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Study on hydrophysical and chemical parameters of Polish and Slovak soils modified with zeolitic additives

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Application of soil modifiers is a widely used practice in agriculture to improve soil quality in support of good agricultural practices. This paper presents an assessment of the effect of zeolite modifiers combined with four soils of Polish and Slovakian origin on their hydrophysical properties. Under laboratory conditions, the characterization of the applied soil additives was carried out along with particle soil analysis. In addition, the prepared soil mixtures were examined using contact angle and saturated hydraulic conductivity. The results suggest that the increase in hydrophobicity occurred mainly after the application of fly ash, and the values for Polish Silty clay soil and Slovak soils: Sandy soil, Silt loam soil, and Silty clay soil compared to the control changed from 73.02° to 96.22°; 8.67° to 24.88°; 29.23° to 33.94° and 19.9° to 26.16° respectively. The increase in hydrophobicity could be explained by the higher CaO content of the high-carbon fly ash compared to the other materials. The parameters of saturated hydraulic conductivity were reduced after the application of all zeolite materials in the case of Polish Silty clay and Slovak Sandy soils. By filling the space between the soil particles, the porous soil additives decreased the hydraulic conductivity. In Slovak soils such as Silty loam soil and Silty clay soil, the opposite effect was observed. The soil additives increased the number and size of pores, which resulted in faster filtration of water and thus an increase in the saturated hydraulic conductivity value.

KEY WORDS: saturated hydraulic conductivity, zeolite modifiers, waste product, fly ash, hydrophobicity

Introduction

Soil is a fundamental element of the environment, being a three-phase system made up of a solid phase, a liquid phase and a gaseous phase. The particles that belong to the solid phase account for about half of the soil volume, the rest being the pores in between them. The geometry, size and connections of the soil particles in its structure create a porous system with unique hydraulic properties. These properties are very important for proper crop production. In agriculture, many well-known soil additives are used to soil improvement (Rousseva et al., 2017). There are zeolites among them, both natural and synthetic ones. They are crystalline, porous aluminosilicates most often of sodium and calcium (Inglezakis and Zorpas, 2012), which can be applied as a component of complex fertilizers. According to Soudejani et al. (2019), the addition of zeolites to the composting of organic solid waste allows the production of a material that leads to higher water retention in the soil, reduces nutrient loss, and improves crop quality. A characteristic property of zeolites is their ability to retain water and nutrients. They are also able to remove water from the tubules (so-called "zeolite water") without destroying their structure (Ghorbani et al., 2022). In this way, zeolites improve infiltration rates and saturated hydraulic conductivity. In addition, they affect cation exchange capacity and prevent water loss from deep percolation (Talebnezha and Sepaskhah, 2013; Chmielewska, 2014; Mondal et al., 2021). Belviso et al. (2022) confirmed that the obtained effects depend on the dose of applied zeolite material. With the increase in the amount of zeolite added to the soil, the range of available water for plants decreased, making the soil less suitable for plant growth (Belviso et al., 2022). Another substance that can be able to absorb a large amount of water (up to 80%) and reduce infiltration (2–16%) is fly ash. The hydraulic properties of this material depend on the density and depth of the studied layer and also the specific chemical composition of fly ash. Specific chemical composition of fly ash is responsible for their different hydraulic properties. Calcium fly ashes (Class C fly ashes according to ASTM C618, 2015) are characterized by significantly lower hydraulic conductivity compared to silicon fly ashes (Class F fly ashes according to ASTM C618). However, mixtures of fly ashes of class F with lime and calcium bentonite may contain some amount of CaO, which significantly affects

the hydraulic properties of the material (Zabielska-Adamska, 2020; Porbaha et al., 2000).
Most of the available scientific papers are focused on natural zeolites. Therefore, in this research, synthetic zeolitic materials, which are produced from energy waste (fly ash), were proposed as soil additives. The main aim of the experiments was to determine hydrophysical and chemical properties of morphologically different soils collected in Poland and Slovakia modified with three types of zeolitic materials.

Material and methods

Experimental design

For the experiment, four soils with different land use were selected from regions in Poland and Slovakia. One soil from Poland, Polish Silty clay soil, was collected from Lubelskie Voivodeship area (Jabłonna, eastern part of Poland, 51°05'N, 22°36'E, long-term abandoned field). Three soils were taken from regions of Slovakia: first, Slovak Sandy soil, was taken from the site of Záhorská nížina (Plavecký Štvrtok, western part of Slovakia, 48°37'N, 16°99'E, long-term abandoned field), the second soil, Slovak Silt loam soil, was collected from the Nitrianská pahorkatina site (Dolná Malanta, central western part of Slovakia, 48°31'N, 18°14'E, agriculturally used country) and the third, Slovak Silty clay soil, was taken from the Východoslovenská nížina site (Senné, eastern part of Slovakia, 48°66'N, 22°02'E, grassland).
Disturbed samples were collected from the top 20 cm of the soil profile at all areas. The natural dry bulk density of all soil types was determined based on undisturbed samples. Disturbed samples were air dried and sieved to a fraction with a particle size ≤ 2 mm in laboratory conditions. The bulk density of all pure soil types (Control) were prepared to be the same as natural bulk density (Table 1). Mixtures of soil and zeolitic materials were prepared by mixing one type of soil with one selected material at 1% dose (per weight) in laboratory conditions. Each treatment (soil and zeolite mixture, also Control) was prepared in three replicates in steel cylinders with a volume of 100 cm^3. Soil mixtures with zeolite materials were compared with control samples, i.e., soils without additives.

Soil characterization

The main properties of the soils were investigated by conventional methods i.e. the pH values were measured electrochemically in H_2O using a digital pH-meter (Radiometer Copenhagen). The ash content (A) was calculated by weighing the residue after 5 h of combustion at 550°C in a muffle furnace (FCF 12 SP, Czylok). The total variable surface charge (Q_{tot}) was established by potentiometric titration with Titrino 702 SM (Metrohm) (Józefaciuk, 2002).

Soil textural analysis

The hydrometer method was used for soils particle size distribution analysis. The principle of this method is based on Stokes' equation (Eq. 1):

$$v = \frac{(\rho_S - \rho_W) g d^2}{18\eta} \qquad (1)$$

where
v – velocity of suspended particles sedimentation [m s^{-1}];
ρ_S – soil particle density [kg m^{-3}];
ρ_W – liquid water density [kg m^{-3}];
g – acceleration of gravity [m s^{-2}];
d – soil particle diameter [m];
η – dynamic viscosity of liquid water [kg m^{-1} s^{-1}].

The measurement of decreasing densities of suspension

Table 1. Natural and prepared dry bulk density of all studied treatments and standard deviations

Treatment	Natural dry bulk density [g cm^{-3}]	Prepared dry bulk density [g cm^{-3}]
Polish Silty Clay	1.06	1.06±3x10^{-3}
Polish Silty Clay + NaP1(C)		1.04±5x10^{-3}
Polish Silty Clay + Na-P1		1.05±6x10^{-4}
Polish Silty Clay + HiC FA		1.04±4x10^{-3}
Slovak Sandy	1.64	1.64±1x10^{-3}
Slovak Sandy + NaP1(C)		1.62±3x10^{-3}
Slovak Sandy + Na-P1		1.62±3x10^{-3}
Slovak Sandy + HiC FA		1.62±1x10^{-3}
Slovak Silt Loam	1.31	1.31±1x10^{-4}
Slovak Silt Loam + NaP1(C)		1.29±1x10^{-3}
Slovak Silt Loam + Na-P1		1.29±4x10^{-3}
Slovak Silt Loam + HiC FA		1.28±7x10^{-5}
Slovak Silty Clay	1.02	1.02±3x10^{-3}
Slovak Silty Clay + NaP1(C)		1.01±5x10^{-3}
Slovak Silty Clay + Na-P1		1.01±5x10^{-3}
Slovak Silty Clay + HiC FA		1.01±3x10^{-3}

as a function of time, due to soil-particle sedimentation. This method was described in detail by Klute (1986). Based on textural fractions of sand, silt and clay were all soils classified according to USDA classification.

Zeolite characterization

For the study, three materials provided by the Lublin University of Technology were used in the experiments. One of them was high-carbon fly ash (HiC FA), a waste material produced as a result of conventional coal combustion. This product was applied as a substrate for the synthesis of the subsequent materials, i.e., Na-P1(C) carbon-zeolite composite and pure Na-P1 zeolite with no ash residue in its structure. The exact procedure for synthesizing the materials was described by Panek et. al. (2021).

The morphology of the high-carbon fly ash (HiC FA), zeolite (Na-P1) and carbon-zeolite composite (NaP1(C)) was analyzed by Scanning Electron Microscopy (Phenom ProX, Thermo Fisher Instruments, Somerset, NJ, USA). The BSD detector was used, and the accelerating voltage was 10 kV. To achieve high quality images, all samples were coated with a layer of gold using a sputter coater (CCU-010 LV, Safematic GmbH, Zizers, Switzerland).

Textural parameters of zeolitic materials were determined by nitrogen adsorption/desorption method (ASAP 2420 analyzer, Micromeritics Inc.). Specific surface area (S_{BET}), total pore volume (V_t), micro pore volume (V_{mic}), and average pore diameter (D) were estimated. Specific surface area was calculated using the BET equation (Brunauer et al., 1938), (Eq. 2):

$$\frac{1}{x\left[\left(\frac{p_0}{p}\right)-1\right]} = \frac{1}{x_m C} + \frac{C-1}{x_m C}\left(\frac{p}{p_0}\right) \tag{2}$$

where

- x – the weight of nitrogen adsorbed at a given relative pressure (p/p 0);
- x_m – the monolayer capacity, which is the volume of gas adsorbed at standard temperature and pressure (STP);
- C –the constant.

The elemental composition of the samples was determined by semi-quantitative energy-dispersive X-ray fluorescence on an Epsilon 3 spectrometer (Panalytical, Eindhoven, the Netherlands). The equipment consisted of a Rh 9 W, 50 kV, 1 mA X-ray lamp, a 4096-channel spectrum analyzer and a high-resolution solid-state SDD detector cooled by a Peltier cell. The results obtained included LOI.

Analysis of contact angle

Measurements of the contact angle of Polish and Slovak soils with and without amendments were performed using goniometer Drop Shape Analysis System (DSA100, Krüss, Germany). The sessile water drop method was used to determine differences in the wettability of the samples. Following the methodology, a drop of water was applied to the surface of the sample using a goniometer syringe, and afterward, the contact angle of the water drop was measured. When $\alpha \geq 90°$, the liquid doesn't wet the studied material and it is considered as hydrophobic.

Analysis of saturated hydraulic conductivity

Saturated hydraulic conductivity (K) was determined by pure soils and prepared mixtures of each soil type and zeolitic materials in steel cylinders. We used a modified falling–head (variable) method to measure the amount of water that goes through the soil/mixture sample in a fixed time interval (modified by Novák and Hlaváčiková, 2019). The water level above the soil/mixture sample decreases over time (Fig. 1). The same method of measurement was used for all soil types to avoid errors with different methodological processes.

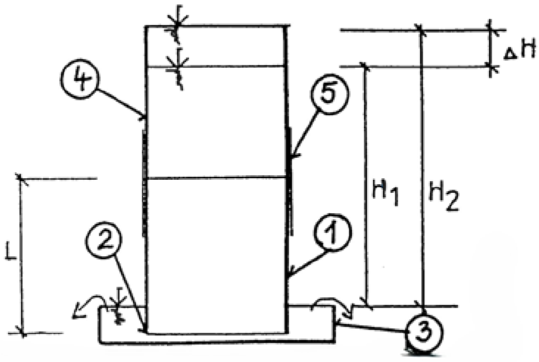

Fig. 1. Measurement of saturated hydraulic conductivity through modified falling head method (modified by Novák and Hlaváčiková 2019). 1=saturated soil/mixture core; 2=cloth sealing; 3=Petri dish; 4=connected steel cylinder; 5=rubber sealing.

A digital calliper (Milomex Ltd., Bedfordshire, UK) was used to measure the water level drop. K values were calculated according to Eq. 3:

$$K = \frac{L}{t} \cdot \ln\frac{H_2}{H_1} \tag{3}$$

where:

- L – soil cylinder height (soil sample) [cm];
- t – time of water level decrease from height H_2 to H_1 [s];
- H_2 – initial water level in the extension [cm];
- H_1 – the height of the water in the extension after the drop [cm].

All values of K measurement were calculated for cm per day unit, for better visualization in all treatments.

Results and discussion

The results of untreated soils' hydrophysical and chemical properties are summarized in Table 2 and they revealed differences of the soils. Polish Silty clay, Slovak Silty clay, and Slovak Silt loam soils belonged to neutral, silty soils with low sand content (Polish Silty clay – 6%, Slovak Silty clay – 11%, and Slovak Silt loam – 15%) whereas Slovak Sandy soil was slightly acidic soil with high sand content (91%). The Slovak Silty clay soil had the highest pH parameter (7.4), specific surface area (44.7 m^2 g^{-1}) and total variable surface charge value (16.6 cmol kg^{-1}) compared to the other tested soils. On the other hand, this soil had the lowest ash content (87.9%). Sandy soil, due to the highest sand content in its structure, had the lowest value of contact angle (8.7°). Nevertheless, all types of soil demonstrated a contact angle below 90°, which indicated their hydrophilic nature.

Textural parameters of zeolitic materials are presented in Table 3. Based on the results, it can be concluded that the S_{BET} surface of the selected materials is poorly developed. Na-P1 had the smallest S_{BET} parameter (69.85 m^2 g^{-1}), while it contained larger pores (D = 6.93 nm) than HiC FA (D = 5.66 nm). Accordingly, the zeolitization process strongly affected the textural properties by increasing the average pore diameter, as well as reducing their total pore volume.

Representative SEM micrographs of the applied soil conditioners are shown in Fig. 2. The analysis of the fly ash sample (Fig. 2A) indicates the presence of irregular forms of quartz, spherical structures of aluminosilicate glaze, and also unburned carbon fragments. Carbon sinters with embedded beads of aluminosilicate glaze are also visible. Pure zeolite Na-P1 (Fig. 2B) has no residual fly ash in its structure. It forms spherical, highly regular zeolite crystals. Na-P1(C), presented in Fig. 2C, creates very well-formed crystals, forming single clusters or

Table 2. Soil texture and chemical properties of the soils used in this study (*A* – ash content, *S_{BET}* – specific surface area, *Q_{tot}* – total variable surface charge, CA – contact angle)

Soil	Size fraction [%]			*A*	*pH*	*S_{BET}*	*Q_{tot}*	CA
	Sand	Silt	Clay	[%]	H_2O	[m^2 g^{-1}]	[cmol kg^{-1}]	[°]
Polish Silty clay soil	6	54	40	95.6	6.6	23.3	15.3	73.0
Slovak Sandy soil	91	8	2	99.7	6.2	2.2	7.1	8.7
Slovak Silty clay soil	11	43	46	87.9	7.4	44.7	16.6	19.9
Slovak Silt loam soil	15	60	25	94.4	7.1	21.1	15.7	29.2

Table 3. Textural parameters and chemical composition of fly ash (HiC FA), synthetic zeolite (Na-P1) and zeolite composite (Na-P1(C)). (*S_{BET}* – specific surface area, *S_{micro}*– micropore area, *V_t* – total pore volume, *V_{micro}* – micropore volume, and *D* – average pore diameter)

Conditioners	*S_{BET}* [m^2 g^{-1}]	*S_{micro}* [m^2 g^{-1}]	*V_t* [cm^3 g^{-1}]	*V_{micro}* [cm^3 g^{-1}]	*D (4 V/A)* [nm]	CaO [%]
HiC FA	46.19	10.72	0.064	0.0060	5.66	3.67
Na-P1	26.71	4.30	0.05	0.0017	6.93	0.31
Na-P1(C)	69.85	16.9	0.12	0.0070	6.75	2.17

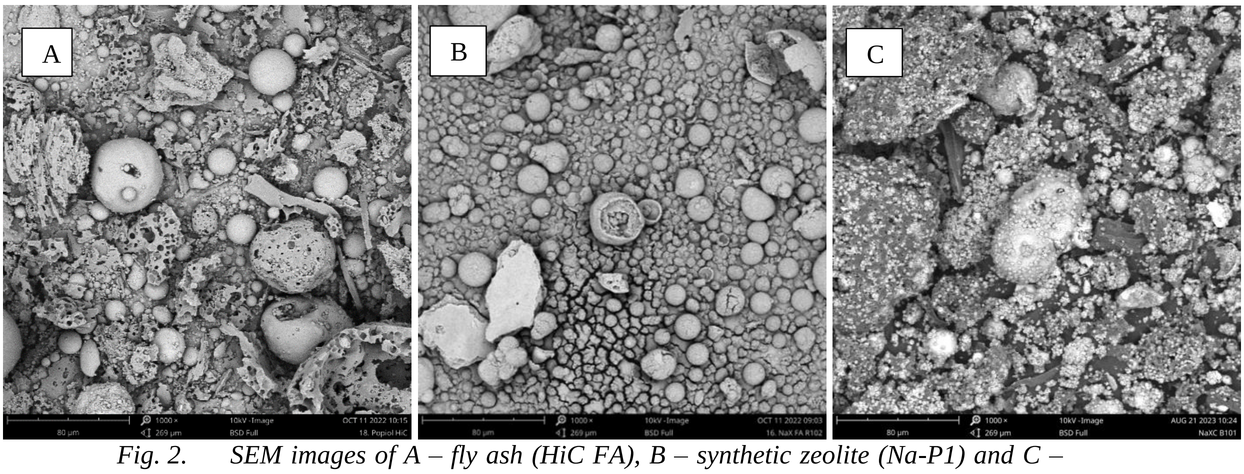

Fig. 2. SEM images of A – fly ash (HiC FA), B – synthetic zeolite (Na-P1) and C – composite zeolite (Na-P1(C)) respectively.

overgrown aggregates. Fragments of aluminosilicate glaze and the fly ash zone are the bases on which these crystals are formed.

Contact angle

Water contact angle measurement can directly show the hydrophilic or hydrophobic properties of the surfaces being tested. Images of water droplets on the surfaces of Polish Silty Clay soil and Slovak soil's, e.g. Sandy soil, Silt loam soil and Silty clay soil with and without the additive of zeolite materials such as Na-P1(C), Na-P1 and HiC FA are shown in Fig. 3.
The individual values of the contact angle measurements for all tested soils (controls and mixtures) are presented in Table 4.
The obtained results suggested that the addition of 1% fly ash increased the contact angle of all studied soils compared to the control. The largest rise was observed for Polish Silty clay soil. The contact angle increased from 73.02º for control to 96.22º after the application of fly ash. A significant increase was also noted for Slovak Sandy soil – the contact angle of the control equalled 8.67º, in contrast to 24.88º for the modified soil sample with fly ash. Smaller increases were observed for Slovak Silt loam soil and Slovak Silty clay soil. Contact angle values raised in comparison with the control from 29.23º to 33.94º and from 19.9º to 26.16º, respectively. Some of the reason for the increased contact angle may be connected with the chemical composition and structure of the fly ash. The one investigated in this study contained 3.67% CaO (Panek et al., 2021), according to the literature, which could significantly affect the permeability of soil. Fly ash grains with higher CaO content are covered with crystallized needle-shaped protuberances, which may have an impact on increasing the hydrophobicity of the studied soil (Zabielska-Adamska, 2020).
The effect of materials synthesized from HiC FA varied depending on the type of studied soil. The value of the contact angle of two Slovak soils: Sandy soil and Silt loam soil decreased after the addition of carbon composite or pure synthetic zeolite. The opposite situation was observed for two Silty clay soils – Polish and Slovak. The carbon-zeolite composite Na-P1(C) turned out to have the highest impact on the increase in contact angle. Both of these soils have a very similar particle size distribution, but the difference is that Slovak

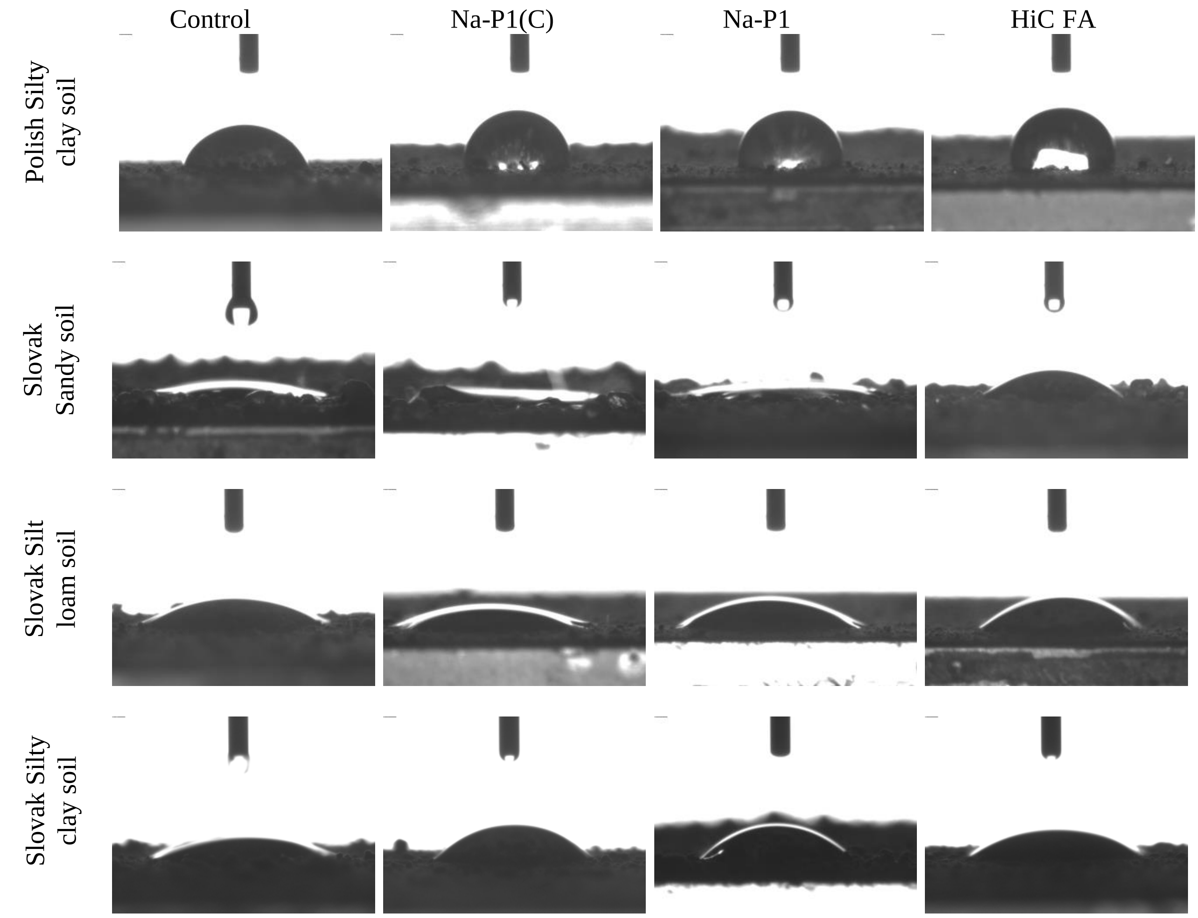

Fig. 3. Representative images of contact angle measurements for Polish Silty clay soil and Slovak soils: Sandy soil, Silt loam soil and Silty clay soil. Images shows control samples with soil without additives and soil mixtures with zeolite materials Na-P1(C), Na-P1 and HiC FA.

Silty clay soil naturally has a much lower ash content compared to Polish Silty clay soil.

Saturated hydraulic conductivity

Saturated hydraulic conductivity (*K*) varied with the addition of zeolitic materials (Fig. 4). The dependence of *K* on soil texture has been well documented and values of *K* depend on the size distribution of soil particles, i.e. soil texture, and the spatial distribution of these particles, i.e. soil structure. Polish Silty clay soil, Slovak Silty loam, and Silty clay soils exhibited low value of the *K*. It was correlated with silt and clay content. These types of particles are small and have small pore

Table 4. Results of the contact angle for the studied mixtures of soils with zeolite materials

Sample type	[°]	Type of surface
Polish Silty clay soil, control	73.02	HYDROPHIL
Polish Silty clay soil + Na-P1(C)	92.95	HYDROPHOBIC
Polish Silty clay soil + Na-P1	92.66	HYDROPHOBIC
Polish Silty clay soil + HiC FA	96.22	HYDROPHOBIC
Slovak Sandy soil, control	8.67	HYDROPHIL
Slovak Sandy soil + Na-P1(C)	0.00	HYDROPHIL
Slovak Sandy soil + Na-P1	0.00	HYDROPHIL
Slovak Sandy soil + HiC FA	24.88	HYDROPHIL
Slovak Silt loam soil, control	29.23	HYDROPHIL
Slovak Silt loam soil + Na-P1(C)	25.02	HYDROPHIL
Slovak Silt loam soil + Na-P1	27	HYDROPHIL
Slovak Silt loam soil + HiC FA	33.94	HYDROPHIL
Slovak Silty clay soil, control	19.9	HYDROPHIL
Slovak Silty clay soil + Na-P1(C)	39.72	HYDROPHIL
Slovak Silty clay soil + Na-P1	35.68	HYDROPHIL
Slovak Silty clay soil + HiC FA	26.16	HYDROPHIL

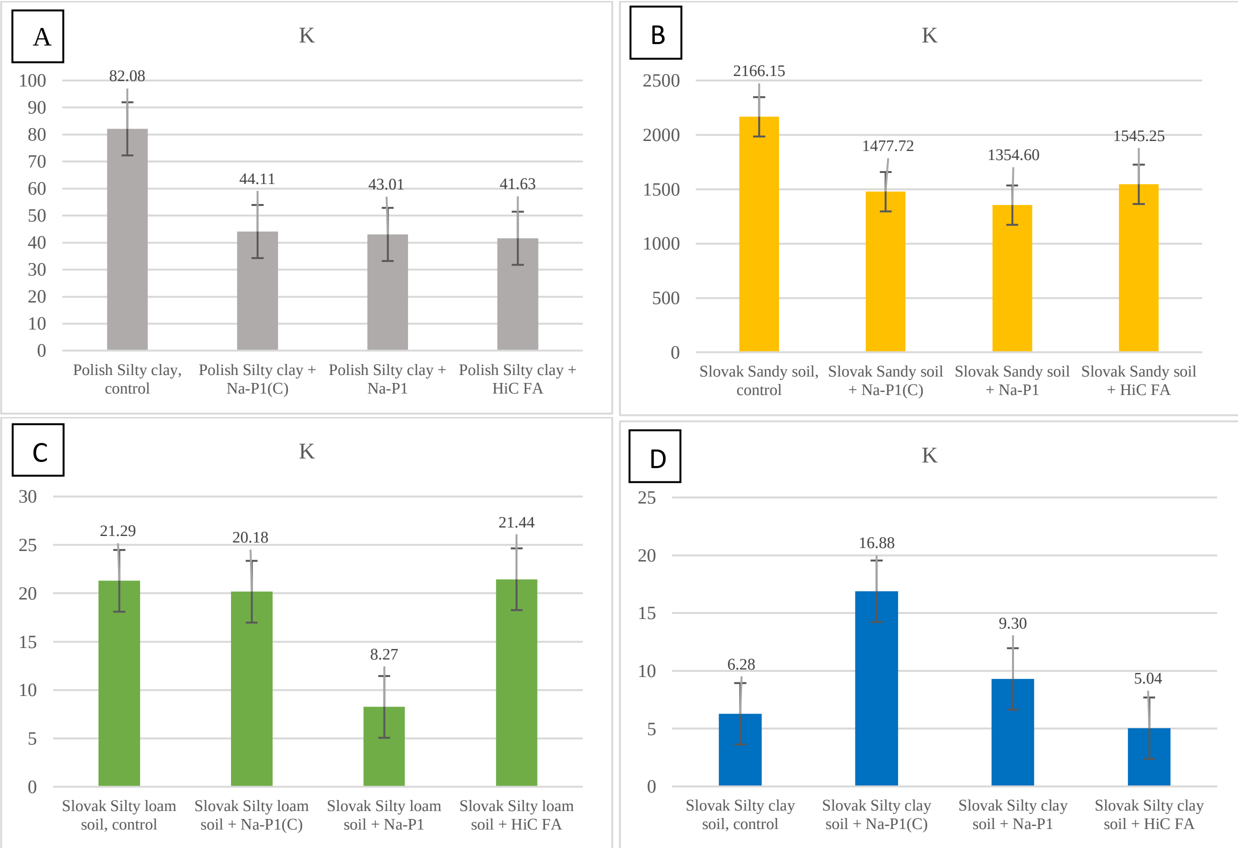

Fig. 4. Saturated hydraulic conductivity (K) values for soils (in cm/day): A – Polish Silty clay soil, B – Slovak Sandy soil, C – Slovak Silt loam soil, D – Slovak Silty clay soil with and without modification with zeolite materials.

spaces, as a result of which the water does not filter quickly. Furthermore, Slovak Sandy soil showed the largest value of *K*. This type of soil dominated by large sand particles tend to have relatively large pore spaces and thus large values of *K*.

In Polish Silty clay soil (Fig. 4A), there was a negative effect of zeolitic materials, when the *K* value decreased by half after the addition of additives in comparison to control. In Slovak Sandy soil (Fig. 4B), the observed effect was positive. The effect of the zeolite additives caused the *K* values to decrease by about 30–38% compared to the control. This is may be a positive finding, because the infiltration is very quick in Slovak Sandy soil and it is necessary to slow it down. In Silt loam soil (Fig. 4C) only the effect of zeolite (Na-P1) on *K* values was found. This effect was negative because, it is not needed to reduce the *K* parameter in this type of soil. The changes in *K* values with the other two additives were not significant in comparison to control. In Slovak Silty clay soil were found also positive changes in *K* values after zeolite additieves in comparison to the control (Fig. 4D). There is needed to increase the infiltration in this type of soil. The highest increase was observed with zeolitic composite (Na-P1(C)), which was more than 600%.

As mentioned in the case of contact angle results, the chemical composition of zeolite materials may affect hydraulic conductivity. As the micropore area and porosity of the additives used increases, the hydraulic conductivity decreased, as can be seen in Polish Silty clay soil and also in Slovak Sandy soil. This was due to the fact that the additive particles filled the space between the soil particles and as a result, the water was filtered lower than unmodified soil (Bilardi et al., 2020). Also other authors observed impact of soil amendments (biochar) on *K* values in different soil (Toková et al., 2023; Vitková et al., 2019; Brockhoff et al., 2010; Uzoma et al., 2011). In the case of Slovak Silty clay soil, the opposite effect was observed. This could be due to the fact that the additives increased the number and size of the pores. Therefore, the water was filtered faster and the *K* values increased.

Conclusion

It can be concluded that the saturated hydraulic conductivity strongly correlates with specific soil properties. The addition of various zeolitic materials had a negative impact on Polish Silty clay soil. Nevertheless, in the case of Slovak sandy soil, a positive effect was obtained due to the slowing down of infiltration, which is preferable for this type of soil. However, it is not possible to select one addition that would show the best results in these soils: the zeolite (Na-P1) had the best effects in the Slovak Sandy soil, it was the zeolitic composite (Na-P1(C)) in the Slovak Silty clay and in the Polish Silty clay soil had all additions similar effects. There was even observed a negative effect of zeolite (Na-P1) in Slovak Silt loam soil; the other two zeolitic additions did not bring significant changes.

The results of the contact angle also clearly indicate that the type of soil being used determines certain values, which were obtained in these experiments. Nevertheless, the application of an addition such as fly ash caused the hydrophobicity of all studied materials to increase. The wettability of modified soils depended largely on the base material which is soil without the additive. As an example, the contact angle for Slovak Sandy soil after the addition of 1% Na-P1(C) and Na-P1 rapidly decreased. Sandy soils with a coarse-grained structure are considered to be less hydrophobic than silty soils, which is attributed to their more permeable structure. In this case, the addition of Na-P1(C) and Na-P1 caused an increase in loosening of the soil structure and consequently a decrease of the contact angle.

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