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PRELIMINARY EXPERIMENTS OF SOIL HYDROPHOBIC CHARACTER WITH KRÜSS DSA DROP SHAPE ANALYSER

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Abstract: In our experiments, we used Krüss DSA 100 drop shape analyzer to test the wettability of different Hungarian soil and sediment samples, which measures the contact angle (cA) and water drop penetration time (WDPT), excellent indicators of the wettability of the solid phase. Two sample preparation methods found in the literature were tested in this preliminary experiments. In the pastille method (PM), distilled water was dropped onto soil disc samples prepared at different pressures, cA and the WDPT were measured. In adhesive stripe method (ASM) only the cA was measured. We chose Sessile drop method with Young-Laplace fitting and automatic baseline adjustment. The cA and WDPT was also measured by the PM on a series of previously hydrophobized soil samples treated with CPC cationic surfactant. The two sample preparation methods mentioned above (PM and ASM) were used to determine the hydrophobicity order of the soil samples. In PM measurements, a verifiable difference in cA values was observed for pastilles produced at different pressures. For both methods, the hydrophobizing effect of the cationic surfactant was clearly detectable. The results confirmed that the hydrophobic character determined by cA measurement and the measured WDPT values are closely related.

Keywords: hydrophobic character, wettability, contact angle, water drop penetration time (WDPT)

1. Introduction

The ability of soils to hold and conduct water is one of the most important soil properties for both water management and environmental protection. These hydrophysical properties can be strongly influenced by the wettability (hydrophobic/hydrophilic character) of soils. Hydrophobic soil properties are defined as the phenomenon whereby soils "reject" water. The cross-linked humic molecules of organo-mineral complexes have both hydrophilic and hydrophobic parts. The development of hydrophobicity may be influenced by several factors, for example forest fires, the use of manure and organic fertilisers or industrial and municipal pollution. On well wettable surfaces, the water droplets are spread out widely to maximise the surface area in contact with the solid phase, whereas on hydrophobic surfaces the water droplets approach a spherical shape, as they contact the water-repellent surface over a smaller surface area (*Figure 1*).

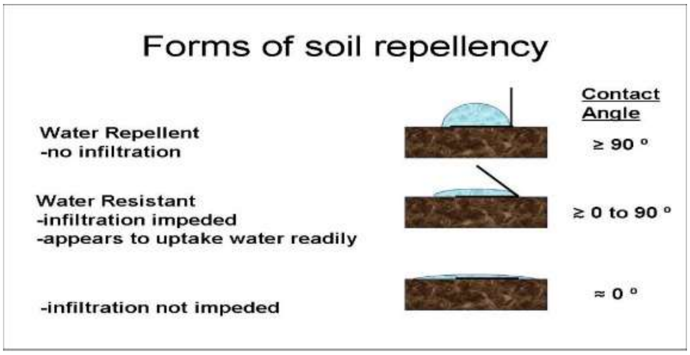

Figure 1: Form of soil repellency (Hallett, 2007)

The spreading of the liquid droplet on the surface of the solid phase provides a measure of the contact angle, which is an excellent characterisation of the wettability of the solid phase.

2. Materials and methods

During our preliminary methodological experiments, we used Krüss DSA 100 drop shape analyzer to test the wettability of crushed sedimentary rock samples with high clay mineral content („kaolin", „illite", „bentonite") and different Hungarian soil and sediment samples.

In a first step, we studied two types of sample preparation methods: „pastilles method" - PM (Bykova et al., 2019) and „adhesive stripe method" - ASM (Adamczuk et al., 2022), (*Figure 2-3*). For the PM method, the effect of the pressure applied during sample preparation was also tested.

Figure 2: Pastille method (PM), Gleyic Vertisol samples in different pressure

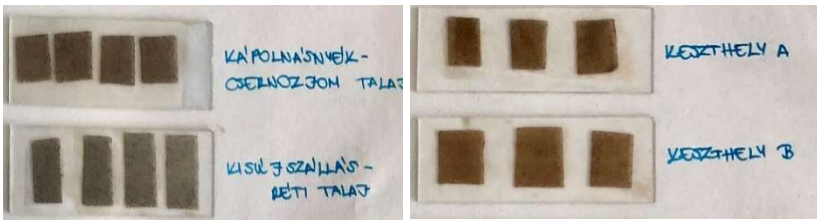

Figure 3: Adhesive stripe method (ASM), different Hungarian soil samples

In the second step, we investigated whether the methods used could detect the hydrophobic character of a verified hydrophobic sample series. We examined control and surfactant-treated soil samples, where a cationic surfactant, namely cetylpyridinium-chloride (CPC) was adsorbed on the surface of the soil samples in a monomolecular layer coating (Barna et al., 2015). For these samples we also measured the Water Drop Penetration Time (WDPT) values in the case of PM method. Calibration was performed before measuring each sample, measurements were performed in several (3-4x) replicates. The appropriate instrument settings (fixed frame rate, applied brightness) were tested on different soils. The frame rate setting can affect the accuracy of the measurement results, so it is important to adjust it to the soil type (a higher setting is required for hydrophilic soils). Last but not least, the effect of the instrument settings on the detection of hydrophobicity was also tested in soil samples from different tillage practices (ploughing, shallow cultivation, direct seeding).

The PM and ASM hydrophobicity sequence were different for the high clay mineral content samples. It is difficult to compare the results obtained with the two preparation methods because the contact angles measured with the PM depend to a large extent on the pressure forces applied during the pastille process (*Figure 4-5*).

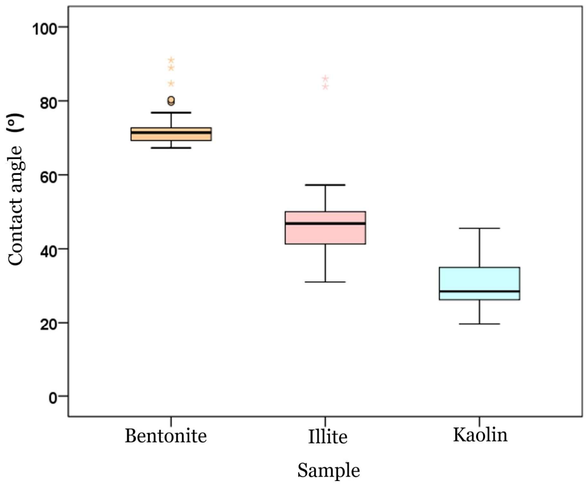

Figure 4:Differences betweeen contact angle data of crushed sedimentary rock samples with high clay mineral content in ASM method

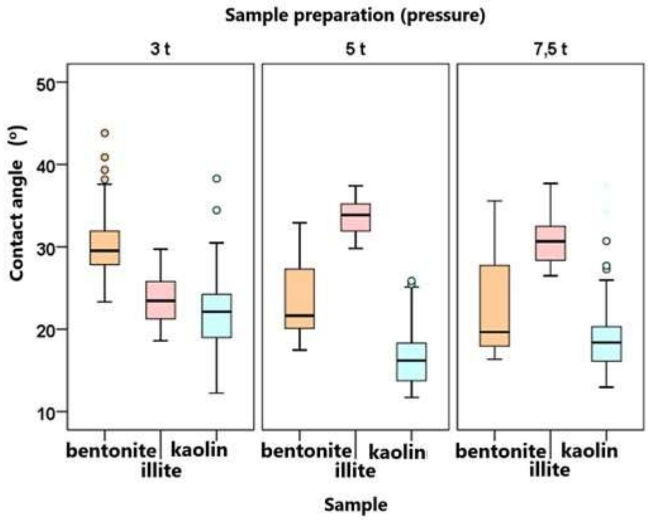

Figure 5: Contact angle of crushed sedimentary rock samples with high clay mineral content in PM method

The increase in hydrophobicity of the samples was well detected in case of surfactant-treated soils (*Figure 6-7*), WDPT values proved this excellently (*Figure 8*).

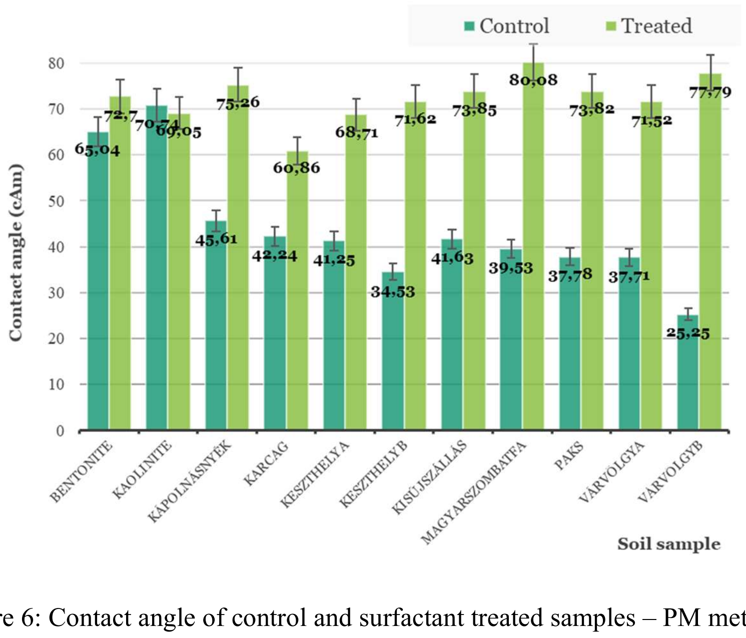

Figure 6: Contact angle of control and surfactant treated samples – PM method

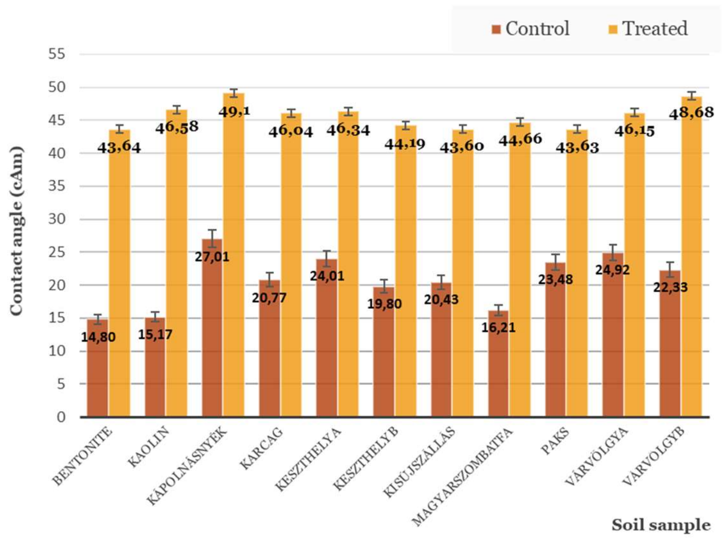

Figure 7: Contact angle of control and surfactant treated samples – ASM method

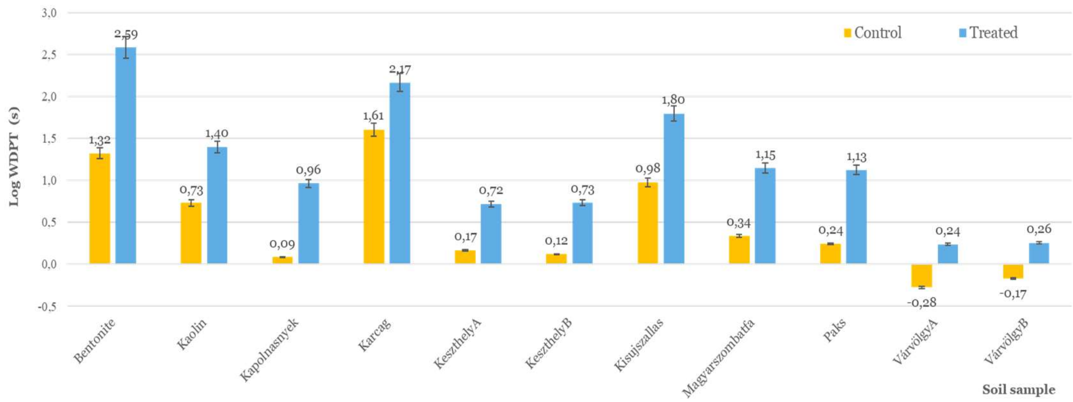

Figure 8. WDPT of control and surfactant treated samples - PM method

In comparing different agro-techniques and measuring different soil samples, we fine-tuned the frame rate setting, which is most reliable at 320 (*Figure 9-10*), as the smallest standard deviations and most distinct treatment results were observed at 160 and 320 fixed frame rates (marked with red ticks).

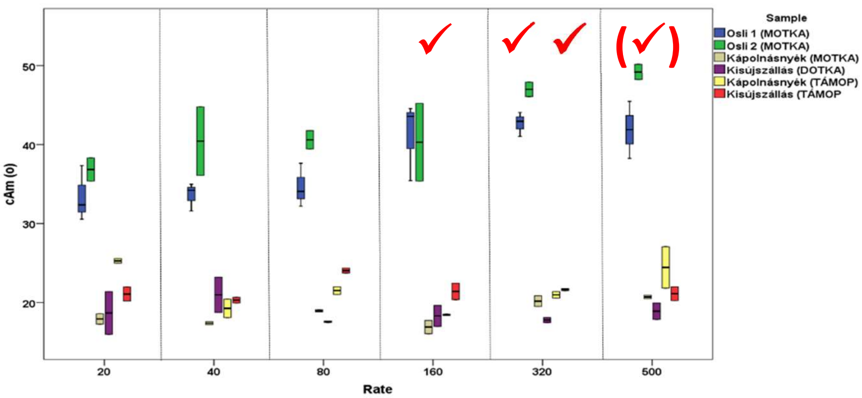

Figure 9: In the measurement the used fixed frame rate was refined on selected soil samples

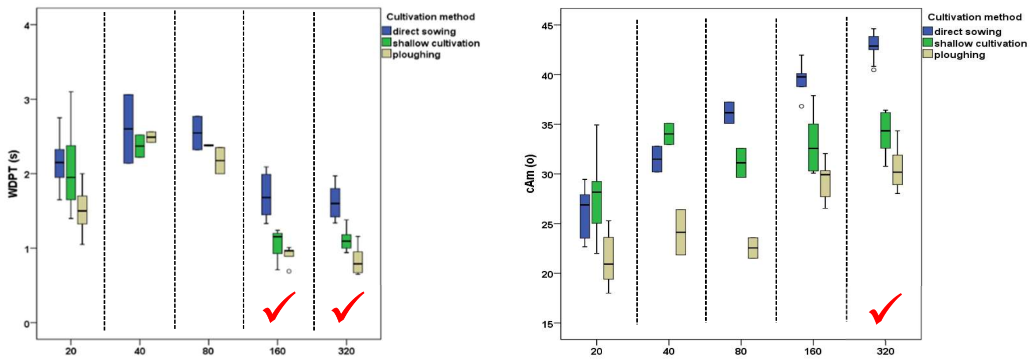

Figure 10. Contact angle and WDPT results with the three different agro-techniques (Hatvan, Józsefmajor, 20-year long-term experiment) measured by different fixed frame rate values (20-320)

4. Discussion

Preliminary tests have confirmed that the appropriate preparation pressure during the PM method was 5 tonnes.

In the study of cationic surfactant-treated samples, the results clearly demonstrated the increasing effect of surfactant treatment on contact angle and WDPT value, the hydrophobicity of the treated samples increased due to the effect of CPC.

The hydrophobicizing effects of the cultivation method were in line with those described in the literature (direct seeding>deep tillage>ploughing).

In the methodological preliminary experiments, the settings of fixed frame rate values have been shown to have an effect on the measured contact angle values.

The use of different agro-techniques was also shown the effect on the hydrophobicity of the samples.

The Krüss DSA 100 provides a simple method for characterising the wetting properties of the solid phase, and it is therefore important to find the applicability of the developed measurement methodology to the soil.

The results of our preliminary experiments on the methodology are promising and it is expected that the indicators of soil wetting properties will be useful in soil physics, soil chemistry and soil biology.

Sample	Clay (%)	Silt (%)	Sand (%)	Humus (%)	$CaCO_3$ (%)	pH (H_2O)
Kápolnásnyék - Vermic Calcic Chernozem (Anthric Siltic) - A horizon	21.07	30.16	48.77	3.70	9.52	7.83
Karcag - Vertic Stagnic Solonetz (clayic) - A horizon	51.09	45.9	0.88	2.00	0.13	6.92
Keszthely - Hortic Terric Cambisol (Dystric Siltic) - A horizon	17.12	17.09	65.79	1.55	0.05	7.04
Keszthely - Hortic Terric Cambisol (Dystric Siltic) - B horizon	22.65	16.14	61.21	0.94	0.00	6.83
Kisújszállás - Gleyic Vertisol (Calcic)	55.56	31.28	13.16	2.76	1.10	7.51
Magyarszombatfa - Vertic Gleyic Luvisol (Mangani-ferric Siltic)	38.96	25.93	34.61	0.49	0.00	5.74
Paks - Loess	16.08	46.00	9.25	0.63	28.04	8.17
Várvölgy - Cutanic Luvisol (Siltic) - A Horizon	15.27	29.35	54.05	1.33	0.00	6.59
Várvölgy - Cutanic Luvisol (Siltic) - B Horizon	22.25	26.56	50.49	0.70	0.00	6.64
Bentonite	67.67	31.71	0.63	0.00	0.70	6.95
Kaolinite	54.53	44.73	0.73	0.00	1.10	8.69

Table 1: Some characteristic features of the tested samples

Acknowledgements

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