The effect of expanded perlite and metakaolin on the physicochemical properties of collapsible soils

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Abstract

Collapsible soils, such as loess, are a kind of problematic soil that is naturally unsaturated and withstands high loads at their natural moisture content, but unexpectedly collapses when wet and saturated, creating a risk to buildings constructed on it. This study highlights the effect of chemical stabilizers, including perlite and metakaolin, on the physicochemical behavior of collapsible soils, especially Zeta potential measurement, and the soil's morphology. The properties of natural soil were compared to those of treated soil using a set of Zeta potential measurement tests. Furthermore, scanning electron microscopy (SEM) analysis was used to validate the results. According to the results, perlite and metakaolin changed the loess soil properties. The results showed that the absolute Zeta potential of soils increased after adding perlite and metakaolin, which indicated a higher dispersity of soils mixed with perlite or metakaolin. The scanning electron microscope (SEM) indicated that untreated samples had a loose structure with extensive pores, whereas treated samples had a dense and uniform structure with particle rearrangement. The flocculation and agglomerations in the soil matrix, which are a significant contributing factor to the mechanical property enhancement of the metakaolin-stabilized samples, were confirmed by SEM images. According to the microstructure and product composition analyses, the calcium-aluminate-silicate hydrate (CASH) generated by the metakaolin enhanced cementation between the flake units of the plain soil, and the soil structure of the plain soil stabilized by the metakaolin was denser.

Keywords

Collapsible soil, stabilization, Zeta potential, perlite, metakaolin.

Roskadásveszélyes talajok tulajdonságainak vizsgálata perlittel és metakaolinnal történő talajkezelés hatására

Kivonat

A roskadásveszélyes talajok, mint például a lösz, olyan problémás talajok, amelyek természetes állapotban telítetlenek, és természetes nedvességtartalmuk mellett jó teherbíró képességűek, de telítve váratlanul összeomlanak, és veszélyt jelentenek a ráépült épületekre. A tanulmány vizsgálja a kémiai stabilizátorok, köztük a perlit és a metakaolin hatását az összeomló talajok fizikai-kémiai viselkedésére, különösen a Zéta-potenciál értékére és a talaj morfológiájára. A természetes talaj és a kezelt talaj tulajdonságai összevethetők a Zéta-potenciál mérési tesztsorozat segítségével, az eredmények validálására pedig pásztázó elektronmikroszkópos (SEM) vizsgálatok szolgáltak. Az eredmények szerint a perlit és a metakaolin megváltoztatta a löszös talaj tulajdonságait. Az eredmények azt mutatták, hogy a talajok abszolút Zéta-potenciálja megnőtt perlit és metakaolin hozzáadása után, ami a perlittel vagy metakaolinnal kevert talajok nagyobb diszperzitását jelzi. A pásztázó elektronmikroszkóp (SEM) azt mutatta, hogy a kezeletlen minták laza szerkezetűek, kiterjedt pórusokkal, míg a kezelt minták sűrű és egyenletes szerkezetűek, részecskék átrendeződésével. SEM felvételek igazolták a geopolimer gél kialakulását a talajmátrixban, amely jelentős mértékben hozzájárul a metakaolinnal stabilizált minták mechanikai tulajdonságainak javításához. A mikroszerkezeti- és termékösszetétel-elemzések szerint a metakaolin által generált szilikát-aluminát kolloid fokozta a sima talaj pelyhes egységei közötti cementációt, a metakaolin által stabilizált sima talaj talajszerkezete pedig sűrűbb volt.

Kulcsszavak

Roskadásveszélyes talaj, stabilizáció, Zéta-potenciál, perlit, metakaolin.

INTRODUCTION

Collapsible soils are meta-stable soils that can experience significant deformation and a complete change in particle structure after being wetted, either with or without stress (*Khodabandeh et al. 2020*). Loess is considered as one of the most widespread collapsible soils in arid and semi-arid areas, covering 10% of the world's land surface (*Khodabandeh and Nagy 2022*). Loess soils are known for their open structure, which is produced by sharp-edged grains and has a low dry density, water content and plasticity (*Nokande et al. 2020, Nokande et al. 2022*).

In recent years, researchers have focused on the treatment of loess soils with cost-effective and environmentally friendly materials. Various types of materials, including nanomaterials, polymers, fibers, biological materials, and industrial waste materials, have been widely used to stabilize loess soils (*Khodabandeh et al. 2023a*). Chemical soil stabilization improves soil cohesion and hence shear strength and structural stability by forming interparticle chemical interactions that bond the soil particles (*Khodabandeh et al. 2023b*).

Geopolymers are clinker-free binding materials that provide for building materials good mechanical properties

(*Bakriet al. 2012*), high thermal resistance (*Zhang et al. 2016*) and great chemical resistance (*Mehta and Siddique 2017*). The influence of metakaolin-based geopolymers on dust control and the tensile strength of loess soils was evaluated by *Hanegbi and Katra (2020)*. The geopolymer utilized by *Hanegbi* and *Katra (2020)* behaved remarkably well in terms of dust control and tensile testing. The most successful geopolymer composition, which produced the most soil strength after 28 days, was a sodium silicate and sodium hydroxide (NaOH) activation solution with a 30% metakaolin addition. For the stabilization of sulfate-rich soil, a calcium-free geopolymer synthesized using metakaolin (MK) as an aluminosilicate precursor showed improved strength with no reduction in volumetric expansion in specimens cured for 7 days, based on *Zhang et al. (2015)*. Metakaolin has been examined for its influence on the geotechnical qualities of cohesive soils. The addition of 2% to 12% metakaolin resulted in a decrease in soil-specific gravity and optimal compaction water content. Metakaolin's pozzolanic reaction influenced soil grain size disruption. More than 90% of the swelling was decreased by adding 10% metakaolin (*Ahmad and Hamza 2015*). Further research utilizing metakaolin revealed that the optimal percent of metakaolin in expansive soil stabilization is 6%; at this concentration, the strength and durability values were raised (*Muhammad et al. 2020*). *Calik and Sadoglu (2014a)* investigated the geotechnical properties of soils stabilized with perlite. The results showed that with an increasing amount of perlite up to 10%, the Unconfined Compressive Strength (UCS) of soils increased, and further increases resulted in a decrease in UCS. In a similar study, a reduction in cohesion and an increase in friction angle were observed in soils stabilized with perlite (*Calik and Sadoglu 2014b*).

In the present study, the treatment of soil with perlite and metakaolin was examined to determine their effectiveness in improving the physicochemical properties of collapsible loess soils. The experimental program of this paper includes Zeta potential measurements, and Scanning Electron Microscopic (SEM) observations.

MATERIALS AND METHODS

Properties of the soils

The loess soils utilized in the present study were provided from two sites in Hungary; the first site was in Balatonakarattya, Koppány sor with 47°1'14.93"N 18°8'38.64"E coordinates, and the second site was in Vál in Fejér county with 47°21'25.6"N 18°40'15.7"E coordinates. *Table 1* presents the basic properties of loess soils. Grain size distribution curves for the soil samples are presented in *Figure 1.* X-ray diffractogram (XRD) of soils is presented in *Figure 2.* Quartz, muscovite, and calcite were the main mineral components of soils.

Table 1. The basic properties of loess soils
1. táblázat. A vizsgált talajok tulajdonságai

Parameter	Soil 1 (Balatonakarattya)	Soil 2 (Vál)	Method Used
Liquid limit (LL), Plasticity limit (PL), Plasticity index (PI) (%)	30, 23, 7	26, 23, 3	AST* D4318
Natural water content (%)	5,5	4,5	ASTM D2216
Specific gravity (G_s)	2,70	2,67	ASTM D854
Void ratio (e)	0,9	0,9	ASTM D7263
Dry density (γ_{dry}) (kN/m^3)	14,2	14,2	ASTM D7263
Soil classification (USCS)	CL-ML	ML	ASTM D2487
Soil classification (Euro code)	SiCL	SaSi	EN ISO 14688-2

* *American Society for Testing and Materials*

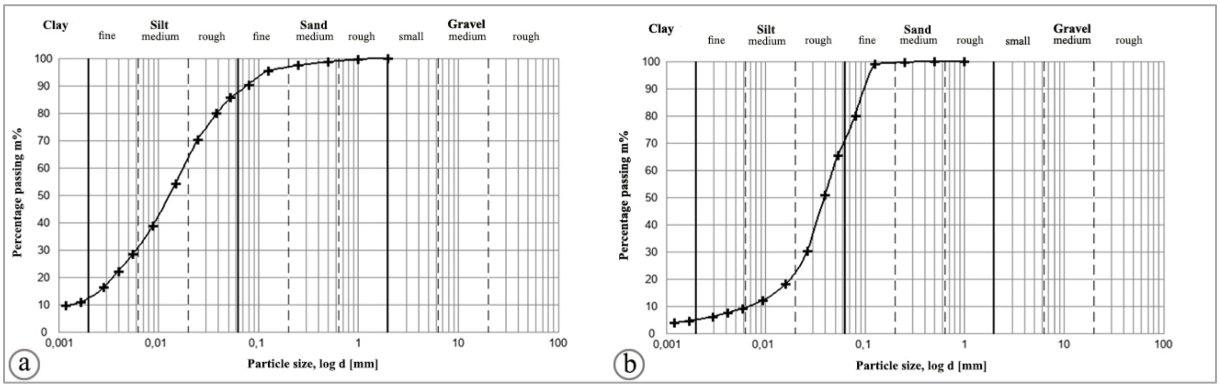

Figure 1. Grain size distribution curves for the soil samples a) Soil 1 b) Soil 2
1. ábra. A talajminták szemeloszlási görbéi, a) Talaj 1, b) Talaj 2 esetére

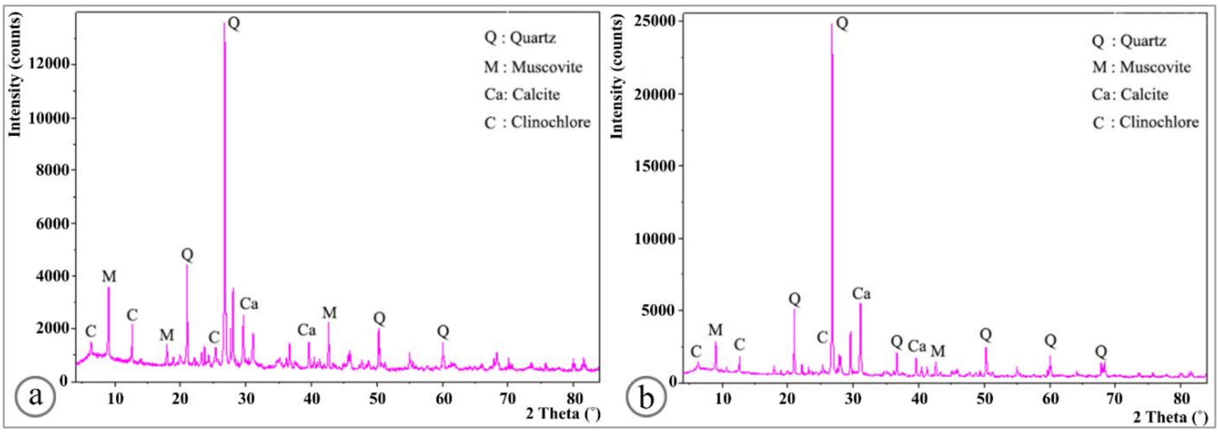

Figure 2. XRD patterns of soils, a) Soil 1 b) Soil 2
2. ábra. A talajminták XRD vizsgálatának eredménye, a) Talaj 1, b) Talaj 2 esetére

Properties of the stabilizing agents

Expanded perlite powder and calcined clay (metakaolin) were chosen as stabilizing agents. The geologic occurrence of perlite is in Pálháza, Tokaj mountain area, Hungary. The expanded perlite powder (Expanded perlite P2) used in this research is a product of ANZO Ltd., Hungary, and was prepared according to *MSZ EN 14316-1*. The main ingredients are SiO_2, Al_2O_3, and Fe_2O_3, which influence the natural pozzolana's activity. Perlite has a siliceous nature based on chemical composition. Metaver N-type metakaolin was produced by Newchem GmbH, Austria. *Table 2* represents the properties of perlite and metakaolin.

Table 2. Chemical composition and particle size of perlite and metakaolin
2. táblázat. A perlit és metakaolin oxidos összetétele és szemcsemérete

Oxide compounds	Perlite	Metakaolin
SiO_2 (%)	68-75%	52-54%
Al_2O_3 (%)	10-14%	40-43%
Fe_2O_3 (%)	<2%	<2.5%
CaO (%)	<2%	<0.5%
MgO (%)	<1%	<0.4%
K_2O (%)	3.2-4.5%	<2.0%
SO_3 (%)	<1.0%	<1.0%
Na_2O (%)	2.8-4.5%	<0.1%
Particle size	0-2 mm	<2 µm

Figure 3 shows the results of XRD patterns for perlite and metakaolin. Both materials show an amorphous nature, and the major crystalline phases of perlite and metakaolin are α-quartz and muscovite.

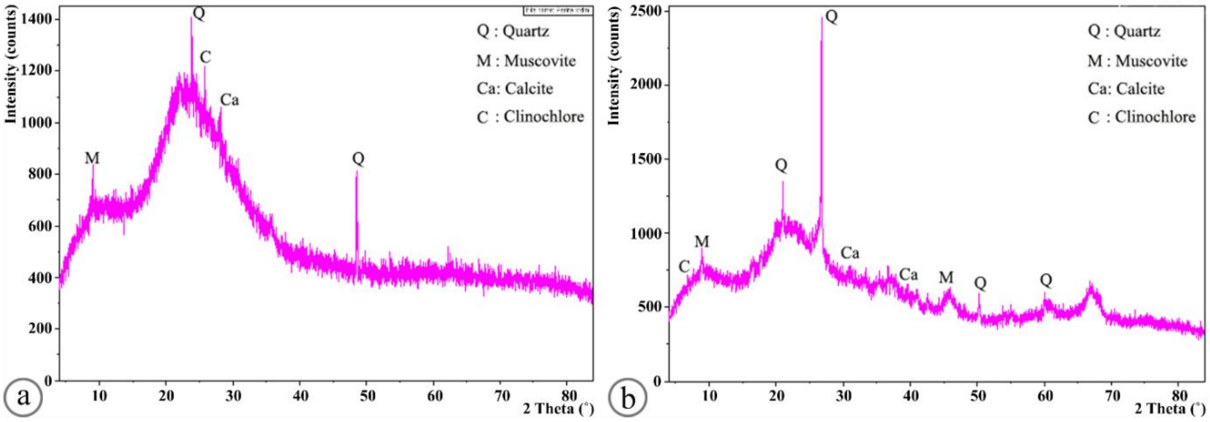

Figure 3. XRD patterns of stabilizing agents, a) perlite b) metakaolin
3. ábra. A stabilizátor anyagok röntgen-diffraktogramjai, a) perlit, b) metakaolin esetére

Sample preparation and experimental plan

At first, the remolded samples were placed into oven at 105 °C temperature for 24 hours to ensure that the soils were totally dry. To remove large particles and achieve a homogeneous distribution of the soil, the oven-dried sample was hand-ground and passed through sieve No. 10 (2 mm) based on ASTM standards. To evaluate the influence of the stabilizing agents on the physicochemical properties of soils, different dosages of perlite and metakaolin were added to the remolded and ground soil at percentages of 2, 4, 6, and 8 by dry weight of the soil. During the hand-mixing of the soil with stabilizing agents, special care was taken to ensure a homogenous mixture. Then, the treated samples were mixed with water to obtain the in-situ moisture content of the soil (5%), according to the previous research (*Khodabandeh et al. 2023b, Nokande et al. 2020, Siddiqua and Bigdeli 2022).* To achieve equilibrium conditions and allow for possible interactions between soil and stabilizing agents, the samples were placed in insulated plastic containers for 7 days for curing based on the literature review (*Ogila and Eldamarawy 2022, Zamani and Badv 2019*). Tests were performed just after this 7-day cure. Treated soil samples with perlite or metakaolin were examined by Zeta potential measurements, and due to Scanning Electron Microscopic (SEM) observations.

Zeta potential measurement

The Zeta potential is an electrokinetic characteristic of dispersed particles in a dispersant (e.g., water) that is used to measure the thickness of the diffuse double layer (DDL) and to understand the physicochemical interactions between instant clay particles and water (*Farahani et al. 2019*). In the present study, Zeta potentials were measured to analyze the electrochemical characteristics of soil samples. At first, hydrometer tests were performed on untreated soil samples in two conditions, including without Calgon solution (Sodium hexametaphosphate) and with Calgon solution. Then, hydrometer tests were performed on soils treated with different percentages of metakaolin and perlite, without and with Calgon solution. After sedimentation of soil samples for 4 hours, the soil solutions were extracted from the top of the hydrometric container and were placed into oven at 60 °C for 48 hours. Zeta potential was examined in the extracted samples dispersed in distilled water. Before each Zeta measurement, the Zeta cuvette was washed three times with distilled water. The Zeta potentials of every sample were measured three times, and the average was used as the final test value. A Zeta potential analyzer (Zetasizer Nano ZS, Malvern, Germany) was used to test the Zeta potential.

Scanning Electron Microscopic (SEM) observations

Scanning Electron Microscopic (SEM) images are one of the best methods to observe and analyze materials' microstructure. Soil samples were studied by scanning electron microscopic method. SEM pictures were scaled to 200 µm, 100 µm, 80 µm, and 30 µm to demonstrate how the microstructure of the soils changed after 8 mass% of stabilizing agents (perlite, and metakaolin) were added to the soil samples. For the SEM observation, a Phenom XL (Thermo Fisher Scientific) desktop scanning electron microscope was used.

RESULTS

The results of Zeta potential measurement

Table 3 presents the results of the Zeta potential measurements. The results show that the Zeta potentials of both soils in the condition without Calgon (Sodium hexametaphosphate) were -17.6 mV and -12.5 mV for Soil 1 and Soil 2, respectively. However, both soils had smaller negative Zeta potentials after adding Calgon to the solutions (-33.1 mV for Soil 1 and -37.7 mV for Soil 2). Because increased electric potential near the soil surface caused a repulsive electric force among the soil particles, the soil became more dispersive at higher absolute Zeta potential levels. Therefore, it can be concluded that with the increase of Calgon, soil particles became more dispersed, and the absolute Zeta potential increased. The Zeta potential of perlite and metakaolin without Calgon were -23.4 and -20.6 mV, respectively, and their absolute values were higher than the absolute Zeta potentials of both soils without Calgon.

Table 3. The results of Zeta potential tests
3. táblázat. A Zéta-potenciál mérések eredményei

Sample	Hydrometer tests condition	Saturation fluid	Zeta potential (mV)
Perlite	without Calgon	water	-23.4
Perlite	with Calgon	water	-
Metakaolin	without Calgon	water	-20.6
Metakaolin	with Calgon	water	-
Clean soil 1	without Calgon	water	-17.6
Clean soil 1	with Calgon	water	-33.1
Clean soil 2	without Calgon	water	-12.5
Clean soil 2	with Calgon	water	-37.7

Figure 4 represents the results of Zeta potential for soils mixed with different percentages of perlite and metakaolin. As shown in *Figure 4*, with increasing perlite and metakaolin the absolute Zeta potential of treated soils increased. However, for soils containing Calgon the rising trend was more evident.

A conceptual model of a diffuse double layer in a water-soil system before and after treatment with perlite or metakaolin is presented in *Figure 5*. As shown in *Figure 5*, by increasing dosages of perlite or metakaolin, the thickness of the diffuse double layer increased. The pore water outside the diffuse double layer is free, but the water inside the diffuse double layer has a certain flow viscosity and is weakly fluid. Lower free layer thickness results in less free water flow space, which can decrease the loess sample's permeability (*Xu et al. 2021*). In other words, the actual cross-sectional area of gravitational water moving through decreases as the double layer's thickness rises. Its permeability thus decreases as the seepage time increases (*Xu et al. 2021*).

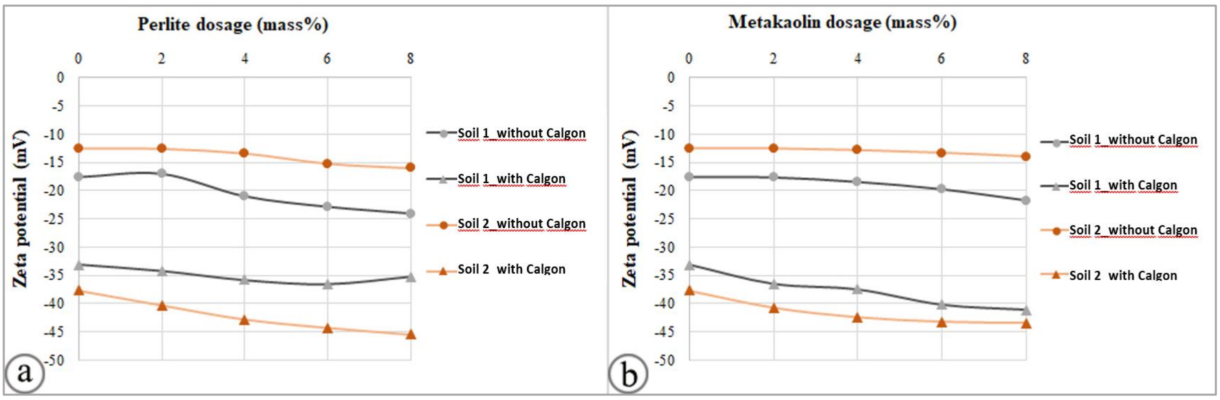

Figure 4. The results of Zeta potential tests, soil samples a) mixed with perlite b) mixed with metakaolin
4. ábra. A Zéta-potenciál mérés eredményei a) perlittel kezelt talaj b) metakaolinnal kezelt talaj esetén

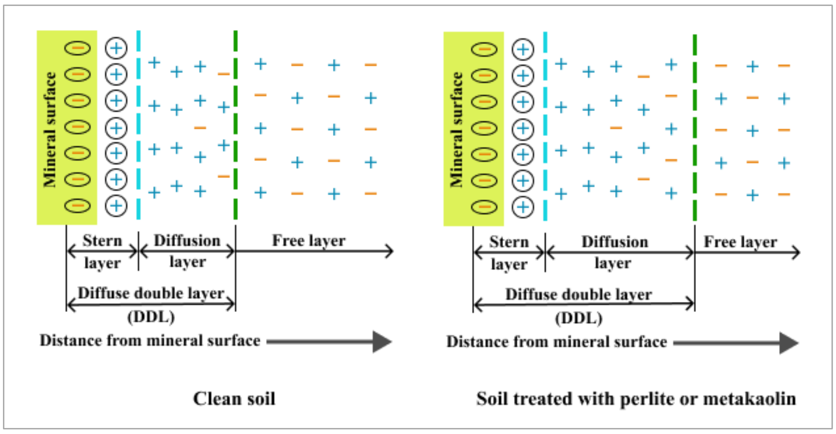

Figure 5. Conceptual model of diffuse double layer in water-soil system before and after treatment with perlite or metakaolin (modified after: Xu et al. 2021)
5. ábra. A víz-talaj rendszer diffúz kettős rétegének koncepcionális modellje, perlittel vagy metakaolinnal végzett kezelés előtt és után (Xu et al. 2021 alapján)

Effect of stabilization on the soil morphology

The results of scanning electron microscopy (SEM) are represented in *Figure 6*. The SEM images of soils treated with perlite are demonstrated in *Figures 6a* to *6d*. As shown in *Figures 6a* to *6d*, with the help of friction and interlocking force, a three-dimensional network of randomly dispersed expanded perlite particles connects soil particle surfaces. Consequently, perlite prevents soil particles from slipping on each other and increases the soil's strength. The spongy structure of expanded perlite particles has been ripped because of the applied axial stress. The failure surface of perlite-treated soil can be seen in *Figure 6c*.

As shown in *Figures 6e* to *6h*, with the addition of 8 mass% metakaolin to the soil, the microstructure of the mixture became denser and more uniform, and the number of cracks and pores decreased significantly. The presence of physicochemical reactions can be responsible for changes in the soil structure. Furthermore, several calcium-aluminate-silicate hydrate (CASH) compounds emerged in the soil structure because of the long-term presence of pozzolanic interactions between soil and metakaolin. CASH phases can fill both macro- and micropores, enhancing cohesion and macro-mechanical parameters.

DISCUSSION

As discussed in the article of Khodabandeh (*Khodabandeh et al. 2023b*) the expanded perlite had a significant influence on the mechanical properties of loess soils (*Khodabandeh et al. 2023b*). With adding perlite, soil became granular, because perlite is mainly composed of sand and gravel-size particles. In soils treated with perlite, a three-dimensional network of randomly dispersed perlite particles binds soil particle surfaces with the help of friction and interlocking forces. This can improve the strength properties of the soil/perlite mixture by preventing slippage of the perlite and soil particles in the matrix.

Metakaolin, which contains 52-54% silica and 40-43% alumina and is considered an aluminosilicate mineral, caused a change in the physical texture of the soil and an increase in the fine-grain content. The chemical interactions between MK and small particles in loess soils (consisting of SiO_2 and Al_2O_3) generated an interparticle bond that made the stabilized soils stiffer. The large surface-to-volume ratio of MK improved particle-soil interaction at the nanoscale. As a result, even a small percentage of the MK changed the physiochemical properties of the soil. It caused flocculation in loess particles, as well as increased particle bond and cohesion. The flocculation and agglomerations in the soil matrix, which are significant contributing factors to the mechanical property enhancement of the metakaolin-stabilized samples, were confirmed by SEM images.

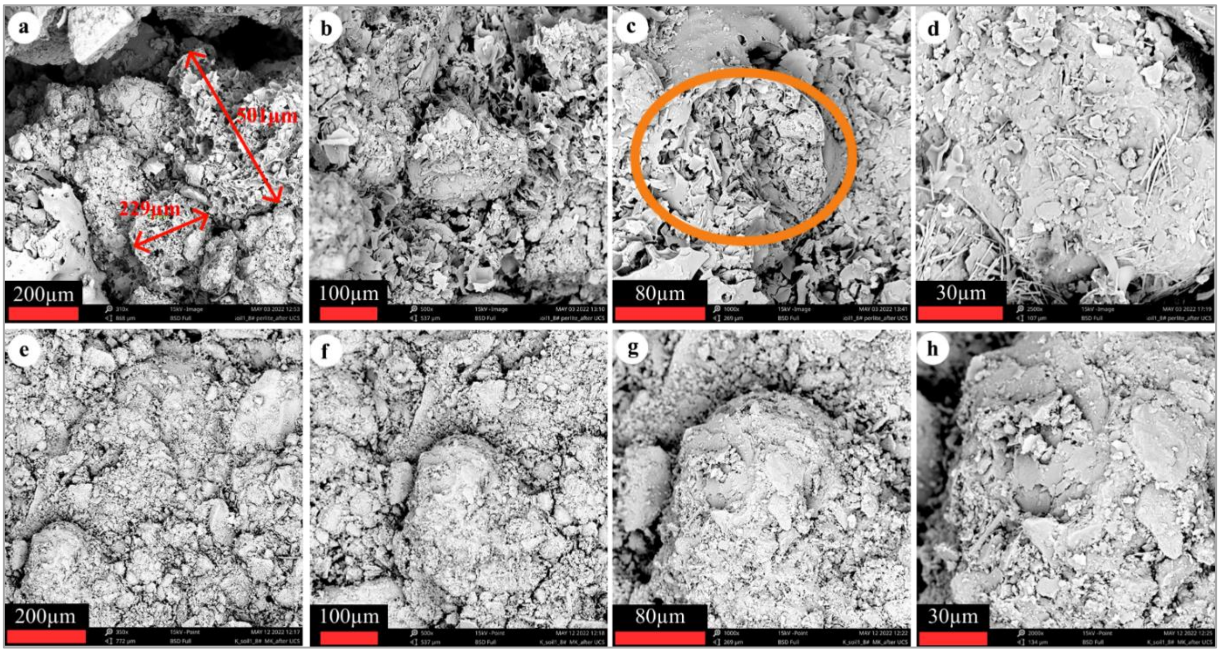

Figure 6. SEM images of soil 1 treated with 8% perlite (a, b, c, d) and soil 1 treated with 8% metakaolin (e, f, g, h)
6. ábra. SEM felvételek 8% perlittel kezelt talaj esetén (a, b, c, d) és 8% metakaolinnal kezelt talajok esetén (e, f, g, h)

The addition of metakaolin to the soil resulted in considerable improvements in the mixture's microstructural properties, such as higher density and improved homogeneity, and a notable decrease in the number of cracks and pores. The presence of metakaolin has caused a few complicated physicochemical reactions that are responsible for the observed modifications in the soil's structure. A variety of calcium-aluminate-silicate hydrate (CASH) compounds emerged within the soil matrix because of the metakaolin's interactions with the soil as a pozzolanic material. Filling macro- and micropores and improving the soil's overall cohesion were shown to be facilitated by the creation of CASH phases. Microstructure and product composition analyses revealed that the CASH colloid generated by metakaolin enhanced cementation between flake units in the plain soil, resulting in a denser soil structure when stabilized by metakaolin. In similar research (*Tabarsa et al. 2018*), soil stabilization was assessed in the field with 1, 1.5, and 2% nano clay. The extent of improvement achieved was evaluated by the flow of irrigation water in the channel. It was concluded that with increasing the percentage of nano clay, less erosion and scouring occurred. A similar behaviour can be predicted for soils stabilized with metakaolin when facing the flow of irrigation water in the channel.

The results of the Zeta potential measurement show that with increasing the percentage of expanded perlite or metakaolin, the values of the absolute Zeta potential increased (which means that the Zeta potential became a smaller negative value); this indicated a higher dispersity of soil. Based on previous research (*Parameswaran 2017*), the dispersity of soils and their Zeta potential has been discovered to be highly correlated. Also, the electrostatic repulsive pressure between soil particles increases as the Zeta potential increases (*Li et al. 2018*).

According to the article of *Khodabandeh et al. (2023b)*, by increasing the percentage of perlite and metakaolin by up to 8%, the highest improvement can be achieved. Perlite stabilization can be suggested when high compaction is not possible, for instance, around the pipelines and the edge of the excavation. However, metakaolin had better functionality after curing time due to the chemical interaction of metakaolin with soil particles during that time. Therefore, when long-term stabilization is considered, metakaolin will have a better effect due to its chemical reactions with soil particles.

CONCLUSIONS

In the present study, soil treatment by two stabilizing agents, with expanded perlite and metakaolin was examined. Our goal was to determine their effectiveness in enhancing the physicochemical properties of collapsible loess soils. The experimental program of this paper includes Zeta potential measurements and Scanning Electron Microscopic (SEM) observations.

The following conclusions may be drawn from the results of this study:

- With the aid of friction and interlocking force, expanded perlite particles connected soil particle surfaces. Metakaolin acted as a filler for honeycomb voids in the collapsible soil, increasing cementation between the grains.
- Perlite-treated soils became more granular in terms of particle size, as soil grains aggregated due to treatment. Perlite particles kept the soil grains from sliding on top of each other. In terms of metakaolin, the calcium-aluminate-silicate hydrate (CASH) colloid generated by the metakaolin enhanced cementation between the flake units of the plain soil, consequently, the metakaolin-stabilized plain soil had a denser soil structure.

- The absolute Zeta potential increased as the dosages of expanded perlite and metakaolin increased, indicating higher dispersity for soils mixed with applied stabilizing agents.
- Scanning electron microscopic observations indicated that untreated samples had a loose structure with extensive pores, whereas treated samples had a dense and uniform structure with particle rearrangement.

Finally, it is obvious that soil improvement with expanded perlite and metakaolin can improve soil engineering characteristics in any construction application. However, expanded perlite is a more cost-effective material than metakaolin.

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