractions). Another indicator of the comparability of the

PSD measured by the two measurement methods, the root mean square error (RMSE) value for the three databases was rather large for the clay and silt fractions (16.30, 19.29 and 24.97 for clay fractions; 15.68, 19.82 and 26.95 for silt fractions) and much lower for the sand fractions (7.26, 9.25 and 5.25, respectively) *(Table 2)*. The high RMSE values suggest that in this case, the LDM method significantly underestimated the clay content, while overestimated silt content, even with relatively good correlation.

It can be clearly seen in texture triangles in *Figure 2,* that by keeping the clay/silt size limit at 2 µm, the point sets of the LDM measurements for all three databases shift towards texture classes characterised by lower clay content and higher silt content, compared to the point sets of SPM PSD measurements. The large number of measurement points on the HunSSD data phase triangle diagram is due to the fact that all replicates of the sample measurements are plotted on this diagram.

In order to identify the optimal LDM clay/silt threshold, the statistical method suggested by *Lin (1989)* seemed to be useful. The results of Lin's concordance correlation coefficient (CCC) analyses showed that for the clay fraction, the CCC value was the highest for the 0–7.0 µm fraction and the upper boundary calculated for clay (7.0 µm) was acceptable as the lower boundary of the silt fraction, as well. The silt/sand LDM size boundaries have not been modified or optimized in this work, but kept the 63 µm (ISO) and 50 mm (MSZ) size limits of the SPM methods.

Clay, silt and sand fractions calculated with the optimal clay/silt size limit (7.0 µm) modified by the above method were also compared with the SPM PSD fractions *(Table 2)*. The correlations between the LDM and SPM values of clay fraction in the European (LUCAS), Hungarian (HunSSD) and regional (TOKAJ) sets has not changed significantly with the change in size limit (R^2 = 0.74, 0.74 and 0.72, respectively). In contrast, RMSE values have fallen sharply (RMSE = 8.99, 6.77 and 6.54), suggesting that underestimation of clay have been substantially reduced. In the case of silt content, R^2 values have also improved (R^2 = 0.83, 0.87 and 0.75)
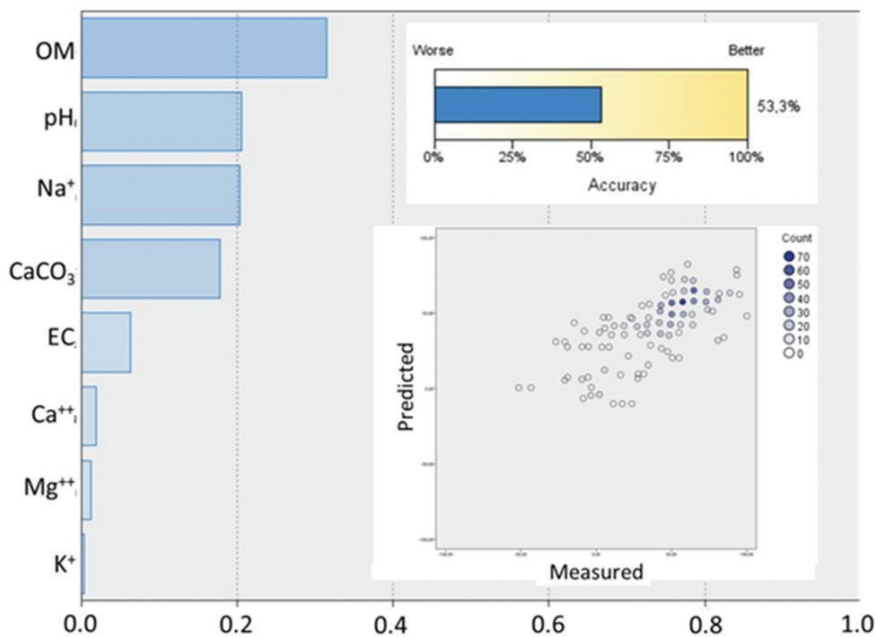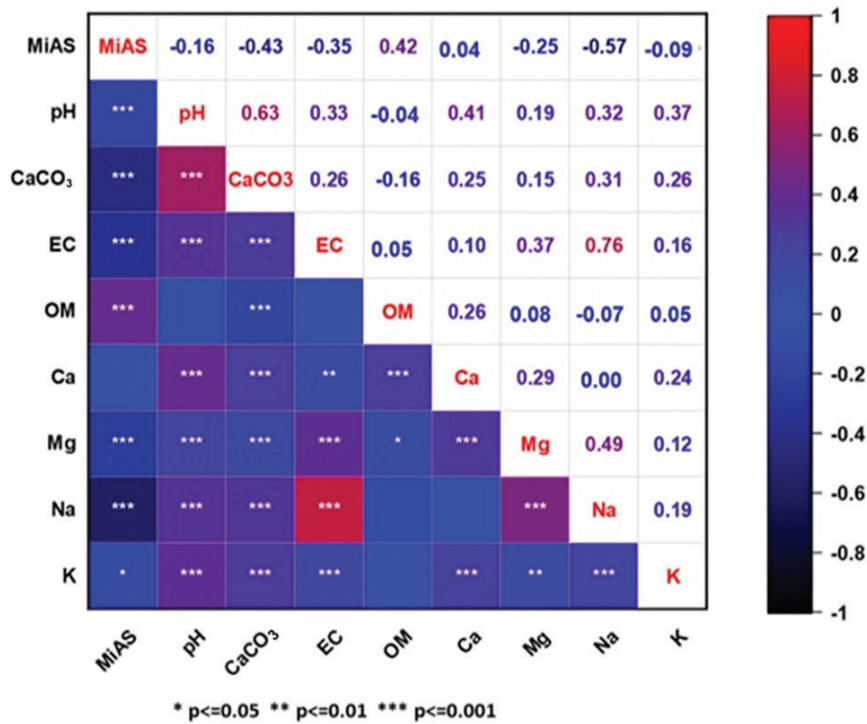
Figure 3 Evaluation of microaggregate stability measurements using a correlation matrix and linear regression (Automatic Linear Modeling) on HunSSD database soils

Source: creation by the authors

and RMSE values have decreased significantly (RMSE = 8.87, 7.46 and 5.74), to varying degrees across databases. This suggests a reduction in silt fraction overestimation for all three databases. As the silt/sand size limits were not changed, the correlation and comparability accuracy of the sand contents were not changed *(Table 2)*.

The above is well illustrated by the texture triangles in *Figure 2*. By choosing a clay/silt size limit of 7 μm, the point sets of the SPM and LDM measurements over-

lapped well. Although in some cases the differences between the texture classes of the measured samples still remained, the distances between the SPM and LDM measurement points on the triangle plots were on average significantly reduced. In general, the 'tail' of the SPM measurement points is much wider than that of the LDM measurement points. This shows that the results of the LDM measurements reflect the variability of the soil samples less than the results of the SPM measurements.

A comparison of databases at different scales (continental, national and regional) shows that the change in the clay/silt size boundary resulted the largest improvement in SPM LDM comparability for the regional (TOKAJ) dataset. This was followed in order by the national (HunSSD) data set, then the continental one (LUCAS).

A statistical evaluation of the results of the microaggregate stability tests is presented in *Figure 3*. Based on the correlation matrix showing the dependence of the microaggregate stability indices (MiAS %) determined by laser diffraction measurement techniques on soil properties, it can be said that there is a strong significant positive relationship between MiAS% and soil organic matter (OM) content; and strong negative relationship between microaggregate stability and pH, lime content, salt content and exchangeable sodium content.

At the same time, of course, individual soil properties are closely related to each other, since soil chemistry can depend on, for example, the lime content, the quality and quantity of salts and the amount of exchangeable sodium ions. Therefore, using the regression method of Automatic Linear Modelling, we tried to select the soil properties that actually influence microaggregate stability and to filter out redundant effects. *Figure 3* illustrates the soil properties that actually influence the microaggregate stability and the extent of their influence. Among the soil properties studied, the humus content (organic matter content) of soils is the most important for microaggregate stability. This is followed in order by the pH of the soils, and then - among the exchangeable basic cations – the sodium content. The $CaCO_3$ content is also important, but the electrical conductivity (EC) value, expressing the total water-soluble salt content of soils, is less important. It can be seen from *Figure 3* that the regression model with the soil properties included in the study can only describe the microaggregate stability of the soils in the best case 53%. Other factors (other soil properties, environmental and anthropogenic factors) influencing the microaggregate stability of the soils in the HunSSD database should be looked for in addition to these properties.

Evaluation, discussion, and conclusions of the results

Compared with the SPM, the precision in detection of the clay and silt fraction with the LDM was generally poor for all databases, when using the conventional clay/silt fraction size boundary (2 μm) *(Table 2, Figure 2)*. Similarly poor correlations between SPM and LDM PSD measurements due to clay underestimation and silt overestimation have been observed previously by e.g. *Eshel et al. (2004)* or *Yang et al. (2019)*.

In the present research when the clay/silt fraction size boundary was set at <7 μm in the soils for any selected database there was very good precision and accuracy *(Table 2, Figure 2)*. It is consistent with the findings of other authors who found that LDM gave an underestimate of clay with a clay/silt size limit <2 μm (e.g. *Konert–Vandenberghe 1997; Taubner–Roth–Tippkötter 2009; Di Stefano–Ferro–Mirabile 2010; Fisher et al. 2017; Faé et al. 2019*). However, based on our Lin CCC studies, we found the proposed revised size limit of 8 μm (e.g. *Konert–Vandenberghe 1997*) to be too high; for our TOKAJ database, the appropriate size limit was 7 μm. For the LUCAS database, the clay/silt boundaries were found to be optimal at even lower values (6.6 μm and 5.8 μm) in our previous work *(Makó et al. 2017)*. The application of the general modified size limit of 7 μm resulted in different degrees of RMSE improvements for the three databases: the most spectacular improvement for the regional database (on which we verified this optimal value) and the least spectacular improvement for the continental database. One explanation for this can be found in a statement by *Thomas et al. (2021),* who said that if a data set is dominated by sand textured soils, the limit is close to 2 μm and if, on the contrary, clay textured soils predominate, the optimal size limit is around 8 μm. In our case, the continental database contained the sandiest soils and the regional database the most clayey soils. Another explanation for the different degree of improvement may be that the continental database was subjected to organic matter destruction prior to both SPM and LDM PSD measurements. The literature shows that H_2O_2 destruction of organic matter can alter also the mineral composition of soils, reduce the amount of clay minerals and thus modify the clay content *(Bieganowski et al. 2018a)*.

The measured sand contents of the LDM PSD measurements are much closer to the SPM PSD sand contents than the clay and silt contents. This has generally been stated by all authors working on the subject (e.g. *Beuselinck et al. 1998; Taubner–Roth–Tippkötter 2009; Ryżak–Bieganowski 2010*). This also suggests that the assumption in sedimentation and laser diffraction measurements that the particles are spherical is mostly valid only for sand particles.

The differences in accuracy may also be related to the diversity of the sample populations, since samples with high organic matter content, high salt or sodium content, high lime content or even samples with high amounts of different iron compounds may require different settings or even different dispersion techniques during LDM measurements *(Eshel et al. 2004; Taubner–Roth–Tippkötter 2009; Makó et al. 2017; Bieganowski et al. 2018a)*.

In future research, we intend to investigate the optimal clay/silt size limits for each of the three databases and compare these size limit optima with the properties of the soils in the databases in detail. We would also like to further refine the comparability of the two methods by using other estimation procedures beyond the size limit change that take into account the variability of soil

properties. We are considering both the development of pedotransfer functions that can calculate more accurate LDM PSD fractions given known basic soil properties, and the development of instrument setup protocols that take into account the known basic properties of the soils that come into the laboratory.

The investigation of the relationship between the microaggregate stability of soils and soil properties on a large database with diverse soil properties can be considered as a preliminary study. The microaggregates are typically considered more stable then macroaggregates because of their stronger organic or inorganic cementing agents and less macropores, more micro-, nano- or cryptopores *(Tisdall–Oades 1982; Oades–Waters 1991; Amézketa 1999; Six et al. 2004)*. Our calculated microaggregate stability indices (MiAS %) based on the measured clay dispersion, with values approaching 100% per case, also indicate strong binding forces within the microaggregates. However, we sometimes measured MiAS % values close to zero and even negative values. These cases need to be examined individually, because the values can be explained partly by the low microaggregate stability of the poorly developed soil samples, mainly from subsoils, partly by the rapid dispersion of the high sodium saturated samples in aqueous media, and partly by possible errors in our LDM measurement technique.

In the current phase of our correlation analysis, we did not perform any filtering on the database before the calculations, so we did not remove outliers. Consistent with the literature, the presence of organic matter was found to have a high stabilising *(Tisdall– Oades 1982; Totsche et al. 2018; Felde et al. 2021)*, while exchangeable sodium a destabilising *(Rengasamy–Marchuk 2011; Bennett–Marchuk–Marchuk 2016; Almajmaie et al. 2017)* effect on the microaggregates. The literature experience was supported by the not very clear negative effect of water soluble salt content, the effect of salt depends on a lot of other soil chemical properties *(Sumner–Naidu 1998; Rengasamy–Marchuk 2011; Voelkner–Holthousen–Horn 2015)*. However, the significant destabilising effect of the lime content was certainly interesting and contrary to the literature *(Rowley–Grand–Verrecchia 2018; Totsche et al. 2018; Getahun et al. 2021)*. The experience that the stability of microaggregates decreases with increasing pH can probably be explained by the close correlation between pH, lime content and exchangeable sodium content, and the fact that our database contained a significant amount of saline and highly calcareous soil samples. Later on, we will continue our correlation studies, refining the data preparation, examining the different soil groups separately, evaluating the effect of outliers.

In our linear regression model, we have already carried out data preparation, albeit in an automated way for the time being. Our studies confirmed the experience that the strongest stabilising components of microaggregates are various humic substances *(Tisdall– Oades 1982;*

Totsche et al. 2018). In the model investigating the combined effect of soil properties, soil pH showed a positive relationship with MiAS values (probably due to the exclusion of outliers), but the effect of lime content remained negative. The negative effect of pH reduction on structural stability is in line with the literature *(Tavares Filho–Barbosa–Ribon 2010; Voelkner–Holthousen–Horn 2015; Almajmaie et al. 2017)*, but the explanation for the significant negative effect of lime content is still being sought. One explanation could be that the stabilising effect of the lime in the soil is strongly dependent on the size distribution of $CaCO_3$ particles *(Inskeep–Bloom 1986; Le Bissonnais 1996b)*, since the size of the $CaCO_3$ particles must be below the size range of the silt fraction to achieve proper aggregation. Among the exchangeable cations, only the negative effect of sodium ion was beyond discussion, the other cations had no significant effect. It was surprising that the statistical analysis did not confirm a stabilising effect for the two valent calcium ions, contrary to the literature *(Calero–Barrón–Torrent 2008; Totsche et al. 2018)*. This may be because exchangeable Ca ions are closely correlated with both soil pH, lime content and organic matter content, so the effect of Ca ions has already been indirectly described by these factors *(Figure 3)*. Several authors have confirmed a positive relationship between soil organic matter content and exchangeable Ca ions (e.g. *Gaiffe et al. 1984; Oades 1988; Bertrand–Delfosse–Mary 2007; Li et al. 2017)*.

The model used described only 53.3% of the microaggregate stability of the soils, which means that many of the influencing factors are not yet known. In the future, we will continue our investigations with regard to the history of cultivation and land use of the soils and the morphological properties of the soil layers studied.

The soil physical properties investigated in this study are very closely related to the water management properties of soils. The hydrophysical parameters (water retention, hydraulic conductivity) characterising the water management properties of soils are very often obtained using PSD data. In order to refine the estimation methods (pedotransfer functions), the structural properties of the soil are increasingly taken into account. If we can quickly and reliably measure the PSD and aggregate stability of soils using LDM, then the input data needed to estimate hydrophysical properties can be produced quickly and in larger numbers, allowing us to characterise soil water management more reliably and in greater detail, based on more samples.

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