Comparison of PSD methods

András Makó

3 examples

1. Comparison of two sieve-sedimentation (pipette) methods:
    - Hungarian standard (MSZ-08-0205-78)
    - ISO/DIS standard (ISO/DIS 11277:1994; ISO/DIS 11277:2009E)
2. Comparison of LDM and sieve-sedimentation (pipette) method:
    - Malvern Mastersizer 2000
    - ISO/DIS standard (ISO/DIS 11277:2009E)
3. Comparison of LDM and sieve-sedimentation (pipette) method:
    - Malvern Mastersizer 2000
    - Hungarian standard (MSZ-08-0205-78)

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A talajok mechanikai összetétel vizsgálata pipettás ülepítéses módszerrel: a hazai és a nemzetközi szabvány szerinti eljárások összehasonlítása és konverziója

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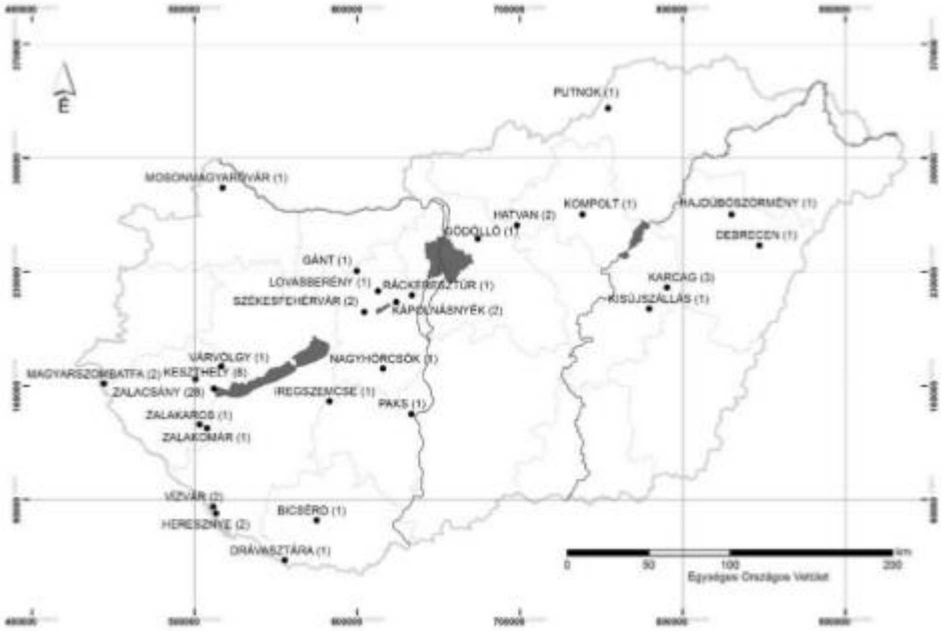


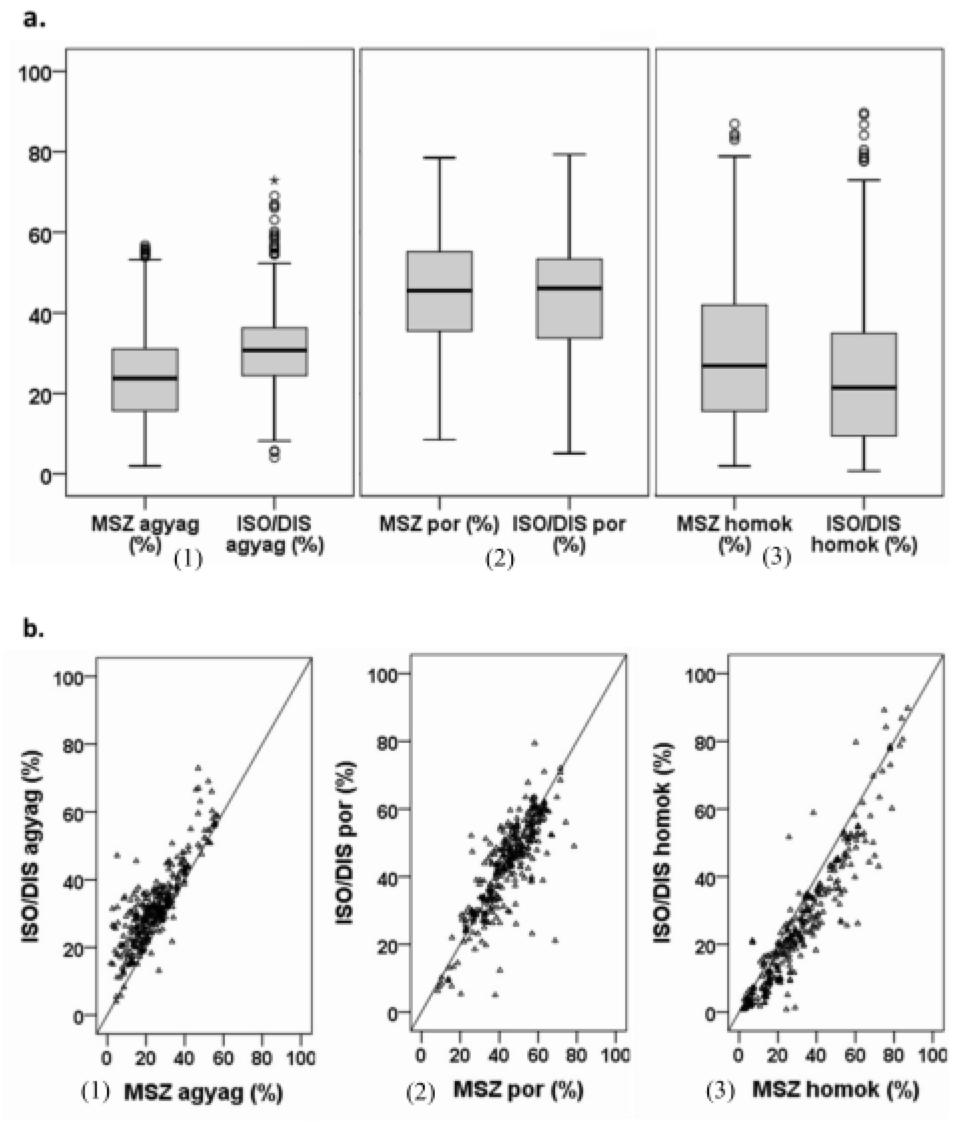
1. ábra
A mechanikai összetétel adatbázis talajmintáinak származási helye (zárójelben a feltárt szelvények száma)

69 soil profiles; 339 soil samples

Two sieve-sedimentation (pipette) methods:

- Hungarian standard (MSZ-08-0205-78)
  - Only dispersion
- ISO/DIS standard (ISO/DIS 11277:1994; ISO/DIS 11277:2009E)
  - Organic matter, $CaCO_3$ and Fe-oxi-hydroxid removal and dispersion

A suggestion was made of how these results could be converted into each other.



2. ábra
A két módszerrel meghatározott mechanikai összetétel frakciók összehasonlítása

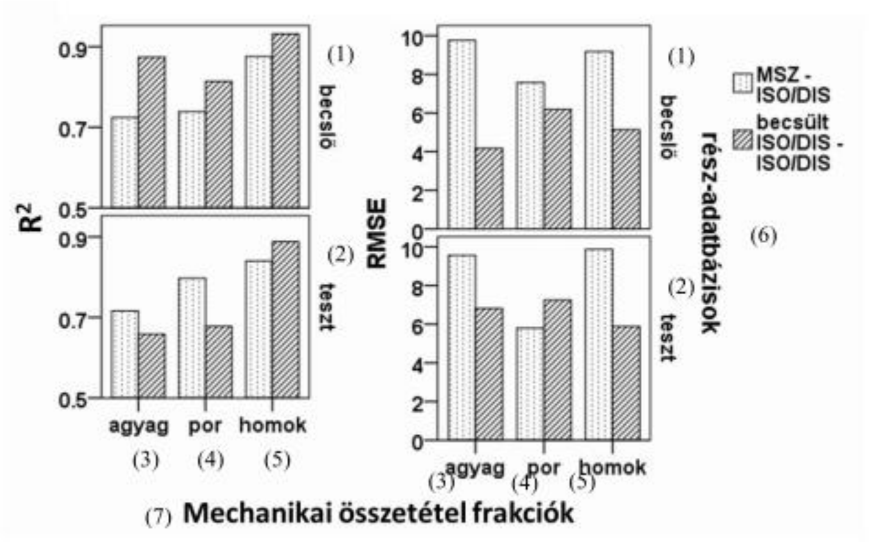
- the pre-treatments applied as part of the ISO/DIS method may change the ratio of particle size fractions
- there was a significant increase in the clay content
- the silt content decreased to a lesser and the sand content to a greater extent,
- possibly because some of the particles remain in micro-aggregate form when the MSZ method is used.

2. táblázat

Az ISO/DIS agyag-, por és homokfrakciók becslésére javasolt pedotranszfer függvények

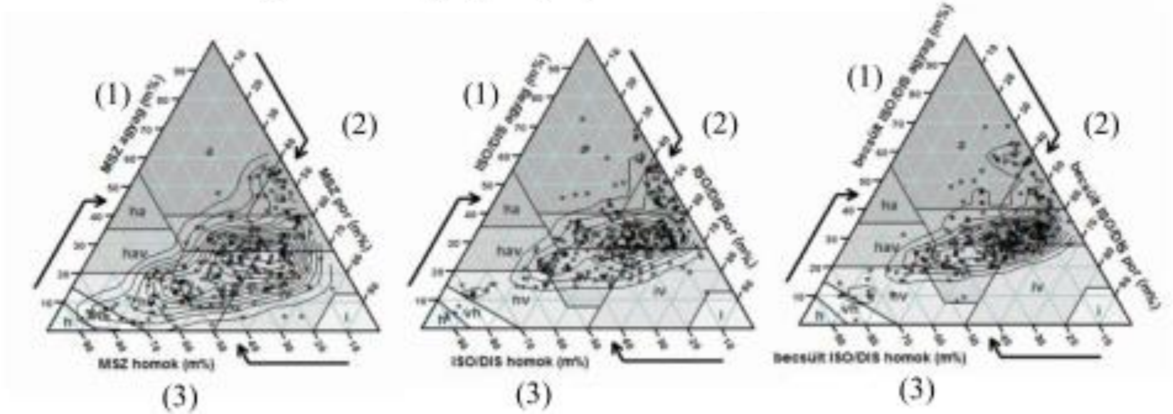
(1) becsült $agyag_{ISO/DIS}$* = 64,501+ 0,013 * $(A_{MSZ})^2$ – 641,424 * (1 / P_{MSZ}) – 0,019 * (A_{MSZ} * P_{MSZ}) + 0,011 * $(CA)^2$ – 0,100 * (H_{MSZ} * OM) – 0,214 * $(pH_{DV})^2$ + 2,977 * (1 / OM) – 0,090 * (P_{MSZ} * OM) + 1,212 * (pH_{DV} * OM) – 0,007 * (OM_{MSZ} * CA) – 0,009 * (P_{MSZ} * H_{MSZ}) + 0,032 * (A_{MSZ} * pH_{DV}) – 0,859 * $(OM)^2$ + 0,002 * (1 / CA) + 4,868 * OM
(2) becsült $homok_{ISO/DIS}$ = – 3,828 + 475,114 * (1 / P_{MSZ}) + 0,009 * (H_{MSZ} * CA) – 0,008 * $(CA)^2$ + 0,374 * H_{MSZ} + 0,005 * (A_{MSZ} * H_{MSZ}) – 0,002 * $(A_{MSZ})^2$ – 0,017 * (H_{MSZ} * OM) + 0,003 * $(H_{MSZ})^2$
(3) becsült $por_{ISO/DIS}$ = 100 – (becsült $agyag_{ISO/DIS}$ + becsült $homok_{ISO/DIS}$)

*A_{MSZ}, P_{MSZ}, H_{MSZ}: az MSZ módszerrel mért MÖ (agyag-, por-, homok-) frakciók (< 0,002 mm, 0,002–0,05 mm, > 0,05 mm) (%); OM: humusz tartalom (%); CA: $CaCO_3$ tartalom (%); pH_{DV}: desztillált vizes szuszpenzióban mért pH

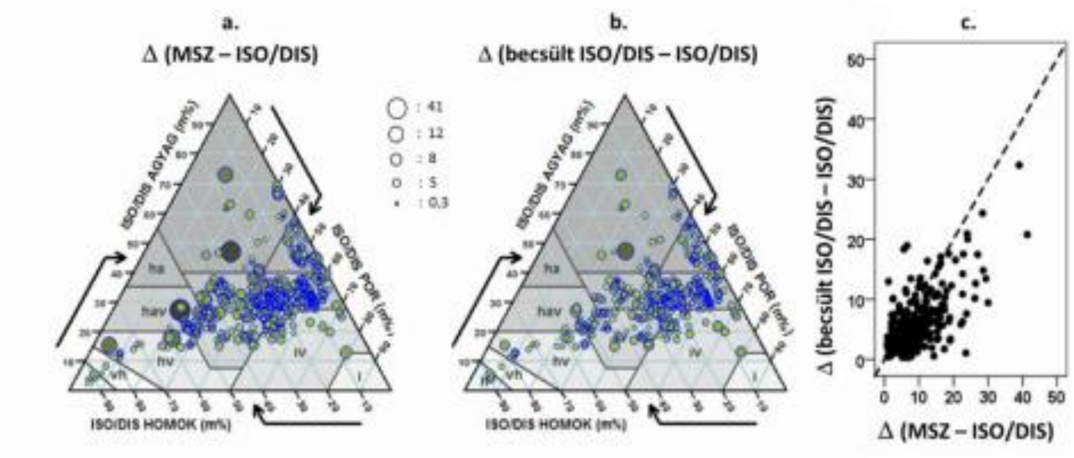


4. ábra
A javasolt konverziós egyenletek becslési pontosságának és megbízhatóságának bemutatása (R^2: a mért MSZ és ISO/DIS frakciók, illetve az MSZ frakciókból becsült és mért ISO/DIS frakciók közti determinációs koefficiens; RMSE: a mért MSZ és ISO/DIS frakciók, illetve az MSZ frakciókból becsült és mért ISO/DIS frakciók közti átlagos négyzetes eltérés négyzetgyöke (m%))

- The estimated ISO/DIS fractions became much closer to the measured ones when the suggested pedotransfer functions were applied.
- The conversion method proved to be more reliable for the prediction of clay and sand content than for silt content.
- In its present form the estimation method is not suitable for replacing the ISO/DIS method, but it could be of good service in research and comparative analysis in cases where only the MSZ method can be used or where only old MSZ PSD data exist.



5. ábra
A különféle módszerekkel mért és a konverziós egyenletekkel becsült mechanikai összetétel eredmények összehasonlítása USDA háromszögdiagramokon, a Kernel sűrűségfüggvény ábrázolásával



6. ábra
A MÖ pontpárok térbeli távolsága a.) az MSZ MÖ és ISO/DIS MÖ, valamint b.) MSZ MÖ-ből pedotranszfer függvénnyel konvertált ISO/DIS és mért ISO/DIS MÖ háromszögdiagramokon ábrázolva (a nagyobb buborékok az egymástól messzebb, a kisebbek az egymáshoz közelebb elhelyezkedő pontpárokat jelölik), c.) a távolságok egymáshoz viszonyított aránya mintánként

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Pedotransfer functions for converting laser diffraction particle-size data to conventional values

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The LUCAS soil database

The Land Use/Land Cover Area Frame Survey (LUCAS) is the first consistent spatial database of the soil cover across Europe. Around 22 000 soil samples were collected from 10% of the survey points by standard sampling procedures, and were analysed in a certified laboratory with unified standard methods (Tóth *et al.*, 2013).

Among the LUCAS topsoil samples, 400 were selected to represent the variation and differentiation in soil cover (Table 1). The selection was stratified by texture classes and land cover, followed by simple random sampling in each stratum using the R package srswor (Tillé & Matei, 2015). Finally, we verified that the representation of soil characteristics (e.g. organic carbon and calcium carbonate content), climate zones and countries in the selected samples was comparable with that in the full dataset.

Sieve–pipette method (SPM). The PSD was determined by a combination of sieving and sedimentation, starting with about 20–30 g (depending on soil texture) air-dried soil (ISO 11277:2009). Particles of 63–2000 µm (sand fraction) were determined by a combination of wet and dry sieving. Particles passing the 63-µm sieve were determined by sedimentation with the pipette method. A particle density of 2650 kg m^{-3} was used to calculate the sedimentation time. 'Calgon' (containing 33 g sodium hexametaphosphate and 7 g anhydrous sodium carbonate in 1 l aqueous solution) was used for chemical dispersion. The pretreated suspension was shaken for 18 hours on an end-over-end shaker. The percentages (mass %) of the constituent fractions (sand, 63–2000 µm; silt, 2–63 µm; clay, < 2 µm) were obtained from the PSD analysis.

Laser diffraction method (LDM). A Mastersizer 2000 (Malvern Instruments, Malvern, UK) laser diffractometer, which measures within a size range of 0.02–2000 µm (Mastersizer 2000 User Manual, 1999), was used for LDM analysis (ISO 13320:2009). Measurements were made with a Hydro 2000G dispersion unit.

The PSD was usually determined on two or three replicates (measurement of distinct subsamples) with the LDM. If the shapes of the PSD curves of two repetitions were largely dissimilar, a third measurement was made. The mass of dry soil samples placed into the dispersion unit was in the range of 0.5–1 g depending on the 'obscuration' of the soil suspension after dispersion. In this context obscuration is a measure of the amount of the light scattered by the soil particles and correlates with the concentration of measured material present in the laser diffractometer. According to the Mastersizer 2000 manual, the obscuration values should be between 10 and 20%.

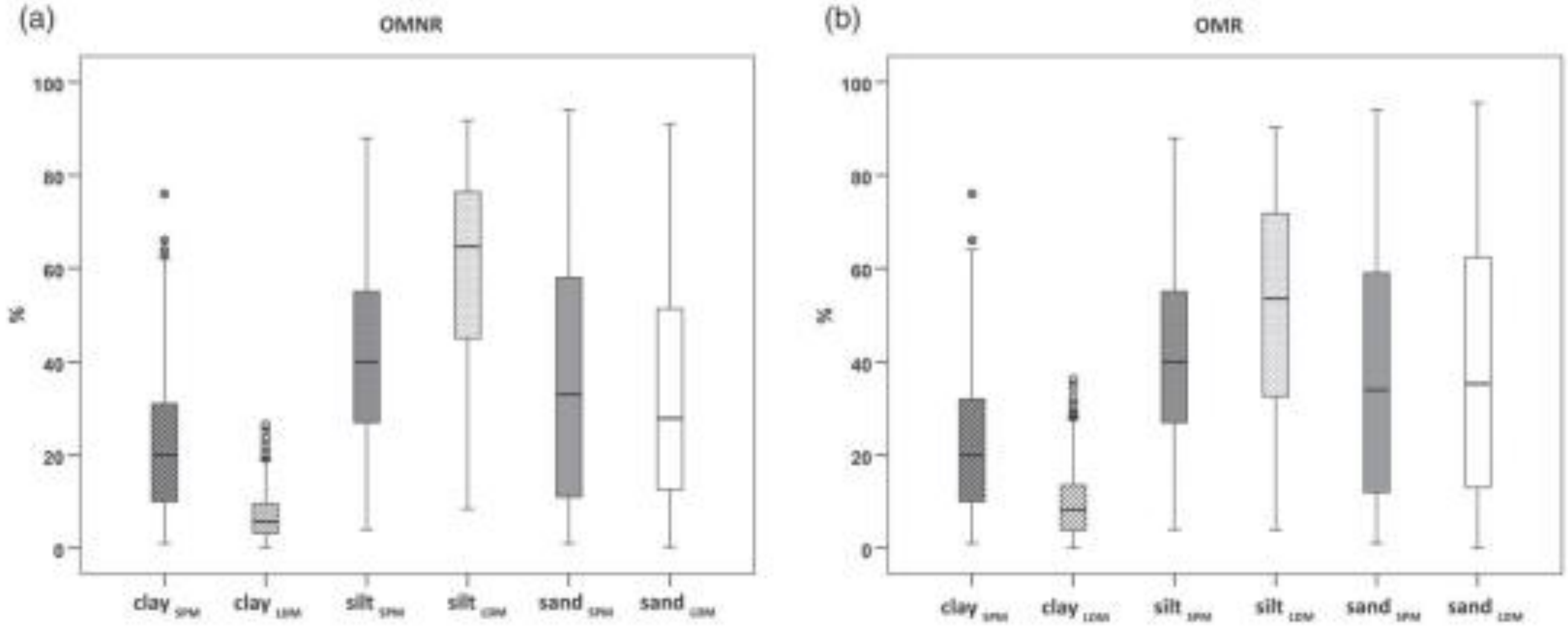

Figure 1 Comparison of the interquartile ranges of soil particle-size fractions measured by different (SPM, sieve–pipette; LDM, laser diffractometer) methods for different pretreatments: (a) OMNR, organic matter not removed; (b) OMR, organic matter removed.

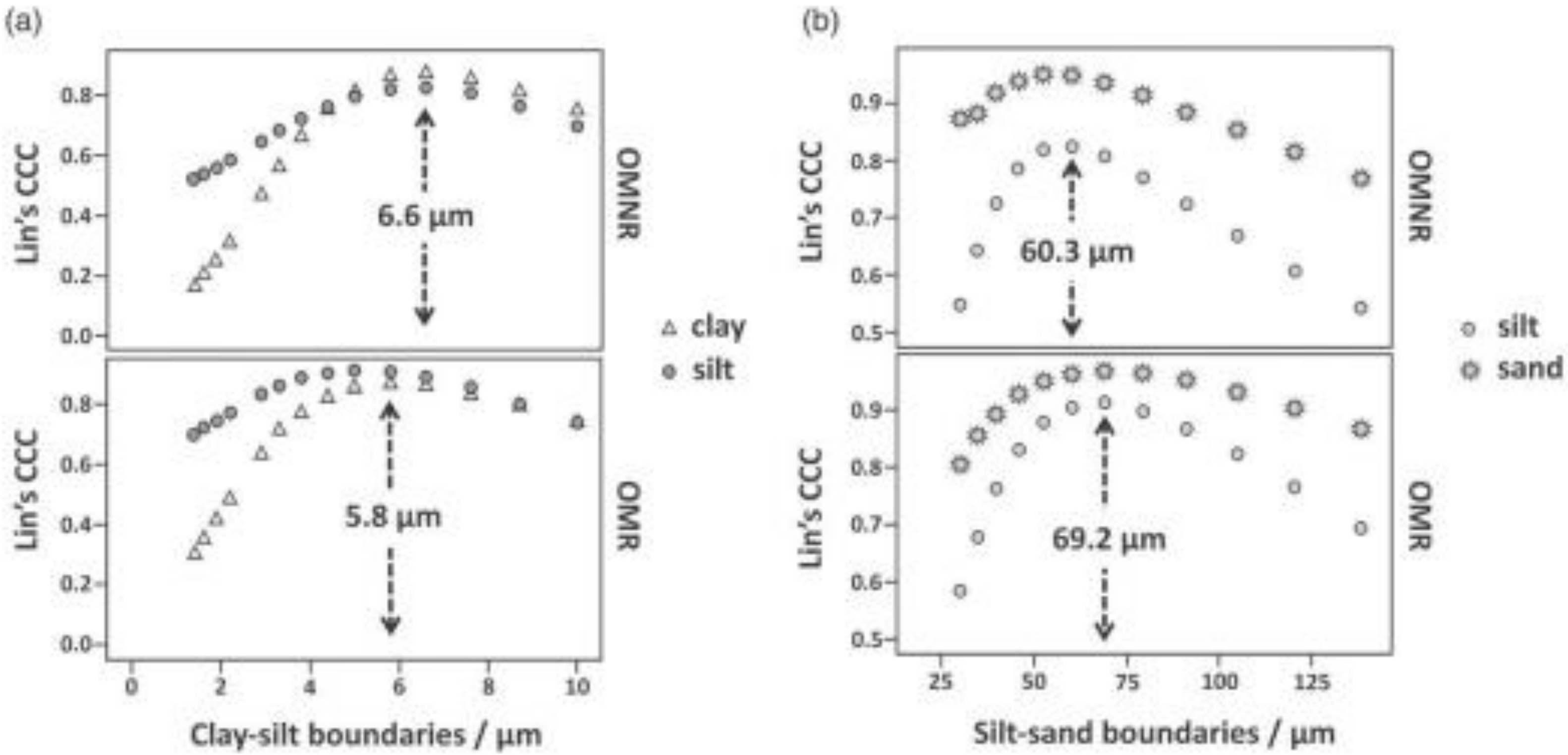

Figure 4 Lin's concordance correlation coefficient (CCC) values of the laser diffractometer methods (LDMs) and sieve–pipette methods (SPMs) (a) clay–silt and (b) silt–sand fraction boundaries measured for the different datasets (OMNR, organic matter not removed; OMR, organic matter removed). The *y*-axis gives Lin's CCC of the (a) 14 cumulative size classes of the LDM clay (from < 1.4 to < 10.0 µm) and the SPM clay (< 2 µm) fraction and (b) 12 cumulative size classes of the LDM sand (from > 30.2 to > 138.4 µm) and the SPM sand (> 63 µm) fraction.

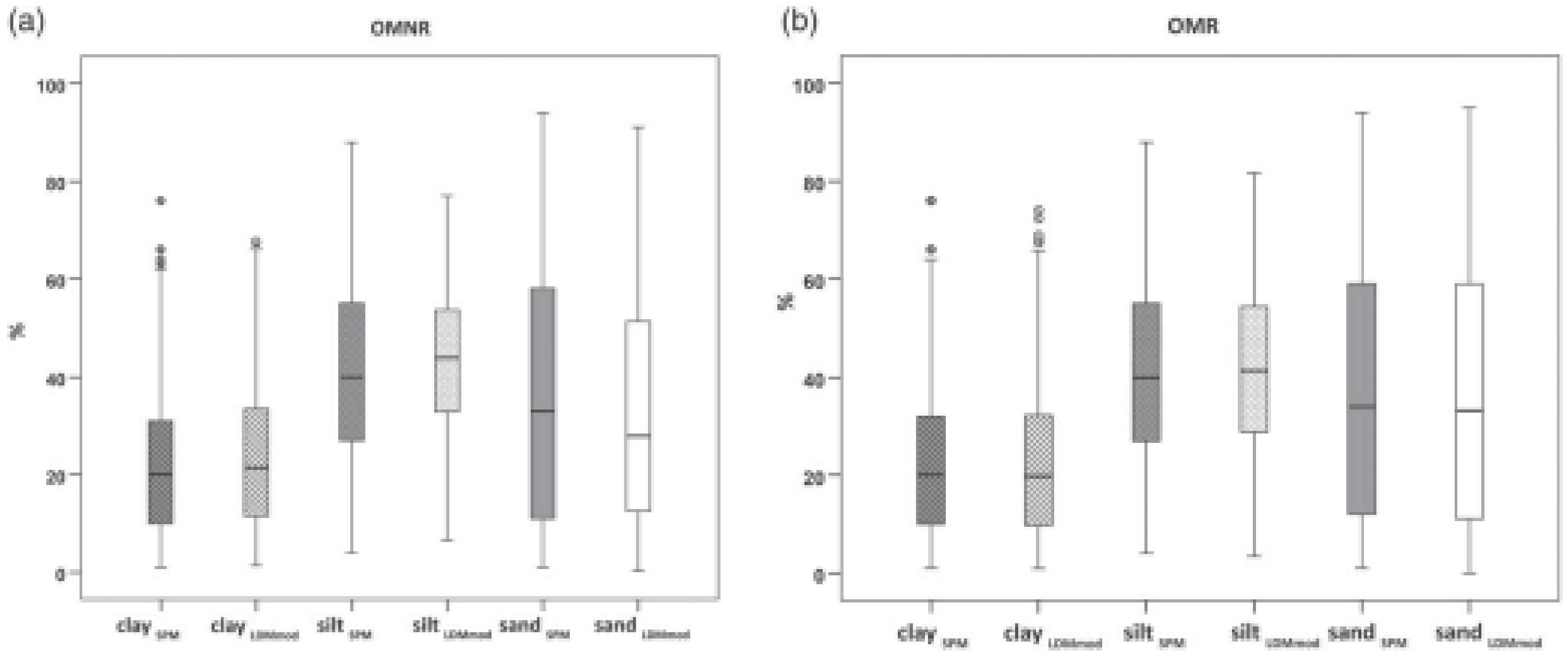

Figure 5 Comparison of the interquartile ranges of soil particle-size fractions measured by different methods (SPM, sieve–pipette; LDM, laser diffractometer) with modified particle-size distribution (PSD) boundaries for different pretreatments: (a) OMNR, organic matter not removed and (b) OMR, organic matter removed. The $clay_{LDMmod}$, $silt_{LDMmod}$ and $sand_{LDMmod}$ indicate the modified fraction boundaries.

Table 2 Recommended conversion pedotransfer functions (PTFs) for continental scale applications in Europe.

Input data options	Number of PTFs	Prediction models[a]
Based on samples without removal of organic matter during pretreatment (OMNR)		
PSD[b] only	2a	$clay_{SPMpred} = 0.69 + 0.92 \times clay_{LDMmod}$
		$silt_{SPMpred} = -6.10 + 1.10 \times silt_{LDMmod}$
PSD + chemical soil properties	6a	$clay_{SPMpred} = 187.40 - 0.055 \times CaCO_3 + 0.008 \times clay^2_{LDMmod} + 0.002 \times sand^2_{LDMmod} - 0.001 \times OC^2 - 47.33 \times \sqrt{pH\,(H_2O)} + 15.74 \times \sqrt{OC} + 1.82 \times \sqrt{CaCO_3} + 37.85 \times 1/clay_{LDMmod} - 359.48 \times 1/pH\,(H_2O) - 168.57 \times 1/OC - 0.012 \times 1/CaCO_3 + 41.59 \times \log_{10} clay_{LDMmod} - 90.26 \times \log_{10} OC - 4.70 \times \log_{10} CaCO_3$
		$silt_{SPMpred} = -170.55 + 0.036 \times CaCO_3 + 0.003 \times silt^2_{LDMmod} + 0.003 \times sand^2_{LDMmod} + 10.94 \times \sqrt{silt_{LDMmod}} + 54.02 \times \sqrt{pH\,(H_2O)} + 1.29 \times \sqrt{OC} - 0.62 \times \sqrt{CaCO_3} - 107.33 \times 1/sand_{LDMmod} + 421.51 \times 1/pH\,(H_2O) - 34.11 \times \log_{10} sand_{LDMmod} - 20.12 \times \log_{10} OC$
Based on samples undergoing pretreatment including removal of organic matter (OMR)		
PSD only	2b	$clay_{SPMpred} = 3.09 + 0.87 \times clay_{LDMmod}$
		$silt_{SPMpred} = 2.41 + 0.93 \times silt_{LDMmod}$
PSD + chemical soil properties	6b	$clay_{SPMpred} = -44.22 + 0.24 \times sand_{LDMmod} - 0.079 \times CaCO_3 + 0.001 \times OC^2 + 12.02 \times CaCO_3^2 - 2.48 \times \sqrt{clay_{LDMmod}} + 13.27 \times \sqrt{sand_{LDMmod}} - 0.81 \times \sqrt{pH\,(H_2O)} + 2.30 \times \sqrt{OC} - 22.95 \times 1/pH\,(H_2O) - 0.02 \times 1/OC - 13.79 \times 1/CaCO_3 + 3.73 \times \log_{10} clay_{LDMmod} - 7.05 \times \log_{10} OC$
		$silt_{SPMpred} = -26.76 - 0.34 \times sand_{LDMpred} - 0.076 \times CaCO_3 + 0.01 \times silt^2_{LDMmod} + 0.0001 \times CaCO_3^2 + 2.92 \times \sqrt{sand_{LDMmod}} - 3.98 \times \sqrt{OC} + 0.82 \times \sqrt{CaCO_3} + 42.77 \times 1/pH\,(H_2O) + 70.45 \times 1/OC + 13.48 \times \log_{10} silt_{LDMmod} - 2.87 \times \log_{10} sand_{LDMmod} + 29.83 \times \log_{10} OC$

[a] $clay_{SPMpred}$, sieve−pipette method (SPM) clay content (mass %); $silt_{SPMpred}$, SPM silt content (mass %), $clay_{LDMmod}$, modified laser diffractometer method (LDM) clay content (vol %); $silt_{LDMmod}$, modified LDM silt content (vol %); $sand_{LDMmod}$, modified LDM sand content (vol %); $CaCO_3$, calcium carbonate content ($g \cdot kg^{-1}$); OC, organic carbon content ($g \cdot kg^{-1}$); pH(H2O), soil pH measured in water−soil suspension (−).

[b] PSD, elements of particle size distribution, clay or silt content (mass%).

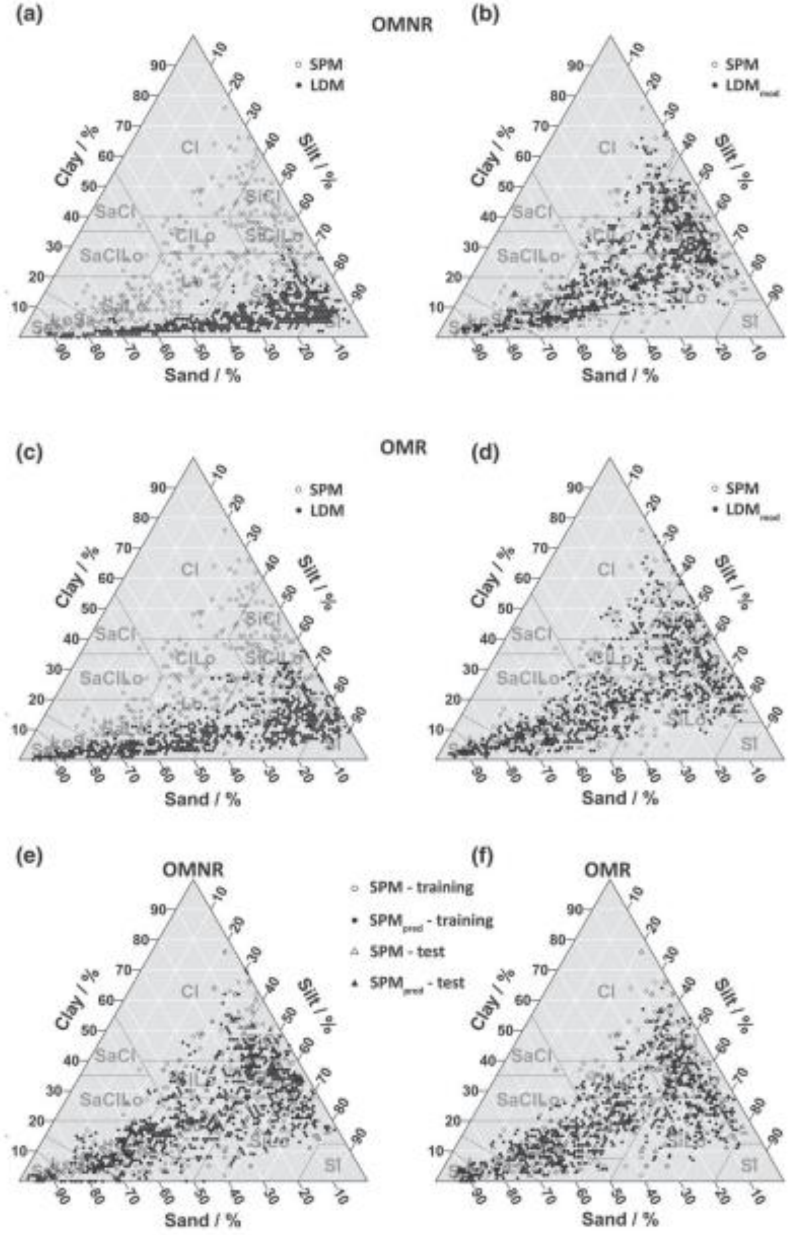

Figure 8 Distribution of soil textures in the USDA textural triangles: (a) measured by sieve–pipette (SPM) and laser diffractometer (LDM) methods for the OMNR (organic matter not removed) dataset, (b) measured by SPM and LDM with modified fraction boundaries for the OMNR dataset, (c) measured by SPM and LDM for the OMR (organic matter removed) dataset, (d) measured by SPM and LDM with modified fraction boundaries for the OMR dataset, (e) measured by SPM and converted LDM for pedotransfer functions (PTFs) 6 with the OMNR dataset and (f) measured by SPM and converted LDM for PTFs6 with the OMR dataset.

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Evaluation of soil texture determination using soil fraction data resulting from laser diffraction method**

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rial. The bulk samples were taken from boreholes with a depth of approximately 2 m, depending on the layering of the profiles. 155 soil samples were collected from the 53 soil profiles (Fig. 1).

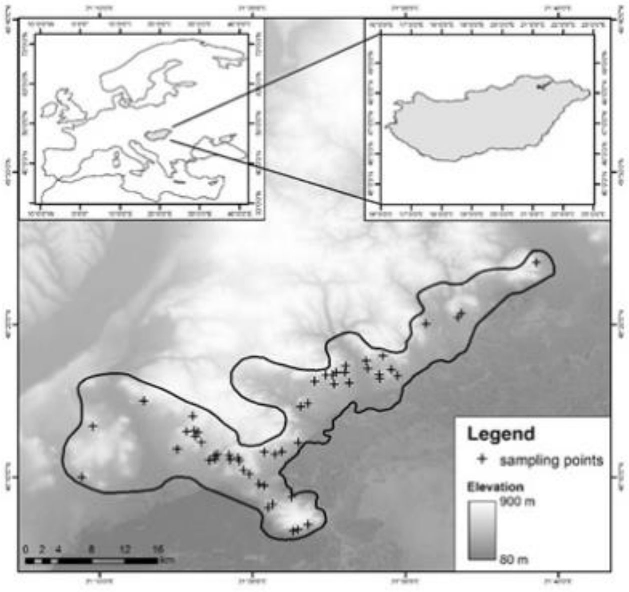


Fig. 1. Map of soil sampling points.

SPM-MSZ procedure: The PSD was determined by a combination of sieving and sedimentation, starting from a 25 g sample of air-dried soil. Particles smaller than 2000 μm and retained on a 250 μm aperture sieve (coarse sand fraction) were determined by a wet sieving procedure. Particles, which passed through the latter sieve were determined by sedimentation using the pipette method. For the calculation of sedimentation time it was assumed that the mean particle density is 2.65 Mg m^{-3}. The method does not require pre-treatments (organic matter, $CaCO_3$ or iron oxy-hydroxides removal) before the measurements take place. The chemical dispersion was performed using sodium pyrophosphate (55.8 g L^{-1}). The physical method used to facilitate the dispersion was shaking the pre-treated suspension for 6-10 h on the end-over-end shaker. By performing the PSD measurements according to the MSZ standard, the percentage (mass %) of the constituent fractions (SPM-MSZ_sand: 2000 μm to 50 μm; SPM-MSZ_silt: 50 to 2 μm; SPM-MSZ_clay: < 2 μm) could be calculated.

LDM procedure: For LDM analysis the Mastersizer 2000 (Malvern Company, UK) laser diffractometer was used (ISO 13320:2009). This instrument allows for the measurement of the PSD (volume, %) within the size range of 0.02-2000 μm (Malvern Operators Guide, 1999). The measurements were conducted using a Hydro 2000G dispersion unit. The PSD measurements using LDM were usually repeated twice. Third or fourth repetitions were measured, when the two previous repetitions were signifi-

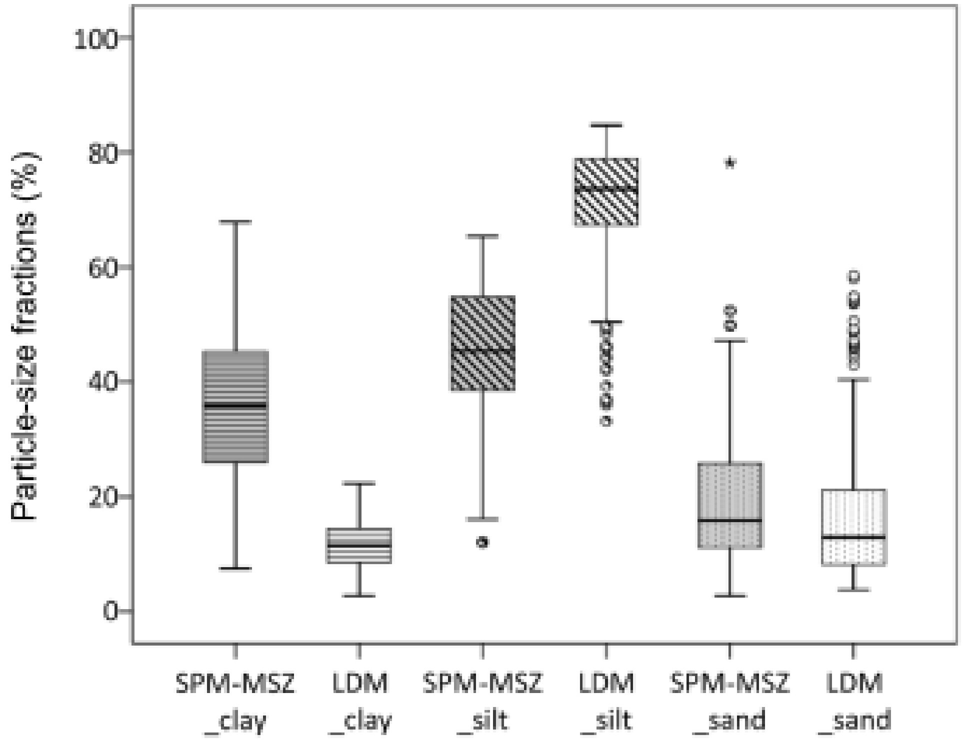


Fig. 2. Comparison of the interquartile ranges of soil particle-size fractions as measured by different PSD measurement methods. SPM-MSZ: PSD from a sieve-pipette according to the Hungarian standard; LDM: PSD from a laser diffractometer using the original fraction boundaries.

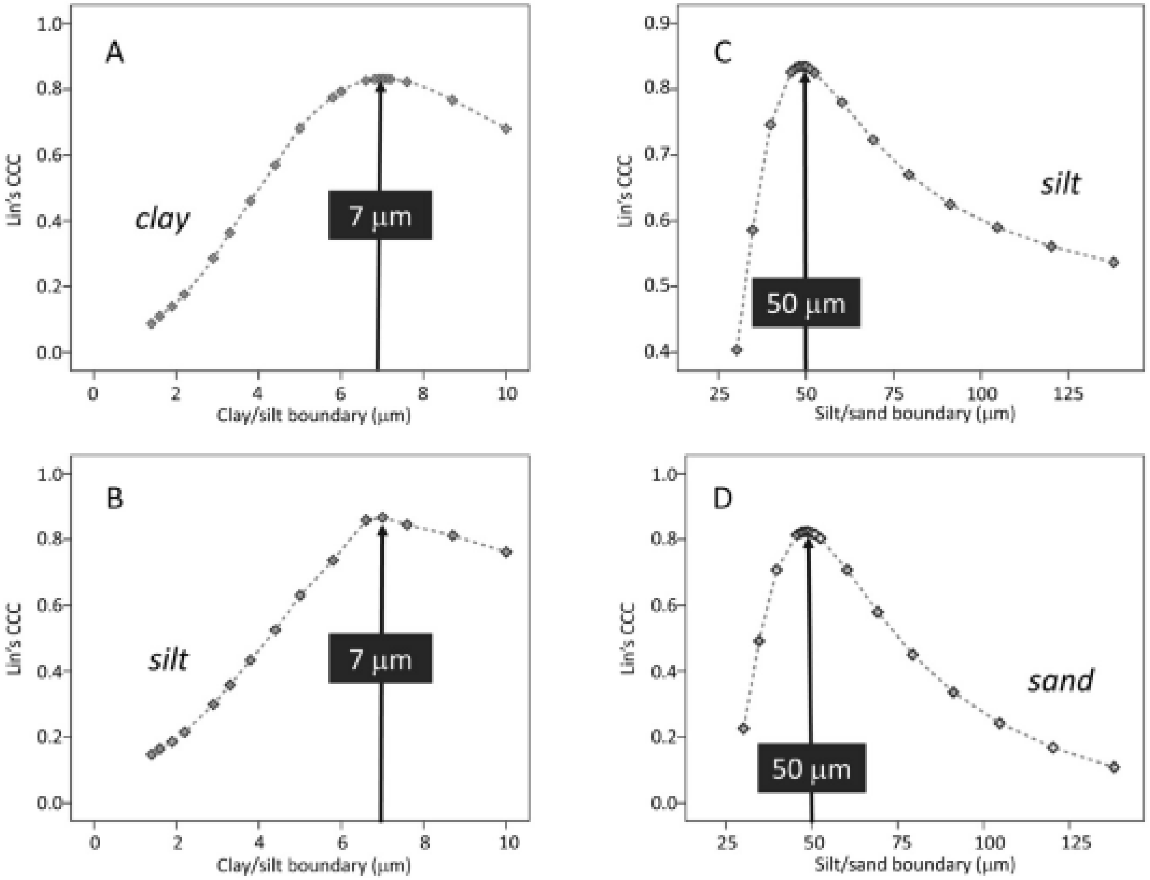
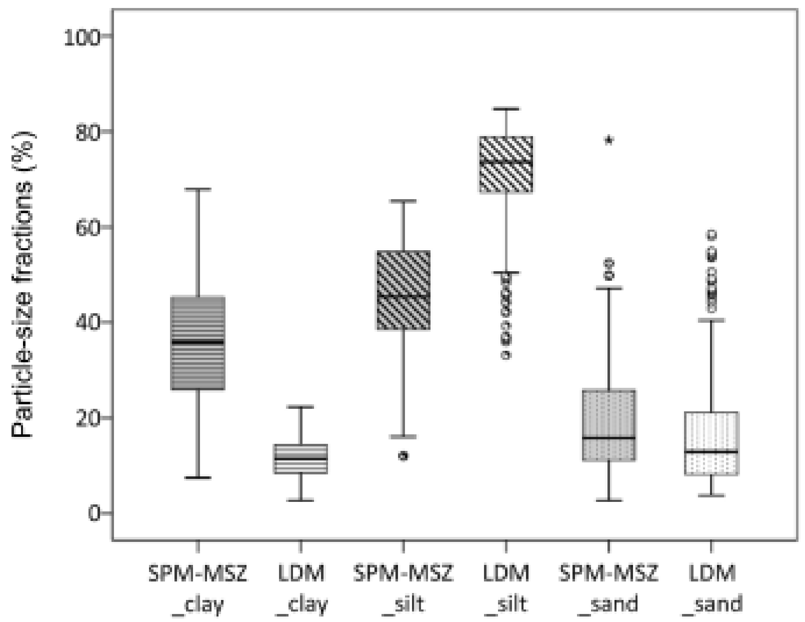


Fig. 3. Lin's concordance correlation coefficient (CCC) values of the LDM's and SPM's (A and B) clay-silt and (C and D) silt-sand fraction boundaries. The y-axis gives Lin's CCC of the (A and B) cumulative size classes of the LDM_clay (from <1.4 to <10.0 µm) and the SPM-MSZ_clay (<2 µm) fraction and (C and D) cumulative size classes of the LDM_sand (from >30.2 to >138.4 µm) and the SPM-MSZ_sand (>50 µm) fraction.



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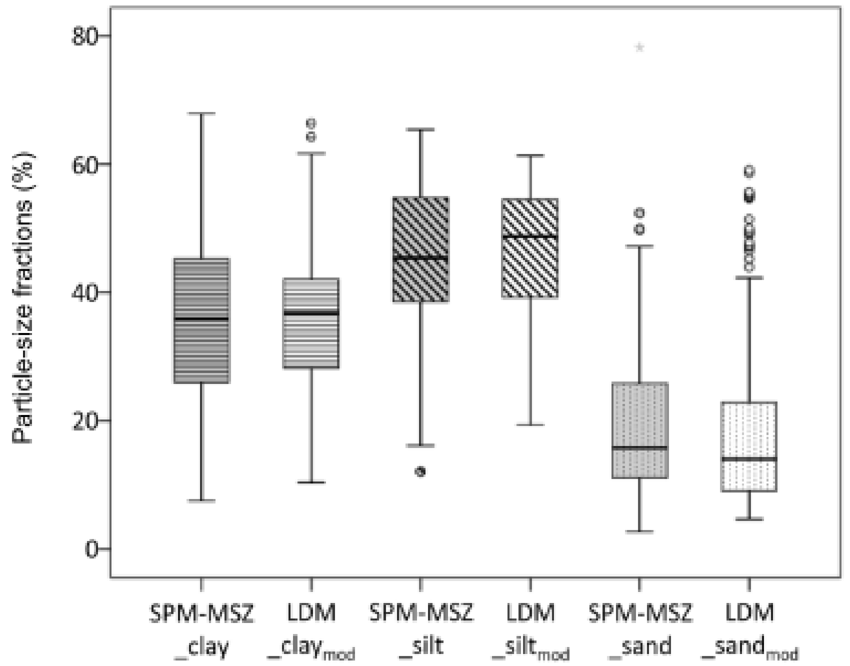

Fig. 4. Comparison of the interquartile ranges of soil particle-size fractions measured by different (SPM-MSZ, sieve-pipette according to Hungarian standard; LDM, laser diffractometer) methods with modified (optimal) PSD boundaries. The 'LDM_clay$_{mod}$', 'LDM_silt$_{mod}$' and 'LDM_sand$_{mod}$' indicate the modified fraction boundaries.

Table 2. Recommended conversion equations (pedotransfer functions)

Conversion model
$SPM\text{-}MSZ\_clay_{pred} = -22.24 + 0.62 \times LDM\_sand_{mod} + 11.89 \times \sqrt{LDM\_clay_{mod}} + 111.93 \times 1/pH(H_2O) - 7.77 \times 1/humus - 25.67 \times \log_{10}LDM\_sand_{mod} - 27.67 \times \log_{10}humus$
$SPM\text{-}MSZ\_silt_{pred} = -10.47 - 0.36 \times LDM\_sand_{mod} + 0.01 \times LDM\_silt_{mod}^2 + 0.18 \times pH(H_2O)^2 + 4.75 \times \sqrt{humus} + 19.92 \times \log_{10}LDM\_sand_{mod}$
$SPM\text{-}MSZ\_sand_{pred} = 100 - (SPM\text{-}MSZ\_clay_{pred} + SPM\text{-}MSZ\_silt_{pred})$

Table 3. Evaluating similarities in the fractions from LDM and SPM-MSZ based on the *RMSE* values

Clay fraction	*RMSE* (m %)	Silt fraction	*RMSE* (m %)	Sand fraction	*RMSE* (m %)
LDM_clay	25.0	LDM_silt	27.0	LDM_sand	5.5
LDM_clay_{mod}	6.5	LDM_silt_{mod}	5.7	LDM_sand_{mod}	5.3
$SPM\text{-}MSZ\_clay_{pred}$	5.5	$SPM\text{-}MSZ\_silt_{pred}$	4.8	$SPM\text{-}MSZ\_sand_{pred}$	4.8

Table 4. Evaluating similarities in the fractions from LDM and SPM-MSZ based on Lin's CCC values

Clay fraction	Lin's CCC	Silt fraction	Lin's CCC	Sand fraction	Lin's CCC
LDM_clay	0.14	LDM_silt	0.19	LDM_sand	0.90
LDM_clay_{mod}	0.83	LDM_silt_{mod}	0.85	LDM_sand_{mod}	0.91
$SPM\text{-}MSZ\_clay_{pred}$	0.89	$SPM\text{-}MSZ\_silt_{pred}$	0.90	$SPM\text{-}MSZ\_sand_{pred}$	0.92

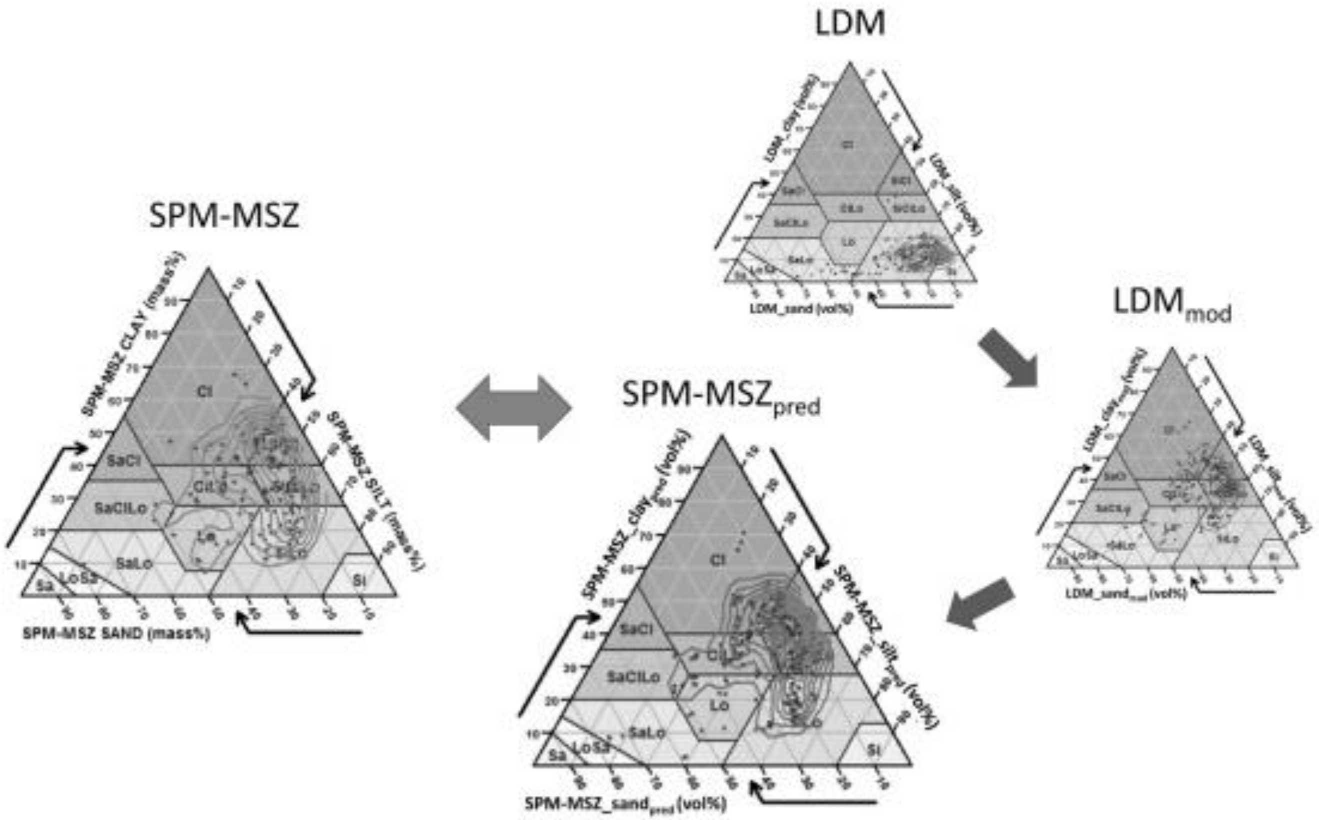

Fig. 6. Particle-size data of samples (N = 325; each sample represented by a black dot) plotted over the USDA soil texture triangle (Moeys, 2014) according to their % of clay, % of silt and % of sand particles. Abbreviations for the textural classes are: Cl – clay, ClLo – clay loam, L – loam, LoSa – loamy sand; Sa – sand; SaCl – sandy clay, SaClLo – sandy clay loam, SaLo – sandy loam; Si – silt, SiLo – silt loam, SiCl – silty clay, SiClLo – silty clay loam. Contour lines denote the points with the same occurrence frequency.