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ВПЛИВ СКЛАДУ МЕТАКАОЛІНІВ НА ВЛАСТИВОСТІ ГЕОПОЛІМЕРНИХ КОМПОЗИТІВ НА ТЕРМОПЕРЛІТНІЙ ОСНОВІ

Геополімери є перспективними сучасними будівельними матеріалами, як альтернатива бетонам на основі портландцементу. Оптимізація хімічного та гранулометричного складу компонентів геополімеру, зокрема метаклаоліну, дозволяє керувати швидкістю твердіння, підвищувати довговічність та механічні властивості, а також адаптувати геополімери до конкретних умов застосування. В даному дослідженні проаналізовано метаклаолін МК 40 та високоактивний метаклаолін ВКМ, їхній хімічний склад та розподіл частинок, а також вплив цих факторів на властивості геополімерів.

Ключові слова: метаклаолін високоактивний ВКМ, метаклаолін МК-40, геополімер, алюмосилікати, композити, полімеризація.

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THE INFLUENCE OF METAKAOLIN COMPOSITION ON THE PROPERTIES OF THERMOPERLITE-BASED GEOPOLYMER COMPOSITES

Geopolymers are promising modern building materials, serving as an alternative to Portland cement-based concretes. Optimizing the chemical and granulometric composition of geopolymer components, particularly metakaolin, allows for controlling the hardening rate, increasing durability and mechanical properties, and adapting geopolymers to specific application conditions. This study analyzes metakaolin MK 40 and highly active metakaolin HAM, their chemical composition and particle distribution, as well as the influence of these factors on the properties of geopolymers.

Keywords: highly active metakaolin HAM, MK-40 metakaolin, geopolymer, aluminosilicates, composites, polymerization.

Introduction

The chemical and granulometric composition of components has a key influence on the properties of geopolymers, affecting their strength, resistance to aggressive environments, thermal conductivity, water absorption, and other properties. The study analyzed metakaolin MK 40 (TU.U 14.2-00191916-001:2005) and highly active metakaolin HAM (TU.U 14.2-36363275-001:2009) were analyzed, which can be used for the production of geopolymers.

Problem statement

Energy efficiency, environmental safety, and durability are important aspects for building materials. Geopolymers are a promising alternative to traditional cement composites. The properties of thermoperlite-based geopolymer composites depend on the composition of the raw materials, in particular on the type and activity of metakaolin, its chemical composition, and granulometric characteristics. The lack of data on the influence of different types of metakaolin (MK-40 and HAM) on the structure formation and operational properties of thermoperlite-based geopolymer composites complicates the process of optimizing the composition of such materials. The lack of a systematic analysis of the relationship between the initial parameters of the raw materials and the final properties of geopolymers does not allow their composition to be formulated to achieve the required characteristics.

Review of Modern Information Sources on the Subject of the Paper

Geopolymer composites are the result of alkaline activation of aluminosilicate materials, which produces a three-dimensional aluminosilicate gel (N-A-S-H) that determines the mechanical and physicochemical properties of the material [1, 4, 5]. One of the most common precursors for the creation of geopolymer composites is metakaolin, whose chemical composition is a key factor in the formation of the structure and properties of the geopolymer. The silicon dioxide (SiO_2) content contributes to the formation of the polymer matrix, increasing the gel density and mechanical strength of the material, as well as its thermal stability [1, 2, 4, 10].

Research results [3, 6] show that a $\text{SiO}_2 / \text{Al}_2 \text{O}_3$ ratio of approximately 3-4 ensures a balance between the formation of a strong matrix and the reactivity of the precursor. Increasing the SiO_2 content to a certain level leads to a denser structure and reduced porosity, which has a positive effect on strength. However, an excess of silicon can reduce the reactivity of aluminosilicates and slow down polymerization, limiting further strength growth [4, 6, 9].

Aluminum oxide $\text{Al}_2 \text{O}_3$ causes the formation of a three-dimensional polymer aluminosilicate network, which provides high thermal and acid resistance of the geopolymer [1, 2, 10]. Works [5, 7] show

that the presence of Al_2O_3 in a certain amount contributes to the formation of stable Si–O–Al bonds, as well as increases the density of the structure and reduces water absorption. The addition of CaO or calcium-containing slags promotes the formation of hybrid gels of the C-A-S-H and N-A-S-H type [2, 4, 11], which significantly increases early strength and accelerates the hardening of the material. However, excess CaO can reduce the chemical stability of geopolymers in an acidic environment, so its amount in the preparation of a geopolymer composite must be regulated [4, 5, 7].

In the geopolymer composition, iron oxide Fe_2O_3 in small concentrations can act as a catalyst for structure formation, increasing thermal stability and stability when heated. In high concentrations, Fe_2O_3 reduces strength due to the formation of less reactive phases and disruption of gel homogeneity [3, 5, 7].

In addition to chemical composition, granulometry and specific surface area of metakaolin particles play an important role [6, 8, 9]. The granulometric composition of raw materials is one of the key factors determining the kinetics of polymerization and the density of the geopolymer matrix structure. Studies show that reducing the particle size of metakaolin and other aluminosilicates leads to an increase in specific surface area, which increases the reactivity of the material during alkaline activation. As Duxson [5] notes, finely dispersed particles promote more intense dissolution of Si and Al in an alkaline environment, accelerating polymerization and the formation of a three-dimensional structure of the geopolymer gel. Similar results were obtained in [10], where it was noted that the use of finely dispersed metakaolin ensures the formation of a more homogeneous and dense structure, which reduces porosity and increases the mechanical strength of the final material. In addition, granulometry significantly affects the microstructure and defectiveness of the matrix.

Kohout et al. [6] experimentally proved that excessively large aluminosilicate particles slow down the polymerization process, causing the appearance of heterogeneous zones and microcracks in the structure, while a uniform distribution of particles smaller than 20 μm contributes to the formation of a dense and uniform matrix. Similarly, Tchakouté et al. [7] note that controlling the particle size distribution allows the degree of gelation to be regulated and the number of unreacted metakaolin grains to be reduced. Thus, granulometry directly determines the polymerization rate, homogeneity, and density of the geopolymer matrix, which is crucial for obtaining materials with high physical and mechanical characteristics.

The assessment of the structural and morphological characteristics of metakaolin, as one of the most effective aluminosilicate bases for the synthesis of geopolymers, allows not only to visualize the morphology of its particles, the degree of amorphousness after calcination and the process of gel formation during geopolymerization, but also the porosity and presence of defects in the formed matrix [5; 13].

According to studies [4; 17], the intensity of gel matrix formation and its porosity depend on the reactivity of metakaolin, i.e., the degree of its structural destruction during calcination. The temperature for obtaining metakaolin is approximately 650–750 °C, resulting in high dispersibility and the largest surface area, which contributes to the effective formation of a three-dimensional aluminosilicate network.

Microstructural observations also show that when the reactivity of metakaolin in the geopolymer matrix is insufficient, unreacted particles remain, forming defects and reducing the density of the structure. SEM analysis in this case reveals characteristic pores and cracks, indicating incomplete polymerization [6, 12, 21]. Conversely, with the correct selection of the alkali solution composition and Si/Al ratio, the microstructure is more homogeneous, without microcracks, with a clearly defined gel background, confirming a high degree of geopolymerization [9; 15].

In addition, the use of SEM in combination with EDS energy dispersive analysis allows the elemental composition of local areas of the structure to be determined, which is important for identifying phases with different concentrations of silicon, aluminum, and alkali cations. This helps to trace the process of metakaolin transition to the N–A–S–H gel phase, characteristic of geopolymers [5, 13, 18, 20].

Assessment of the degree of microstructure compaction and the presence of residual voids, which determine the mechanical strength of geopolymers [19; 20]. Comparison of microstructural data with the results of mechanical tests demonstrates a clear correlation between morphological homogeneity and the strength of samples.

Geopolymers synthesized on the basis of metakaolin are characterized by high density and the presence of an amorphous gel matrix formed as a result of the polymerization of silicate and aluminate groups. According to research [15], when metakaolin is used as the main component, N-A-S-H-type gels are formed, which are responsible for the mechanical properties of the material. Additionally, in studies [15, 19], a microstructural analysis of metakaolin-based geopolymers was performed using SEM methods,

which allowed the identification of characteristic features of the structure and phase transformations in the material.

Objectives and Problems of Research

The aim of this study is to investigate the relationship between the elemental and granulometric composition of aluminosilicate raw materials, in particular metakaolin MK 40 and HAM, the nature of their influence on the course of polymerization, microstructure formation, and the final properties of geopolymer composites. In the search for environmentally sustainable and durable building materials, understanding the role of particle size and nature in structural and chemical processes is of particular importance. The reaction rate of alkali activation, the level of homogeneity of the mixture, and, consequently, the density and strength of the formed matrix depend on the particle size distribution. Therefore, the task is not only to describe the physicochemical patterns, but also to identify practical approaches to optimizing the composition in order to obtain stable, heat-resistant, and mechanically strong geopolymers.

An important task of the study is to gradually investigate the relationship between granulometry and structure formation in geopolymer systems, in particular, to determine the role of particle size in the formation of an aluminosilicate network and to determine the critical parameters that affect the polymerization rate [6, 11]. Therefore, the study aims to establish the limits of metakaolin dispersibility, at which the highest reactivity and maximum matrix density are achieved [4, 7]. By performing a comparative analysis of the microstructure of materials synthesized with different particle size distributions, it is possible to understand how changes in morphology affect the performance properties of the finished samples [10, 12].

Main Material Presentation

The elemental and oxide composition of VCM and MK-40 was measured using an X-ray fluorescence spectrometer (XRF) by two methods. In the first method, loosely packed metakaolin powder was used for measurements. In the second method, measurements were performed on compressed material. To prepare the sample for measurement, 6 g of metakaolin was weighed and 1.5 g of wax was added. The prepared mixture was pressed with a load of 15 T on a Hercules 25T press. The results of the second method are shown in Fig. 1. The increased content of Al in the form of oxide in HAM metakaolin is explained by the high content of the γ - Al_2O_3 phase. This phase is formed during the production of metakaolin in the calcination process, as a result of which, at temperatures of 500–700 °C, gibbsite is transformed into γ - Al_2O_3 . This phase remains inert during geopolymerization, but contributes to the compaction of the structure, effectively acting as a filler that increases compressive strength [5,7].

The formation of γ - Al_2O_3 is also accompanied by a decrease in the specific surface area of metakaolin, which can affect the reactivity of the raw material, but the formation of γ - Al_2O_3 compensates for this effect by improving the mechanical properties of geopolymer materials and their thermal stability [4,8]. The strength of geopolymers also depends on the molar ratio of Si/Al. Studies show [3, 5] that at a Si/Al ratio of ≈ 1.95 , maximum compressive strength is achieved. From which it can be concluded that this ratio, i.e., an increase in the Al_2O_3 content, can lead to a decrease in strength due to the formation of less strong Al–O–Al bonds in the geopolymer structure.

The granulometric composition of geopolymer components has a direct impact on the reactivity of materials. This is because fine particles have a higher specific surface area, resulting in faster alkali activation of geopolymers and increased initial strength. Excessively fine metakaolin particles can reduce the mobility of the mixture and require a larger amount of activator, leading to microcracks and shrinkage. Coarse particles are less reactive but can serve as fillers [9, 10]. The granulometric analysis of the samples was performed using an Anton Paar GmbH (Graz, Austria) laser particle analyzer, which uses the light diffraction method. The measurements were performed in wet mode, which required the preparation of samples of BMC and MK40 metakaolin and their introduction into the device, where light scattering analysis was then performed. The study established statistical values for grain size based on six measurements (Fig.2).

In metakaolin MK 40, a more uniform distribution of particles is observed, which in turn contributes to the compaction of the structure of the geopolymer mixture. Thus, a decrease in the average particle size of metakaolin (to 3.9–10 μm) contributes to an increase in the specific surface area, which improves the dissolution of aluminosilicates during geopolymerization. This leads to the formation of a larger amount of amorphous (glass) phase, which provides a denser microstructure and increased strength of the material [7, 10], as well as a decrease in the porosity of the material, an increase in its strength, and improved frost resistance and water resistance.

Using scanning electron microscopy (SEM) analysis, the morphological features of HAM and MK 40 particles were studied at magnifications from 150 to 1500, and the elemental composition of metakaolins used as an aluminosilicate base for the synthesis of geopolymer binder was determined.

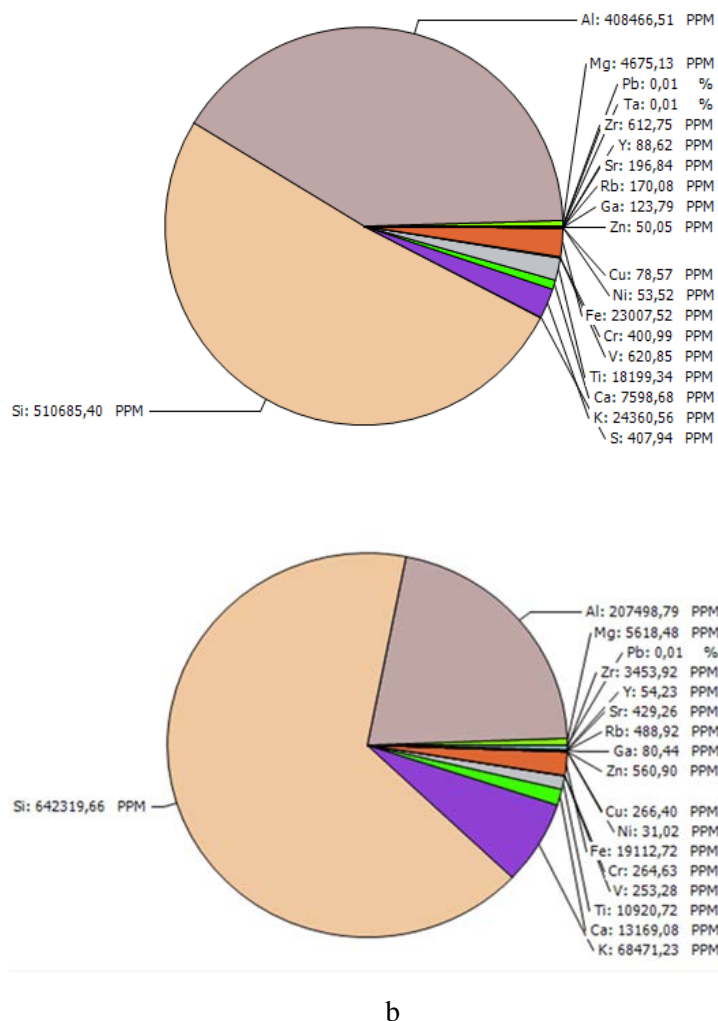


Fig. 1. Elemental composition of metakaolin HAM (a) and MK40 (b)

The obtained VCM images (Fig.3a) demonstrate the aggregated structure of particles with a pronounced leafy surface character, which corresponds to the morphology of kaolinite after thermal activation. The particles have an irregular shape, a size within the range of 1–10 μm , and are characterized by the presence of microcracks and porosity, which are formed as a result of dehydroxylation of kaolinite at a temperature of 650–800 $^{\circ}\text{C}$. The surface of the particles is partially loosened, with amorphous areas, indicating the destruction of the layered structure and the formation of reaction-active centers. Such morphological features indicate the effective conversion of kaolinite into metakaolin, an amorphous aluminosilicate characterized by high reactivity in alkaline activation processes. The appearance of amorphous areas confirms the loss of crystallinity necessary for the formation of an N–A–S–H-type gel, which is formed during the geopolymerization process [1, 3]. The results of EDS energy dispersive analysis of VCM indicate that the main elements of the sample are oxygen, silicon, aluminum, as well as a small amount of potassium and carbon (Table 1). The presence of gold within 10 wt.% is explained by the application of a metal coating prior to SEM analysis to ensure the electrical conductivity of the surface.

The calculated Si/Al ratio within the range of 1.1–2.3 is characteristic of highly active metakaolin used in N–A–S–H-type geopolymers. This range is consistent with the results of studies [15] and [16], where the optimal silicon-to-aluminum ratio ensured the formation of a homogeneous gel phase with high density. The presence of potassium indicates natural impurities of feldspars in the initial kaolinite, while the insignificant carbon content may be due to residual organic impurities or carbon contamination during sample preparation. The absence of iron and titanium indicates the high purity of metakaolin, which is an important prerequisite for the stable chemical composition of future geopolymers.

According to SEM data (Fig.3, b), the MK40 metakaolin sample is characterized by an agglomerated microstructure with a predominance of finely dispersed irregularly shaped particles connected in loose clusters. Local pores and breaks are observed on the surface, which are typical for partially amorphized metakaolin that has undergone dehydroxylation. Unlike the HAM sample, where lamellar fragments with remnants of a layered structure prevailed, the structure of MK40 is more porous and loose, indicating a higher degree of kaolinite thermal activation. Such surface destruction can contribute to increased reactivity during alkaline activation, but excessive porosity potentially reduces the density of the future geopolymer.

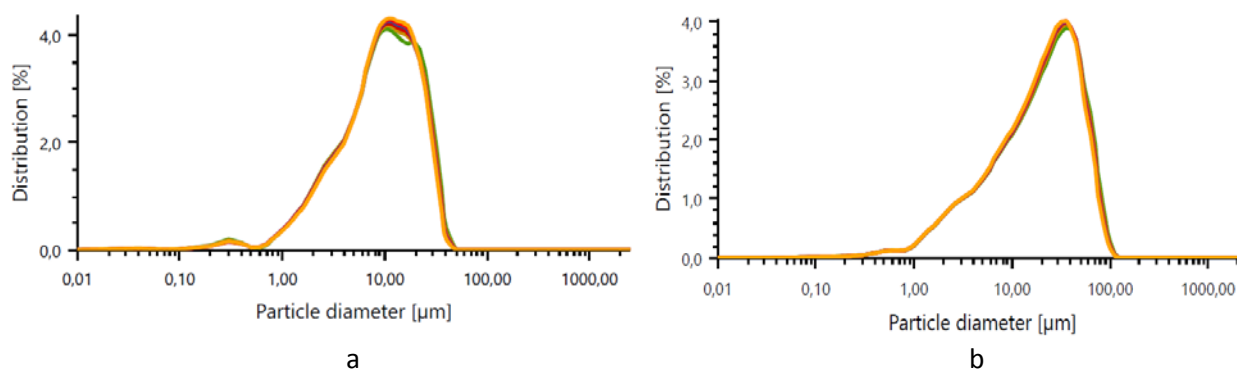


Fig. 2. Results of granulometric analysis of metakaolins HAM (a) and MK40 (b)

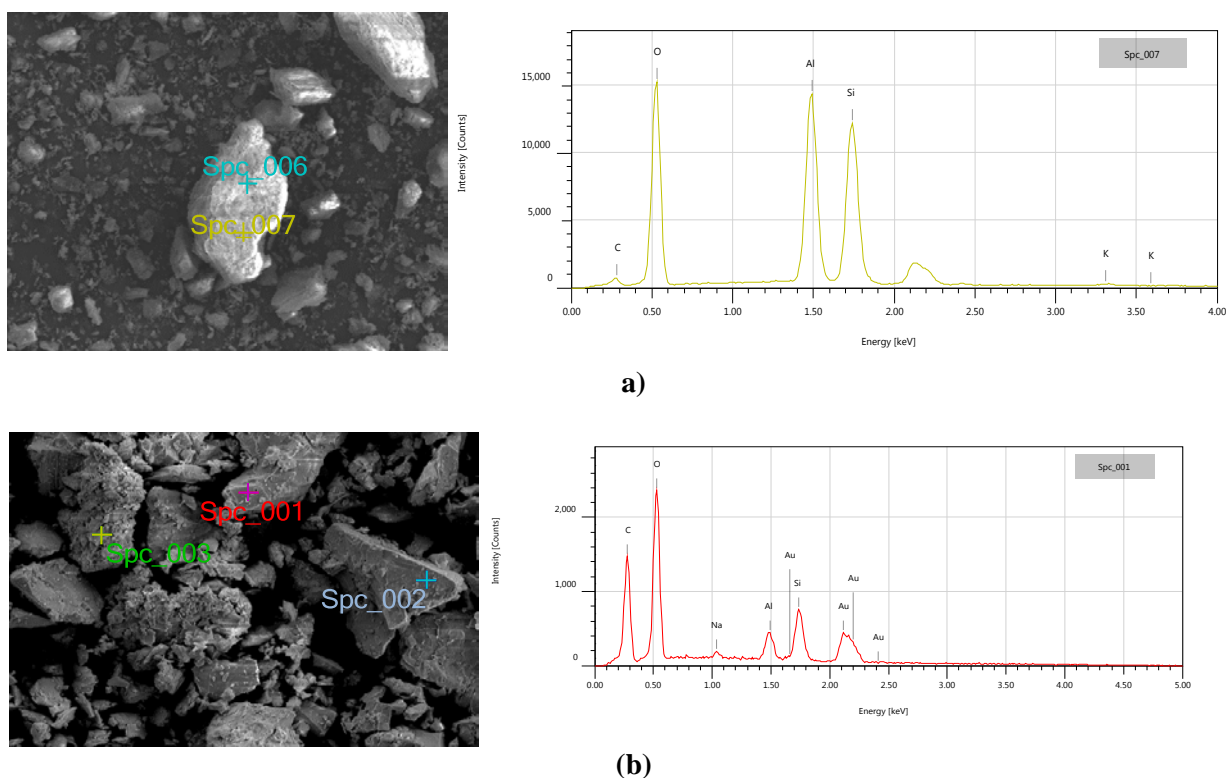


Fig. 3. Results of scanning electron microscopy (SEM) analysis of metakaolins HAM (a) and MK40 (b), x1500

Based on the EDS results of metakaolin MK40, its composition was determined (Table 2), with a Si/Al ratio in the range of 1.5–2.6, which is typical for reactive metakaolins suitable for forming N–A–S–H gel during geopolymerization. Compared to VCM, the MK40 sample has a slightly higher Si content and lower Al content, indicating a higher proportion of the amorphous phase. The presence of up to 2.2 wt% sodium may be the result of prior contact with alkaline environments or natural impurities of feldspars, which is sometimes observed in thermally activated kaolinites.

As can be seen from the SEM results, sample MK40 shows more intense destruction of the crystalline structure of kaolinite, indicating a higher degree of activation. This has a positive effect on reactivity, but excessive porosity may require correction of the alkali activator composition to ensure uniform formation of the N–A–S–H gel. The HAM sample has less developed porosity but a more stable morphology, which

ensures more controlled polymerization. It is likely that its structure will promote the formation of a denser geopolymer matrix with an optimal Si/Al ratio.

Table 1

Results of energy dispersion analysis of HAM

Element	Line	Mass%	Atom%
C	K	6.80±0.12	10.78±0.18
O	K	49.81±0.26	59.29±0.30
Al	K	20.43±0.13	14.42±0.09
Si	K	22.63±0.15	15.35±0.10
K	K	0.33±0.03	0.16±0.01
Total		100.00	100.00
Spс_007		Fitting ratio 0.0402	

For geopolymer synthesis, MK40 can be considered a more reactive metakaolin, suitable for rapid polymerization at low temperatures, while VCM is a structurally more stable material that provides better uniformity and less shrinkage during hardening. For geopolymer synthesis, it is advisable to combine both types of metakaolin in a ratio of 1:1 or 2:1 to achieve a balance between the reaction activity and structural density of the final material.

Table 2

Results of energy dispersive analysis of MK40

Element	Line	Mass%	Atom%
C	K	14.72±0.14	36.93±0.36
O	K	19.53±0.26	36.78±0.48
Na	K	0.69±0.07	0.90±0.09
Al	K	4.75±0.19	5.30±0.21
Si	K	11.80±0.34	12.66±0.36
Au	M	48.52±1.42	7.42±0.22
Total		100.00	100.00

The results obtained are consistent with the morphological characteristics of metakaolin given in [16], [17], [18], [19]. According to these studies, after thermal activation, metakaolin exhibits a heterogeneous microstructure with amorphous areas that promote alkaline activation and the formation of an aluminosilicate gel matrix. The high oxygen and silicon content with a relatively low aluminum content indicates a well-dehydroxylated phase capable of polymerization when interacting with alkaline activators. A similar correlation between the degree of amorphization, reactivity, and microstructure was also established in the works [19], [20], and [21].

Geopolymer components with an optimized SiO₂ and Al₂ O₃ ratio and fine grains, such as MK40 metakaolin, are raw materials for producing high-quality geopolymers. Unlike fly ash, which has low reactivity and requires greater grinding or the use of activator additives, metakaolin MK40 requires less Na₂ O/K₂ O for polymerization. The high Al₂ O₃ content in BMK metakaolin can reduce the reactivity in the geopolymerization process and cause a decrease in the mechanical properties of the geopolymer due to the formation of weaker Al–O–Al bonds in the geopolymer structure.

Conclusions

The study found that the chemical and granulometric composition of metakaolin significantly affects the structure formation process, polymerization rate, and operational properties of thermopearlite-based geopolymer composites. The main factors determining the quality and homogeneity of the formed geopolymer matrix are the Si/Al ratio and the degree of amorphization of the initial aluminosilicate material.

A comparative analysis of two types of metakaolin — HAM and MK40 — showed that the MK40 sample has a higher degree of thermal activation, increased silicon content (approximately 35 wt.%) and a more developed porous microstructure, which determines its high reactivity in the alkali activation process.

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At the same time, HAM metakaolin is characterized by a more stable, lamellar morphology, a Si/Al ratio in the range of 1.8–2.2, and lower porosity, which ensures controlled hardening and the formation of a dense matrix with minimal shrinkage. The calculated Si/Al ratios within the range of 1.1–2.6 for both types of metakaolin correspond to values sufficient for the formation of an N–A–S–H type aluminosilicate gel, which determines the strength and stability of geopolymers.

An effective Si/Al ratio of approximately 1.9–2.0 ensures the formation of a uniform three-dimensional network with increased density. It has been established that a decrease in the average particle size of metakaolin (to 3.9–10 μm) contributes to an increase in specific surface area, improved dissolution of aluminosilicates in an alkaline environment, intensification of the polymerization process, and the formation of a denser amorphous structure of the geopolymer matrix. This leads to an increase in the strength, frost resistance, and water resistance of the material. The results of SEM and EDS analyses confirm that MK40, having a more porous and amorphous structure, contributes to rapid initial hardening, but requires optimization of the activator composition to prevent the formation of defects. In contrast, HAM exhibits stable particle morphology and ensures uniform polymerization with the formation of a dense matrix, which contributes to the increase in the final strength of the composite.

High performance characteristics of thermopearlite-based geopolymers can be achieved by combining VCM and MK40 metakaolins in a ratio of 1:1 or 2:1, which allows for a balance between reaction activity and structural density. The results obtained indicate the possibility of targeted control of the properties of geopolymer composites by varying the composition, dispersibility, and ratio of metakaolin components, which opens up prospects for the creation of environmentally friendly, energy-efficient, and durable building materials of a new generation.

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