# „Use of structural soil properties for improving prediction methods in soil systems wetting by water or non-polar liquids"

## *Final report*

## *(2016-2022)*

## I. Research history

**2016-2017.** The research started on 1 October 2016. Based on the information of the Detailed Soil Physical and Hydrological Database of Hungary (MARTHA) and the Agrochemical Information and Management System (AIIR) database, the typical soil profiles (*N=73*) representing the soil types of the country were selected (the soil profiles representing each soil type were selected according to their incidence rate). Undisturbed soil samples with original structure and disturbed soil samples were also collected from each site (48-60 undisturbed soil cores and 4-5 disturbed soil samples per profiles). Due to dry weather conditions, the fieldwork and sampling were delayed for a third year. In each profile excavation, field descriptions of the soil profiles were made, morphological characteristics were recorded and photographic documentations were made. Soil samples (*N=326*) were prepared for laboratory analysis and the analysis were started. Determination of the typical soil physical, soil chemical and mineralogical properties of the collected soil samples (basic soil tests, BET surface area measurements, soil mineralogical and clay mineralogical tests) was started. Particle size distribution tests were also started on the collected samples using the sieve-pipette methods (SPM) according to national (MSZ 08-0205:1978) standard. (Due to lack of laboratory capacity, we did not measure according to the international standard (ISO 11277/2009), but based on our previous measurements we developed an estimation method to determine this.) In the same way, particle size distribution with laser diffraction method have been started. Our original plan has been modified, because in the meantime our institute has acquired a Malvern Mastersizer 3000 laser particle analyser device, so these measurements were carried out in our institute. The specific surface area measurements have been started in the laboratory of MOTIM Zrt. An Eijkelkamp (wet sieve) macro-aggregate stability measuring instrument was purchased for the project, with which methodological preliminary experiments were carried out (concerning the required pre-wetting method and time), and then the aggregate stability measurements of the collected samples were started. Methodological pre-tests were also carried out with the laser diffraction aggregate stability measurements. The pipette microaggregate stability measurements (Vageler-type structure factor determination) have been also begun. Measurement of water retention of soils using conventional sand and kaolin box method and the pressure membrane method, respectively was started. The measurement of hydraulic conductivity of two-phase soils was started with the conventional falling head method. The original number of people who joined the project has been increased (Hilda Hernádi, Dorottya Kovács, Tamás Varga).

**2017-2018.** Physical, chemical and mineralogical measurements of key soil attributes, including basic soil analyses, BET measurements, particle size analysis by pipette method determination of soil mineral composition and clay mineral characteristics were carried out using a part of the samples collected. The methodology of particle size analysis by laser diffraction method using Mastersizer 3000 (Malvern) developed/refined. Comparative measurements were made with the previous Mastersizer 2000 device (e.g. comparison of settings parameter). Particle size analysis by laser diffraction method (LDM) were carried out using a part of the samples collected. Aggregate stability measurements, including wet sieving method, SPM and LDM were carried out using a part of the samples collected. Water retention measurement and saturated hydraulic conductivity measurements using falling head method were carried out using a part of the undisturbed samples collected. $K_{nearsat}$ (near saturated hydraulic conductivity: conductivity of soil matrix) were also measured by Mini-disc infiltrometer in laboratory using a part of the undisturbed samples collected. Non-polar liquid retention measurements using modified pressure cell apparatus (Soilmoisture Equip. Corp.) were carried out using a part of the undisturbed samples collected. Preliminary experiments were performed to measure non-polar liquid conductivity of soils. The original number of people who joined the project has been temporarily increased (Viktória Dellaszéga-Lábas, Emese Ujj, Borbála Kovács). A new PhD student joined the project (Viktória Labancz). Tamás Varga has left the project.

**2018-2019.** The measurements of some parameters (e.g. clay mineralogy, NAPL conductivity) did not go according to plan (for various unforeseen reasons). At the same time, we added new soil properties to the measurement repertoire (humus quality, carbon: nitrogen ratio). We continued the description and sampling of the characteristic Hungarian soil profiles. Physical, chemical measurements of key soil attributes, including basic soil analyses, BET measurements, determination of soil mineral composition and clay mineral characteristics were carried out using an additional part of the samples collected. Particle size analysis by pipette method were continued. Methodological measurements were performed using Mastersizer 3000 and Mastersizer 2000 (Malvern) laser diffractometer devices. Various types of liquids were also compared as dispersants during the studies. Particle size analysis by laser diffraction (LDM) were continued. Aggregate stability measurements, including wet sieving method, SPM and LDM were also continued. We successfully applied the methods developed in the project for measuring the aggregate stability of soil samples treated with various additives (lignite, compost, and biochar). Water retention measurement using undisturbed soil cores were completed. Saturated hydraulic conductivity measurements using falling head method were continued. $K_{nearsat}$ (near saturated hydraulic conductivity: conductivity of soil matrix) were also measured by Mini-disc infiltrometer in laboratory using an additional part of the undisturbed samples collected. Particle shape investigation by Morphologi device (Malvern) were started in Lublin (Institute of Agrophysics, Poland). Non-polar liquid (Dunasol 180/220) retention measurements using modified pressure cell apparatus (Soilmoisture Equip. Corp.) were started using undisturbed samples collected. Water vapor absorption measurements according to Sík ($hy_1$) were begun. Measurements of the Hargitai indices and the C: N ratio of soils were started to characterize the humus quality of the soils. Measurement of the CEC value and exchangeable cation content of soil samples was finished. This measurement was carried out in an external laboratory (Soil Conservation Laboratory, Velence) because the originally planned partner (SZIE) cancelled the measurements. The measurements of non-polar liquid conductivity of soils could not be taken for methodological reasons, we stopped this kind of experiment. The original number of people who joined the project has been increased (Mihály Kocsis, Eszter Némethné Herceg). Viktória Dellaszéga-Lábas has left the project.

**2019-2020.** We started to work on the photo-illustrated brochure of the excavated and described soil profiles. Measurements of most basic physical and chemical soil properties have been completed and organized into a database. All BET specific surface area measurements have been completed. Particle size analysis by sieve-pipette method (SPM) and laser diffraction method (LDM) was performed on all soil samples collected. The results were organized into a database. The methodological comparative study with Mastersizer 3000 and Mastersizer 2000 (Malvern) laser diffractometer devices were continued under other measurement conditions. Aggregate stability measurements, including wet sieving method, SPM and LDM were carried out using an additional part of the samples collected Saturated hydraulic conductivity measurements using falling head method have been completed. $K_{nearsat}$ (near saturated hydraulic conductivity: conductivity of soil matrix) have also been completed. Particle shape investigation by Morphologi device (Malvern) were continued in Lublin (Institute of Agrophysics, Poland). Non-polar liquid retention measurements using modified pressure cell apparatus

(Soilmoisture Equip. Corp.) were carried out using an additional part of the undisturbed samples collected. Unfortunately, these measurements were much slower than expected. The Hargitai indices and the C: N ratio of soils were measured to characterize the humus quality of the soils. These tests have been completed. Soil mineralogical studies could only be performed on a reduced number of samples due to epidemic and other reasons. Rita Földényi and Sándor Molnár have left the project. We requested and received a one-year extension for the project due to delays in sampling and laboratory work.

**2020-2022.** We have finished work on the photo-illustrated brochure of the excavated and described soil profiles. Measurements of all basic physical and chemical soil properties, BET Specific Surface Area measurements, particle size analysis by different methods, aggregate stability measurements, including wet sieving method, SPM and LDM, water retention and saturated hydraulic conductivity measurements using falling head method, $K_{nearsat}$ measurements, non-polar liquid retention measurements, measuring the Hargitai indices and the C:N ratio have been completed. The results were organized into a database. FTIR analyses were also carried out to investigate the humus quality of the soil samples. We also measured the differences between the particle size distribution and humus quality (C:N ratio, FTIR spectra) of the stable and unstable aggregates separated during the macroaggregate stability measurements. The XRF analysis of micro- and meso-element content (mainly iron content) was started on the collected samples. We also participated in an iron concretion study with the soil samples we collected. In 2021 we have asked for a further one-year extension of the project duration (COVID). In 2022, we requested a six-month postponement of the submission of the final report due to publications in preparation.

## II. Results

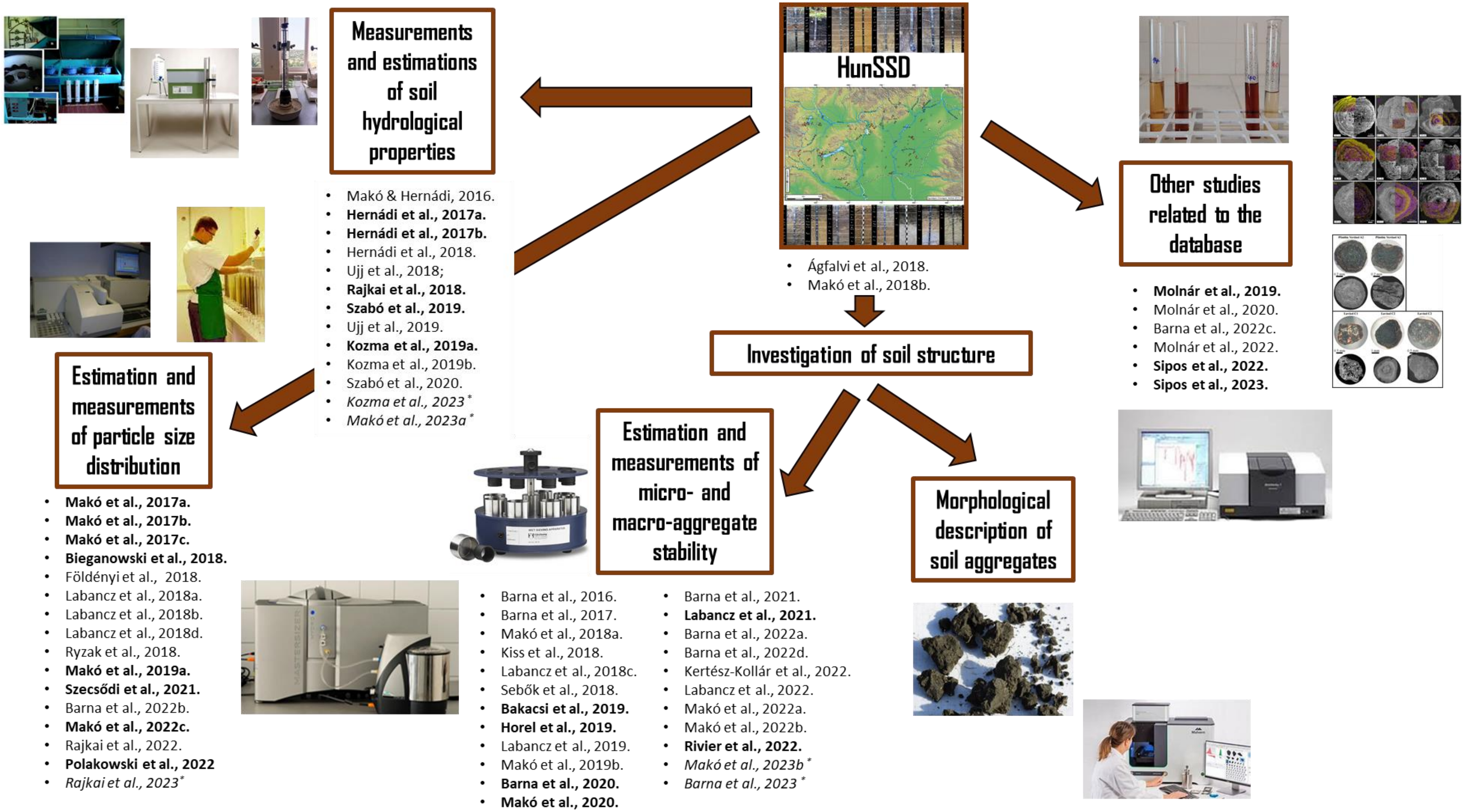
The research directions of the project, which have evolved in the meantime and have been slightly modified from the originally planned ones, and the related published research results are presented in *Figure 1.*

### II/A. Hungarian Soil Structural Database (HunSSD)

For the characterisation of the most important soil types in Hungary, representative soil profiles were excavated and described at 73 sites, from which disturbed and undisturbed soil samples of 326 soil horizons were collected (*Figure 2*).

Soils in the database can be classified into the following main soil types (based on the current national soil classification): Skeletal soils (11), Lithomorphous soils (5), Brown forest soils of Central and South-eastern Europe (23), Chernozem soils (16), Salt-affected soils (5), Meadow soils (9), Bog soils (2), Alluvial and sedimentary soils of rivers and lakes and slope deposits (1). Within the main soil types we can distinguish 24 soil types and 33 soil subtypes (Jassó et al., 1989) (*Table 1*). According to the international soil classification the following reference groups can be distinguished: Arenosol (6), Cambisol (5), Chernozem (8), Fluvisol (2), Gleysol (6), Histosol (2), Kastanozem (3), Leptosol (1), Luvisol (9), Phaeozem (17), Regosol (3), Solonetz (4), Vertisol (2), not classified so far (5) (IUSS WORKING GROUP WRB, 2015).

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 were investigated using different methods. On the basis of these analyses, we compiled the first nationally representative database of soil physics and hydrology including soil structural and morphological properties (*Table 2*).



HunSSD

- Ágfalvi et al., 2018.
- Makó et al., 2018b.

Measurements and estimations of soil hydrological properties

- Makó & Hernádi, 2016.
- **Hernádi et al., 2017a.**
- **Hernádi et al., 2017b.**
- Hernádi et al., 2018.
- Ujj et al., 2018;
- **Rajkai et al., 2018.**
- **Szabó et al., 2019.**
- Ujj et al., 2019.
- **Kozma et al., 2019a.**
- Kozma et al., 2019b.
- Szabó et al., 2020.
- *Kozma et al., 2023* *
- *Makó et al., 2023a* *

Estimation and measurements of particle size distribution

- **Makó et al., 2017a.**
- **Makó et al., 2017b.**
- **Makó et al., 2017c.**
- **Bieganowski et al., 2018.**
- Földényi et al., 2018.
- Labancz et al., 2018a.
- Labancz et al., 2018b.
- Labancz et al., 2018d.
- Ryzak et al., 2018.
- **Makó et al., 2019a.**
- **Szecsődi et al., 2021.**
- Barna et al., 2022b.
- **Makó et al., 2022c.**
- Rajkai et al., 2022.
- **Polakowski et al., 2022**
- *Rajkai et al., 2023* *

Investigation of soil structure

Estimation and measurements of micro- and macro-aggregate stability

- Barna et al., 2016.
- Barna et al., 2017.
- Makó et al., 2018a.
- Kiss et al., 2018.
- Labancz et al., 2018c.
- Sebők et al., 2018.
- **Bakacsi et al., 2019.**
- **Horel et al., 2019.**
- Labancz et al., 2019.
- Makó et al., 2019b.
- **Barna et al., 2020.**
- **Makó et al., 2020.**
- Barna et al., 2021.
- **Labancz et al., 2021.**
- Barna et al., 2022a.
- Barna et al., 2022d.
- Kertész-Kollár et al., 2022.
- Labancz et al., 2022.
- Makó et al., 2022a.
- Makó et al., 2022b.
- **Rivier et al., 2022.**
- *Makó et al., 2023b* *
- *Barna et al., 2023* *

Morphological description of soil aggregates

Other studies related to the database

- **Molnár et al., 2019.**
- Molnár et al., 2020.
- Barna et al., 2022c.
- Molnár et al., 2022.
- **Sipos et al., 2022.**
- **Sipos et al., 2023.**

*Figure 1.*
The research directions of the project and the related published research results
(Publications in bold are articles published in peer-reviewed journals, articles in italics with an asterisk are publications submitted but not yet accepted or in preparation)

*Table 1.*
Distribution of soils in the HunSSD database by soil type and subtype (based on the current national soil classification)

| **Type** | **Subtype** | N |
|---|---|---|
| Earthy barren | Calcareous earthy barren | 2 |
| Humus sand | Calcareous humus sand | 3 |
| Humus sand | Calcareous multilayer humus sand | 4 |
| Humus sand | Humus sand with alternating clay lamellae | 2 |
| Humus-carbonate soil | Humus-carbonate soil | 2 |
| Rendzina | Black rendzina | 2 |
| Erubase soil | Erubase soil | 1 |
| Strongly acidic non-podzolic brown forest soil | Strongly acidic non-podzolic brown forest soil | 1 |
| Brown forest soil with clay illuviation | Non-podzolic brown forest soil with clay illuviation | 8 |
| Pseudogley | Pseudogley with clay illuviation | 6 |
| Brown forest soil (according to Ramann) | Typical brown forest soil (according to Ramann) | 4 |
| Brown forest soil with alternating thin layers of clay substance | Clay illuviation brown forest soil with alternating thin layers of clay substance | 1 |
| Chernozem-brown forest soil | Calcareous chernozem-brown forest soil | 2 |
| Chernozem-brown forest soil | Non-calcareous chernozem-brown forest soil | 1 |
| Chernozem soils with forest | Non-calcareous chernozem soils with forest | 1 |
| Leached chernozem | Leached chernozem | 2 |
| Calcareous chernozem | Typical calcareous chernozem | 6 |
| Calcareous chernozem | Lowland calcareous chernozem | 1 |
| Meadow chernozem soil | Calcareous meadow chernozem soil | 2 |
| Meadow chernozem soil | Non-calcareous meadow chernozem soil | 1 |
| Meadow chernozem soil | Meadow chernozem soil salty in deeper horizons | 2 |
| Meadow chernozem soil | Meadow chernozem soil solonetz-like in deeper horizons | 1 |
| Solonchak | Calcareous solonchak | 1 |
| Meadow solonetz | Shallow meadow solonetz | 3 |
| Meadow solonetz | Middle meadow solonetz | 1 |
| Solonchak-like meadow | Calcareous solonchak-like meadow | 1 |
| Solonetz-like meadow soil | Solonetz-like meadow soil | 1 |
| Meadow soils | Non-calcareous meadow soil | 5 |
| Meadow soil | Meadow soil salty in deeper horizons | 1 |
| Chernozem meadow soils | Calcareous chernozem meadow soils | 1 |
| Lowmoor fen soils | Lowmoor fen soils | 1 |
| Drained and cultivated lowmoor fen soils | Drained fen soil with highly decomposed peaty substance (kotu) | 1 |
| Humus alluvial soils | Calcareous multilayered humus alluvail soil | 1 |

*Table 2.*
Soil data in the HunSSD

| **Soil parameters** | **N** | **min** | **max** | **average** | **standard deviation** |
|---|---|---|---|---|---|
| **Plasticity according Arany ($K_A$ )** | 301 | 21.50 | 84.00 | 42.20 | 12.05 |
| **pH ($H_2O$)** | 326 | 4.15 | 10.15 | 7.61 | 1.20 |
| **pH (KCl)** | 113 | 3.38 | 7.70 | 5.19 | 1.29 |
| **EC (microS/cm)** | 326 | 21.00 | 9720.00 | 316.86 | 629.05 |
| **$CaCO_3$ (%)** | 324 | 0.00 | 53.10 | 7.64 | 10.70 |
| **humus (%)** | 326 | 0.02 | 44.05 | 1.70 | 2.62 |
| **Humus quality (Q) according Hargitai** | 237 | 0.13 | 177.25 | 15.66 | 29.29 |
| **Humus quality (K) according Hargitai** | 237 | 0.02 | 69.60 | 6.79 | 11.41 |
| **MSZ_clay (%)** | 324 | 0.19 | 67.75 | 26.41 | 14.00 |
| **MSZ_silt (%)** | 324 | 1.31 | 77.90 | 44.50 | 18.51 |
| **MSz_sand (%)** | 324 | 0.46 | 96.30 | 29.09 | 27.86 |
| **LDM_clay (%)** | 326 | 2.29 | 47.89 | 25.02 | 10.92 |
| **LDM_silt (%)** | 326 | 2.41 | 61.61 | 43.35 | 14.87 |
| **LDM_sand (%)** | 326 | 3.92 | 95.30 | 31.63 | 23.42 |
| **Spec. surf. area ($m^2$/g)** | 257 | 0.38 | 61.48 | 21.59 | 14.84 |
| **C/N-ratio** | 198 | 5.26 | 50.22 | 9.52 | 4.66 |
| **Total Nitrogen (%)** | 198 | 0.03 | 2.23 | 0.14 | 0.22 |
| **CEC (meq/100g)** | 306 | 0.75 | 112.00 | 24.28 | 12.09 |
| **Exchangeable Ca_mmol_100g** | 306 | 0.10 | 86.40 | 16.84 | 10.30 |
| **Exchangeable Mg_mmol_100g** | 306 | 0.05 | 17.60 | 3.55 | 2.68 |
| **Exchangeable Na_mmol_100g** | 306 | 0.01 | 25.70 | 0.80 | 2.48 |
| **Exchangeable K_mmol_100g** | 306 | 0.04 | 2.62 | 0.51 | 0.56 |
| **Base saturation_mmol_100g** | 306 | 0.44 | 106.79 | 21.70 | 12.17 |
| **Soda content (%)** | 44 | 0.00 | 0.23 | 0.06 | 0.07 |
| **y1** | 114 | 0.00 | 37.20 | 8.53 | 10.10 |
| **y2** | 72 | 0.00 | 19.04 | 2.77 | 4.62 |
| **$hy_1$** | 306 | 0.14 | 11.75 | 2.29 | 1.54 |
| **Munsell soil colour -labor** | 253 | | | | |
| **Munsell soil colour- field** | 253 | | | | |
| **bulk density (g/cm3)** | 229 | 0.72 | 1.83 | 1.45 | 0.16 |
| **pF0** | 229 | 35.53 | 76.25 | 48.03 | 6.21 |
| **pF0.4** | 229 | 34.53 | 74.56 | 47.17 | 6.09 |
| **pF1.0** | 229 | 32.94 | 69.26 | 44.19 | 5.41 |
| **pF1.5** | 229 | 32.13 | 67.13 | 41.90 | 5.26 |
| **pF2.0** | 229 | 11.43 | 65.69 | 38.43 | 6.59 |
| **pF2.3** | 229 | 7.51 | 62.39 | 35.14 | 7.90 |
| **pF2.5** | 229 | 6.51 | 60.21 | 33.40 | 8.18 |
| **pF3.4** | 229 | 2.45 | 54.53 | 22.85 | 9.38 |
| **pF4.2** | 229 | 1.31 | 41.41 | 15.05 | 7.21 |
| **NAPL retention** | 229 | | | | |
| **Knearsat (cm/s)** | 178 | 0.00 | 0.00 | 0.00 | 0.00 |
| **Ksat (cm/day)** | 229 | 0.00 | 839.08 | 60.22 | 120.30 |
| **Vageler type structure factor** | 251 | 2.15 | 94.10 | 58.02 | 15.73 |
| **MaAS (%)** | 224 | 7.67 | 98.24 | 62.98 | 20.65 |
| **MiAS %** | 326 | -54.47 | 100.00 | 49.72 | 23.03 |
| **Aggregate size distribution** | 223 | | | | |
| **Gravel content** | 223 | | | | |
| **Morphologi G3 shape analysing** | 31 | | | | |
| **Soil mineral composition** | 162 | | | | |
| **Clay mineralogy** | 43 | | | | |
| **Soil XRF measurements** | 137 | | | | |

The database has been presented at conferences so far (Ágfalvi et al., 2018; Makó et al., 2018b), but we are planning to produce a small information booklet.

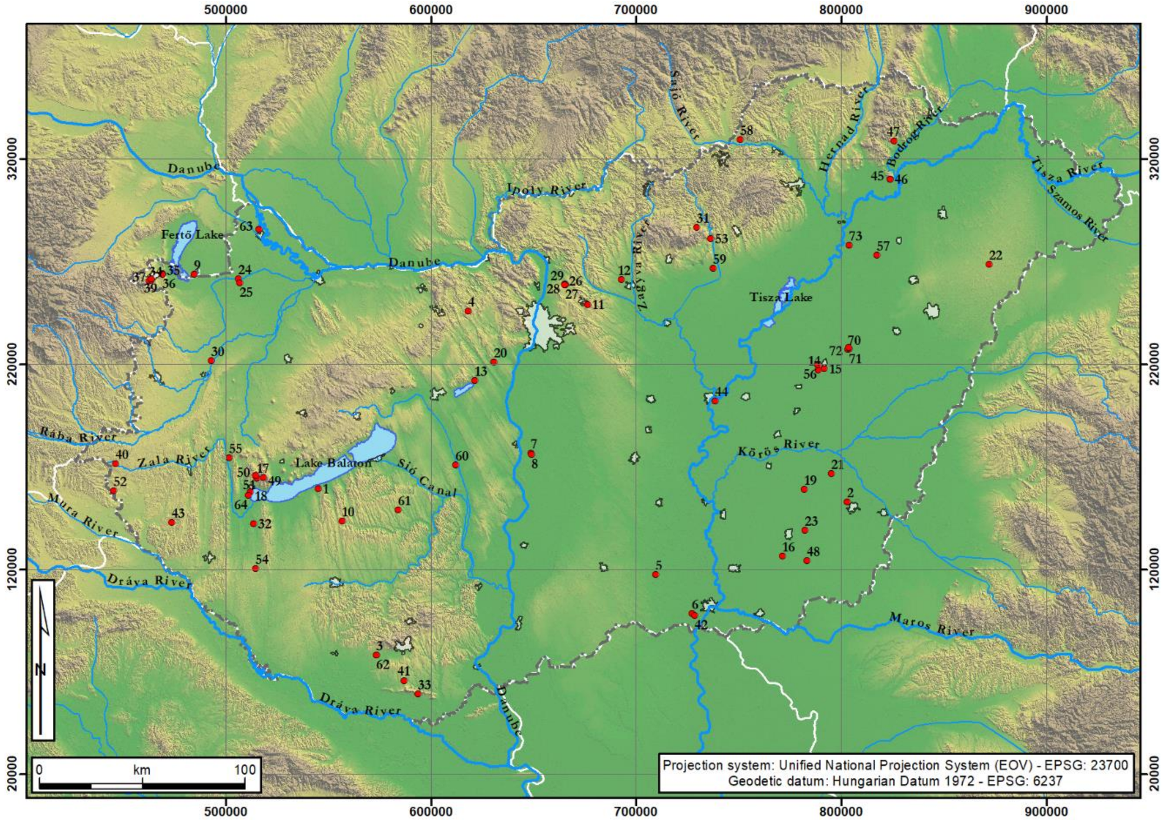


*Figure 2.*
Spatial distribution of soil profiles in the HunSSD

### II/B. Estimation and measurements of particle size distribution

In the years preceding the research, we were actively involved in the development of both national (MARTHA, Polish soil physical database) and continental (EU-HYDI, LUCAS) scale large soil physical databases with our Polish colleagues and also carried out some of the measurements required for these. At that time, we were confronted with the fact that the diversity of particle size distribution (PSD) measurements (different pretreatments, and methods) makes it difficult to compile uniform databases, and some kind of conversion between the results of different methods is strictly necessary. This is of particular importance if the aim is to develop pedotransfer functions (PTF) estimating soil hydrological properties based on the data in the databases. We also saw a need to carry out methodological studies that could help standardize new PSD measurement methods in the longer term.

Existing databases in the first years of the research project were used to investigate the conversion potential. Completing the work started in a previous research project, a new estimation procedure was developed on the 400 soils of the LUCAS database. This has allowed us to create a conversion between laser diffraction measurements (LDM) with the Malvern Mastersizer 2000 apparatus and the ISO standard sieve-pipette measurements (SPM) with different accuracies (Makó et al., 2017c). This estimation method, developed on a continental scale, was subsequently tested and refined on a regional scale (Tokaj hillslope) database (here we investigated the possibility of conversion between

SPM PSD measurements according to the national MSZ standard and LDM measurements with Mastersizer 2000) (Makó et al., 2019a).

In later years, when the HunSSD was also completed, the conversion method was successfully tested on a national scale on a database of representative Hungarian soil profiles (Makó et al., 2022c). Most recently, we successfully tested the effectiveness of the conversion method on the Polish soil physical database with Polish colleagues (Polakowski et al., 2023). The conversion method was also successfully used to process data from LDM PSD measurements on forest soils (Szecsődi et al., 2021).

Similar to the previous ones, we have developed a conversion method between two common SPM PSD methods (Hungarian and ISO standards), based on a larger existing national dataset from our own research, with the aim of creating data harmonization (Makó et al., 2017b).

Another direction of PSD research has been the development of standardization options for the measurement LDM methodology. For this purpose, we collected and systematized existing literature (Makó et al., 2017a; Bieganowski et al., 2018) and started our own methodological studies (mainly on HunSSD samples). To evaluate and interpret the effects of different pretreatment methods and settings a series of LDM PSD measurements were performed. Results illustrate setting (Ryżak et al., 2018) and preparation (Földényi et al., 2018; Labancz et al., 2018a; Labancz et al., 2018b; Labancz et al., 2018d; Barna et al., 2022b; Polakowski et al., 2022) details' importance of the LDM and call attention to the need of working out standard protocols for the LDM PSD measurements.

As a transition between PSD research and soil hydrology research, our studies express both the grain size distribution and the pore size distribution of soils by calculating fractal dimensions and investigate the relationship between the two to develop a possible estimation direction. These studies were started on a national scale on the MARTHA database (Rajkai et al., 2022).

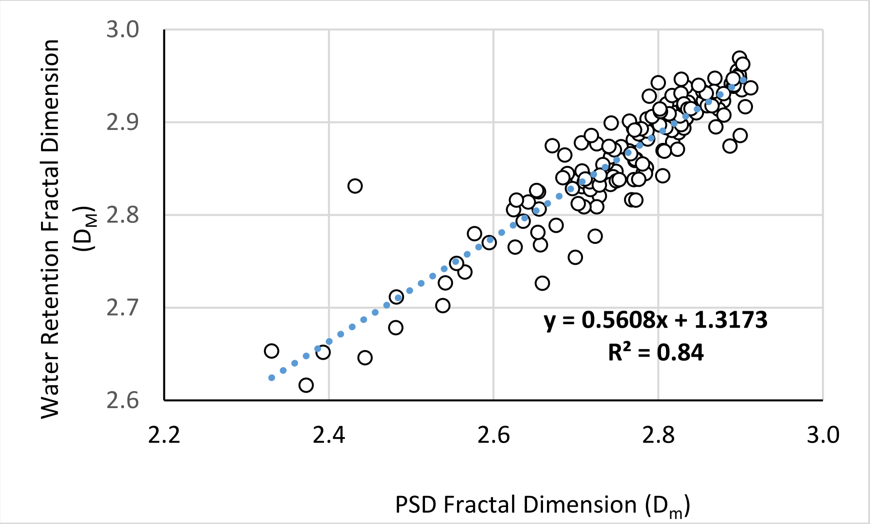


*Figure 3.*
Correlation of WRC and PSD fractal dimensions in MOTKA soil samples.

Currently using the collected soil data of HunSSD, a novel domestic experiment was started to estimate the soil's water retention properties from PSD data and some other easily measurable soil characteristics, such as bulk density. In the case of the novel pedotransfer functions, we first calculated the mass fractal dimension $D_m$ of the PSD data determined by the traditional pipette method. (We intend to include also the LDM PSD values in future studies.) $D_m$ was calculated according to Tyler and

Wheatcraft (1992). To calculate the $D_M$ fractal dimension of the pore size distribution, we used the measured water retention characteristic (WRC) data of the HunSSD. $D_M$ was calculated according to Tyler and Wheatcraft (1989). Since the correlation of the fractal dimensions $D_M$ and $D_m$ is highly significant ($R^2=0.84$) shown in *Figure 3*, we expect a good estimation of the water retention data or function using the pedotransfer function will be determined by the fractal dimensions of the soils.

A publication is currently being prepared from our studies (Rajkai et al, 2023).

## II/C. Measurements and estimations of soil hydrological properties

Water and nonaqueous phase liquid (NAPL) retention tests were carried out on undisturbed soil samples taken from different layers of the soil profiles of the HunSSD database to study the interrelationships between the PSD, structure and pore size distribution (in connection with fluid retention) of soils allowing to refine existing estimation methods (PTFs). A comparison of pore size distributions calculated from the results of water and NAPL retention studies was first presented at two conferences (Makó & Hernádi, 2016, Hernádi et al., 2018). Later, as fluid retention results were continuously accumulated in the HunSSD database, we investigated the relationship between calculated pore size distributions and aggregate stability with the aim of developing new soil structure indicators (see structure studies) (Makó et al., 2022b). In our research we found that the determination of NAPL retention and conductivity based on the commonly used simplified empirical prediction methods using the hydraulic properties of soils is fairly questionable because of the different phase interactions might be ascertained in air/water/soil, as opposed to in air/NAPL/soil systems. Interactions which led to various degrees of structural changes depending on the properties of the solid and the liquid phase might cause enormous alteration in soil hydrological parameters. In our study, water and NAPL (DUNASOL 180/220) retention of undisturbed soil samples were measured and the modified three-parameter van Genuchten function was fitted to the measured data. Our results verified that significant differences might be found between NAPL and water retention curves of soil samples, and differences between total porosity and pore size distribution of soils calculated on the basis of NAPL and water retention exists, as well. Structure might undergo remarkable changes in the case of water saturation of the soil samples (which might be resulted primarily from polar solid surface – polar liquid molecules interactions, namely disaggregation or swelling processes), but no significant structural changes were experienced in the case of NAPL saturation. Ratio of pore size classes (especially the proportion of macropores) measured with different liquids can be used as an indicator of soil aggregate stability (*Figure 4*). Our research results are in a pre-publication state. Estimation of the porosity and the pore size distribution using unimodal empirical functions might give opportunity for comparison of the structural changes during different fluid saturation. However, unimodal functions might give poor estimations near saturation. It is possible that the bimodal or multimodal functions would give more reliable retention and PoSD results, we intend to continue our research in this direction in the future. We have published two review articles (Hernádi et al., 2017a; 2017b) and a PhD thesis was also written on this topic (see Chapter III).

In the HunSSD soils, we had originally intended to measure both saturated hydraulic conductivity and NAPL conductivity, similar to the liquid retention capacity. However, our intention to do so was thwarted for methodological reasons (the special sample preparation for NAPL measurements destroyed most of our samples). We were therefore unable to carry out the planned comparative analyses. Instead, a new measurement method was introduced: in addition to the saturated hydraulic conductivity ($K_{sat,measured}$), the so-called matrix conductivity ($K_{nearsat}$) was determined on the undisturbed soil samples with Mini-disc infiltrometer in laboratory. We have found that most of the $K_{sat,measured}$

values were higher compared to $K_{nearsat}$, which showed that water flow through macropores was eliminated by adjusting constant suction (Ujj et al., 2018; 2019). A statistical analysis of the HunSSD conductivity data is still underway, with publication planned at a not too distant date.

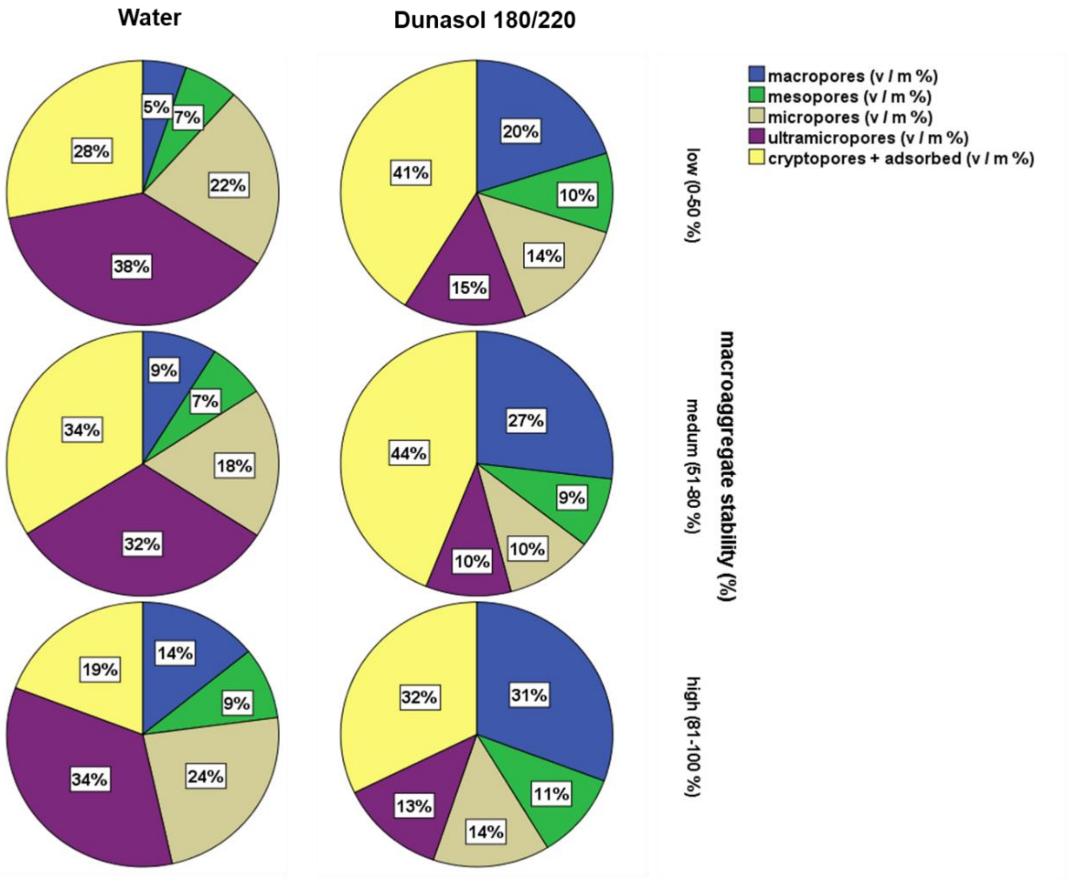


*Figure 4.*
Presentation of pore size distributions calculated from the liquid retention curves of soils, grouped according to the macroaggregate stability of soils

However, measured water retention and conductivity data from some HunSSD soil profiles have already been used to model the water balance of the selected soils. In these studies, we investigated whether procedures for estimating soil hydrological parameters based on different datasets (i) provide a reliable input data source and (ii) improve the goodness of fit of hydrological calculations in soil profile-level hydrological models compared to previously available datasets (Kozma et al., 2019a; 2019b; 2023).

In the meantime, we have also written a book chapter on the water retention of soils, in which we have also described new methodological options for estimation and the factors influencing it (including soil structure) (Rajkai et al., 2018).

The water retention and conductivity results measured in the HunSSD soils have been incorporated into the MARTHA database, thus enriching it with new data sets.

One of the main objectives of the project was to further develop methods for estimating soil hydrological properties. These objectives could be supported by analyses of existing databases using new approaches, and by a series of statistical analyses on the completed HunSSD database. An example of the former is the work carried out with my colleagues in the Lake Balaton catchment. Soil hydraulic properties of the catchment can be calculated by applying PTFs on available soil maps. Our aim was to analyse the performance of (i) indirect (using PTFs) and (ii) direct (geostatistical) mapping methods to derive 3-D soil hydraulic properties. Maps of saturated water content (0 cm matric potential), field capacity ( − 330 cm matric potential) and wilting point (−15 000 cm matric potential) for 0–30, 30–60 and 60–90 cm soil depth were prepared. PTFs were derived using the random forest method on the whole Hungarian soil hydraulic dataset (MARTHA). As a direct and thus geostatistical method, random forest combined with kriging (RFK) was applied to 359 soil profiles located in the Balaton catchment area. Detailed comparisons of predicted maps derived from the PTF-based method and the RFK were presented in a paper (Szabó et al., 2019). The development of estimation methods based on the HunSSD data (inclusion of soil structure information in the estimation methods) is also ongoing, and we intend to publish the first results of our studies in this area also in the not too distant future (Makó et al., 2023a).

Also during the project, we started to update the soil water management classification system - on a statistical basis. Our first results were presented at a conference (Szabó et al., 2020).

### II/D. Estimation and measurements of micro- and macro-aggregate stability

First, the relationships between the soil structure information retrieved from the MARTHA database and the pore size distributions calculated from the pF-curves were studied (Barna et al., 2016). To estimate soil structural degradation hazard, class-based relationships were developed based on soil profile data of MARTHA. Soil type, organic matter content, carbonate content, soil reaction and texture class (USDA) were taken into consideration to develop pedotransfer functions for modelling the correlations between primary soil properties and threats indicators (Bakacsi et al., 2019).

The use of the pore size distributions calculated from the results of fluid retention measurements in aqueous and non-aqueous systems for the indication of soil structure stability has already been reported in Chapter II/C.

One of the main planned directions of soil structure research was to study the methodology of measuring the stability of macro- and micro-aggregates in soils and to develop possible estimation methods.

Literature experience in measuring macroaggregate stability is summarized in a review article (Labancz et al., 2021). (We are also planning a follow-up article on microaggregate stability.)

Using the Eijkelkamp-type macro-aggregate stability device, which was purchased as part of the research project, we started our methodological investigations. We have studied the appropriate methodology (dispersant, pre-wetting) in aqueous media, but we have also performed tests in non-aqueous media with Dunasol 180/220. We also developed a measurement methodology for the determination of the microaggregate stability of soils using the analogy of the Vageler-type structure factor (measured by pipette method) with laser diffractometry (Malvern Mastersizer 2000 and 3000). Using the same method, we were able to measure the dynamics of soil disaggregation (the temporal dispersion of aggregates). These studies were also carried out as a preliminary experiment on a sample population in a non-aqueous medium. Our experience in this area has been presented at conferences and published (Barna et al., 2017; 2022a; 2022d; 2023; Makó et al., 2018a; 2022a; 2022c; Kiss et al., 2018).

The methods developed in the project were then successfully applied to newly involved studies where aggregate stability was an important soil indicator. Thus, for example, both macro- (MiAS) and micro-aggregate (MiAS) stability measurements have been fruitfully applied to investigate the structure stabilizing effect of different soil additives: lignite (Labancz et al., 2018); biochar (Horel et al., 2019; Barna et al., 2020; Makó et al., 2020); vermicompost (Rivier et al., 2022). The methods were applied to study the aggregate stability of soils incubated in a reducing environment to investigate the soil physical effects of persistent inland water cover (Sebők et al., 2018).

The study of saline soils is a shaky area of soil physics. We reported on our experience with macroaggregate stability measurements on HunSSD saline soils at a conference in China. We demonstrated, among other things, that there are great differences between topsoil and subsoil horizon's aggregate stability, since soil organic matter is the determinant factor of MaAS. Other factors affecting MaAS were alkalinity, sodicity, salinity. Grassland topsoil horizon showed larger aggregate stability than croplands (Barna et al., 2021).

Using the micro- and macro-aggregate measurement data and other measurement data from the HunSSD database, we had the opportunity to study the potential of estimating aggregate stability of soils using data mining methods. In particular, we were interested in the extent to which the reliability of aggregate stability estimates can be increased by increasing the detail of soil information, and the potential for aggregate stability estimates when relatively little is known about a soil. As these studies have only been presented at conferences (Makó et al., 2022a) and are in manuscript form unpublished (Makó et al., 2023b), we describe them in a little more detail. The statistical processing methodology was as follows. The vast majority of samples were measured in 3 replicates. As a first step replicates of the same sample were averaged. XGBoost algorithm (Chen and Guestrin, 2016) was used to find predicting model for MiAS and MaAS. The optimal hyperparameters of the model was searched for by means of LOOCV cross-validation on the following way. Randomly selected 20% of the profiles were kept as test set. Parameter tuning was performed on the remaining 80% of profiles. In each step one profile was used as validation profile, while the model was trained on the others. The performance of the model was assessed by comparing the predicted values of validation profile to its observed values. This training-assessment process was repeated until each profile was used as validation-profile. After that RMSE was calculated from the predicted and observed data. Due to the nature of a random tree model different group of predictor variables may have been selected in each training step. The aim of cross-validation was to find the optimal values of hyperparameters and not the optimal set of predictor variables. 2 hyperparameters, the learning rate and the maximum depth of a tree were searched for the cross-validation, while the others were set to constant value. Learning rate was varied among 0.05, 0.15, 0.2, 0.25, maximum depth was varied from 1 to 6. Subsample ratio was set to 1, column subsampling ratio to 0.7, gamma to 0, maximal number of boosting iterations to 150, weights of both L1 and L2 regularization to 0. Finally, the hyperparameter combination corresponding to maximal RMSE was used to train the model on the whole train-validation set. This model-fitting process was repeated with different groups of predictor variables. Calculations were performed by using the R statistical software (R Core Team, 2022) and the xgb package (Chen et al., 2022). Our results indicate that both MaAS and MiAS values can be estimated from the soil properties with about medium accuracy ($R^2$ = 0.49 and $R^2$ = 68, respectively), thus, we were able to estimate MiAS results from soil properties more reliably.

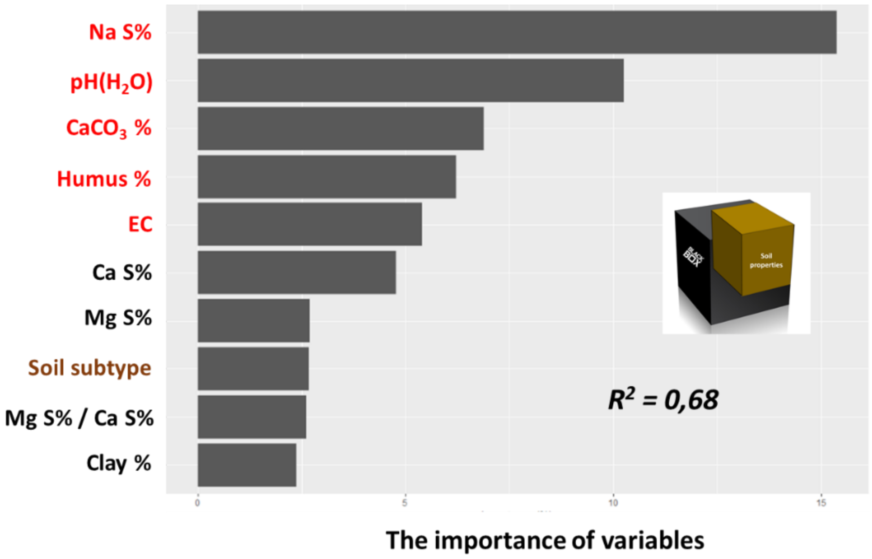


*Figure 5.*
Microaggregate stability (MiAS %) in relation to soil properties

The most important soil parameter in the MaAS estimates was the humus content of the soils, followed by soil pH. For the MiAS estimates, exchangeable Na content and pH were the most important soil properties, followed in order of importance by $CaCO_3$ and humus content (*Figure 5*).

## II/E. Morphological description of soil aggregates

The structural morphological properties of the soils were partly recorded and photographed during the field section description and partly investigated with the help of our Polish colleagues at the Institute of Agrophysics in Lublin using a Morphologi G3SE ID shape analyzer (Malvern, UK). The results are still being evaluated and integrated into the database.

## II/F. Other studies related to the database

The data from the HunSSD database allowed us to identify and describe other correlations between soil properties measured on samples representative of the country's soils. On the one hand, we investigated the relationships between BET specific surface area, humus substances and other soil properties on typical Hungarian soil types (Molnár et al., 2019; 2020). On the other hand, similar studies were carried out between the cation exchange capacity of soils and other soil properties (Barna et al., 2022c).

From the soil profiles of the HunSSD, we selected those that contained iron concretions and collected these separately at the time of sampling. Geochemical methods were used to investigate the characteristics of iron concretions on different soil types (Sipos et al., 2022; 2023).

From the above it can be seen that although the project has been completed, the planned research is far from being finished, there are many unpublished results and further studies are planned on the HunSSD database. The ongoing K 134563 NKFIH project („Development of new soil structure indicators to characterize the hydrophysical effects of soil degradation processes") can be seen as a continuation of the current project, in which we intend to continue the work started here.

## III. Other results of the research

PhD students and BSc, MSc thesis students were also involved in the research. The following papers were produced during the research:

Hilda Hernádi (2020): Possibilities for estimating soils' NAPL retention with pedotransfer-type relationships. Doctoral (PhD) thesis. University of Pannonia, Georgikon Campus, Festetics Doctoral School. Keszthely.

Henrietta Janek (2017): The applicability of laser diffraction method during the routine particle size distribution analysis in soil physics. BSc thesis. University of Óbuda. Budapest.

András Kallós (2017): Studying the relationship between soil particle size distribution and environmentally important soil constituents. MSc thesis. University of Pannonia. Veszprém.

Márta Kertész-Kollár (2021): Quantitative and qualitative analysis of soil organic matter and macroaggregate stability in different soil types. BSc thesis. Hungarian University of Agriculture and Life Sciences, Szent István Campus, Gödöllő.

Károly Kiss (2018): Experiences of the soil aggregate stability measurements. BSc thesis. University of Óbuda. Budapest.

Viktória Labancz (2020): Methodological studies for the adaptation of particle size distribution measurements with laser diffractometers to soil physics. Dissertation thesis. Szent István University, Szent István Campus, Gödöllő.

Tibor Németh (2020): Mineralogical and clay mineralogical characterisation of the main Hungarian soil types. Dissertation thesis. Szent István University, Szent István Campus, Gödöllő.

Orsolya Szecsődi (2020): Granulometric analysis of soil: comparison of „pipette" sedimentation and laser diffraction particel size analysis methods. BSc thesis. University of Sopron. Sopron.

Márton Tóth (2021): Comparison of the information content of different soil classification systems on representative Hungarian soil profiles. Dissertation thesis. Hungarian University of Agriculture and Life Sciences. Georgikon Campus, Keszthely.

Other projects to which we have been associated during or after the project with the results of the project and/or with the research methods developed:

"Development of new soil structure indicators to characterize the hydrophysical effects of soil degradation processes" (NKFIH K-134563., 2020-2025, project leader: András Makó);

„Development of a new soil water-holding capacity model" (NKM-108/2017, project-based bilateral grant between the Institute of Agrophysics of the Polish Academy of Sciences in Lublin and TAKI, 2017-2019, project leader: András Makó);

"Comparison of aggregate stability of soils measured in water and non-aqueous phase liquid (NAPL)" (NKM 2019-17. project-based bilateral project between the Institute of Agrophysics of the Polish Academy of Sciences in Lublin and TAKI, 2020-2023, project leader: András Makó);

"Impact of biochar application on soil nitrogen cycling in different land use systems" (OTKA PD-116157, project leader: Ágota Horel);

"Utilisation of organic manures from livestock production and the nutrient content of organic wastes, which play a key role in the development of a circular economy" AM project, 2021, project leader: Nikolett Uzinger);

"Development of the best large-scale mapping method for saline soils in areas with different land use" (NKFIH K-142290, 2017-2022, project leader: Tibor Tóth, then László Pásztor);

"Indication of soil organic carbon stock quality" (ELKH Priority Research Project SA-26/2021, 2021-2023, project leader: Tünde Imréné Takács);

"National Laboratory for Water Science and Water Safety" (RRF-2.3.1-21-2022-00008, 2022-2026, project leader in TAKI: László Pásztor);

"National Programme for Sustainable Development and Technologies - Soil-environment interaction studies in the light of soil safety, sustainable soil quality, environmental and food safety" (NP2022-II-2/2022, 2022-2026, project leader in TAKI: László Pásztor)

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