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Patterns of formation of the pore matrix structure and thermophysical properties of calcareous-ash mortars modified with opoka and recycled basalt fiber

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Abstract. The study investigates patterns in the formation of the structure and properties of calcareous-ash mortars modified with silica rock opoka and recycled basalt fiber. The research goal was to establish the relationship between the composition, pore structure, and thermophysical and physico-mechanical characteristics of the material.

The raw components used included lime, fly ash, granulated blast furnace slag, opoka, quartz sand, and recycled basalt fiber obtained from spent mineral wool substrate after heat treatment. The conducted studies involved the determination of density, compressive and bending strength, thermal conductivity, shrinkage strain, and microstructure.

The introduction of opoka was found to contribute to the formation of a microporous structure, providing a 20-35% reduction in average density and a decrease in thermal conductivity to 40-55%. Recycled basalt fiber reduced shrinkage strain and improved crack resistance by stabilizing the pore matrix structure. The study showed that synergetic interaction of components leads to the formation of a hierarchical structure providing the optimal combination of strength and thermophysical characteristics.

The optimal density range achieving the best balance of properties has been determined (1,400–1,600 kg/m³).

Keywords: calcareous-ash mortar, opoka, basalt fiber, thermal conductivity, microstructure, porosity, mortar

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1. INTRODUCTION

Improving the energy efficiency of building materials is one of the key objectives of modern construction, since a significant proportion of operational energy costs is formed by heat losses through enclosing structures [1-5]. In this connection, the development of heat-efficient mortars combining low density, low thermal conductivity, and sufficient strength characteristics presents a topical area of research [6-10, 11].

Traditional cement-sand mortars are characterized by high density and thermal conductivity, which limits their use in energy-efficient structures low density and low thermal conductivity while maintaining mechanical characteristics [1, 2, 12]. Among the considered alternatives are calcareous-ash and calcareous-pozzolanic systems, which can provide better thermophysical characteristics [6-10]. As suggested by Malathy et al. [6], pozzolanic additives in lime mortars can significantly reduce thermal conductivity but create the problem of insufficient strength and instability of the structure.

The introduction of man-made mineral components such as fly ash and granulated blast furnace slag is recognized as an effective way to regulate the structure and properties of mortars [13-20], similar principles are applied in modern drilling fluid development using superhydrophobic clays [23]". Fly ash has been shown to reduce the density and change the morphology of the pore structure [1, 2, 13], while slag ensures the formation of a strong cementing matrix due to hydraulic activity [15-17]. Moreover, researchers note a pronounced synergistic effect of the combined use of ash and slag that makes it possible to achieve reduced thermal conductivity while maintaining sufficient strength [18-21].

One of the key determinants of the thermophysical properties of building materials is their pore structure. Recent studies have shown that advanced analysis methods can significantly improve the understanding of material behavior [24]. A number of studies have shown that the thermal conductivity coefficient depends not only on total porosity but also on the distribution of pores by size, the degree of their closure, and their spatial organization [3-5, 21, 25-26]. Khushefati and Ademaj [3] note that a decrease in thermal conductivity is most effectively achieved by forming a developed system of small closed pores. Similar conclusions were drawn by Asadi et al. [4], who have demonstrated the close relationship between the characteristics of the pore space and the thermophysical properties of cement composites.

A promising option for forming such a structure deliberately is the use of natural silica rocks with a high specific surface and pozzolanic activity practical applications in ceramics and polymer composites [25]. One of such materials is opoka containing amorphous silica [28-34]. Studies by Šiaučiūnas et al. [28] have established that opoka actively participates in hydration processes and facilitates the formation of a denser and more uniform structure of cement systems. Moreover, several studies have demonstrated that opoka also affects the hardening kinetics and the development of strength in composite binders [30-32]. However, the influence of opoka on the formation of pore structure and thermophysical properties of mortars has not been thoroughly researched.

Another effective way to modify the structure of building materials is dispersed reinforcement with fibers. The efficiency of the use of dispersed reinforcement of fine-grained concrete is proved [35]. Basalt fiber, due to its high strength and chemical and heat resistance, is widely used for the purposes of improving the crack resistance and durability of composites [36-43]. Fibers and their properties for concrete reinforcement [44]. Amran et al. [45] and Guo et al. [46] have shown that reinforcement with fiber improves microstructure and prevents the development of microcracks. Zhou et al. [47] note that basalt fiber raises the durability of cement mortars by stabilizing their structure.

The use of basalt fiber in lime systems is of particular interest. The research by Illampas et al. [9], Villanueva et al. [10], and Mercuri et al. [38] indicates that the introduction of basalt fiber improves strength characteristics and reduces cracking. However, the influence of recycled basalt fiber obtained from waste on the structure and properties of lime-ash mortars is virtually unexplored. Fiber concrete for industrial and civil construction [48].

Thus, our literature analysis concludes that current research has thoroughly investigated calcareous-ash and ash-slag systems [13-20], the effect of pore structure on thermophysical properties [3-5, 22, 26], and the reinforcing effect of basalt fiber [37-43, 45-47, 49]. However, we can note a lack of systematic research on the complex effect of opoka and recycled basalt fiber on the formation of the pore matrix structure and thermophysical properties of calcareous-ash mortars.

In this connection, the present study aimed to establish the patterns of formation of the pore matrix structure and thermophysical properties of lime-ash mortars modified with opoka and recycled basalt fiber, as well as to identify the mechanisms behind the influence of these components on the strength and operational characteristics of the material.

2. MATERIALS AND METHODS

2.1. Physico-Mechanical Properties

The determined parameters included: average density according to the standard methodology for mortars; compressive strength 28 days after hardening; bending strength according to the three-point flexural test; and shrinkage strain based on change in the linear dimensions of samples over time.

Thermophysical properties

The thermal conductivity coefficient (λ) was determined by the method of steady-state heat flow. The tests were carried out on samples dried to constant weight.

Microstructural analysis

Microstructure was studied by means of scanning electron microscopy on a JSM-IT 200 scanning electron microscope. The analysis made it possible to evaluate porous structure, phase distribution, and the interaction of fibers with the matrix.

2.2. Phase Analysis

The phase composition of the examined samples was determined by X-ray diffraction (XRD), which made it possible to detect the formation of hydrosilicate phases (C–S–H) and evaluate the intensity of pozzolanic reactions.

Structural and property analysis procedure

Patterns in the formation of material properties were established through a comprehensive approach that included comparing composition and density, analyzing the dependence of thermal conductivity on porosity, testing of the effect of fiber on crack resistance, and correlation analysis of structural and performance parameters. The main focus of the study was on establishing the interrelationship of "composition – porous structure – material properties".

Raw materials

The study used mineral and man-made components capable of forming a lime-ash binding system with a controlled pore matrix structure.

The main binding component used was construction lime (slaked lime) from the lime plant of West Kazakhstan Building Materials Corporation JSC (WKBMC JSC), which contained no less than 90% of active oxides CaO + MgO and conformed to the requirements of GOST 9179-2018.

Lime served as a source of calcium hydroxide, which enables pozzolanic reactions.

Fly ash from the thermal power plant was used as an active silica additive. The chemical composition of the fly ash is provided in Table 1.

Table 1. Chemical composition of fly ash from Ekibastuz GRES-2.

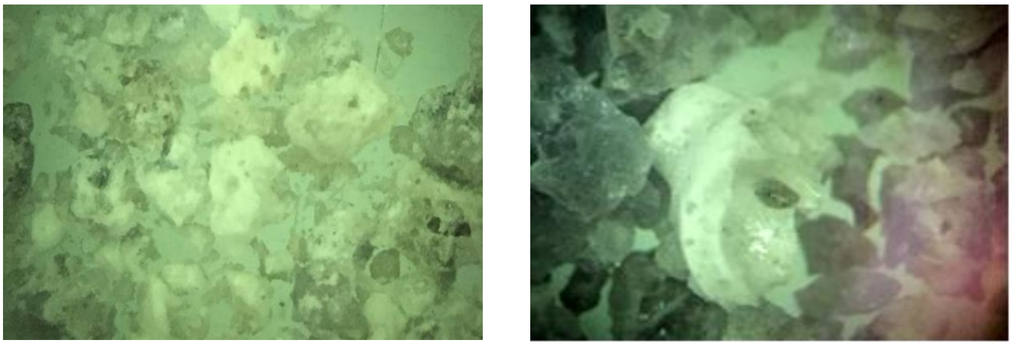
No.	Sample title	Oxides, wt%										
		SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	MnO_4	TiO_2	P_2O_5	LOI
16	Raw material "Fly ash GRES-2"	61.04	23.78	5.82	2.78	0.79	0.59	0.36	<0.04	1.01	0.37	3.04

The chemical composition of the ash is distinguished by a high content of SiO_2 and Al_2O_3, and its physico-chemical characteristics (Table 2) determine its pozzolanic activity and ability to participate in the formation of calcium hydrosilicates.

Table 2. Physico-chemical characteristics of fly ash from Ekibastuz-GRES-2.

Physico-chemical characteristics	Name
	Fly ash from Ekibastuz-GRES-2
Moisture (W^{rt}),%	0.2866
Ash content (A^{r}),%	97.1469
Fugacity (V^{a}), %	1.9636
Total pore volume, cm^3/g	0.78
Bulk density ρ, g/cm^3	0.804
pH of aqueous extract	8.40
Adsorption activity for methyl orange, mg/g	58.00
Adsorption activity for methylene blue, mg/g	37.50
Adsorption activity for iodine, %	127.0

Granulated blast furnace slag from QAZQARMET JSC (Karaganda, Kazakhstan) is a loose gray material. Its glassy structure is mainly caused by the abrupt cooling of the slag melt during granulation (Fig. 1).



a) b)

Fig. 1. Microstructure of glassy granulated blast furnace slag from QAZQARMET JSC (Levenhuk 320 Series microscope): a – magnification x20; b – magnification x50.

The microscopic images clearly show the glassy structure of the slag. The content of the glass phase, which was used as a hydraulically active component, reaches 65–97%. The chemical and granulometric composition of the slag is reported in Tables 3 and 4.

Table 3. Chemical composition of granulated metallurgical slag from QAZQARMET JSC.

Raw material	Oxides content, wt%												
	SiO_2	Al_2O_3	Fe_2O_3	CaO	FeO	MgO	SO_3	Na_2O	K_2O	CO_2	TiO_2	S_hO	LOI
Granulated blast furnace slag from ArcelorMittal Temirtau JSC	40.62	16.24	0.19–0.52	42.11	0.43	5.33–10.39	1.66	0.36–1.5	0.42–1.32	-	0.62–0.88	0.11–1.37	0.92

Table 4. Granulometric composition of metallurgical slag from QAZQARMET JSC.

Sieve hole diameter, mm	2.5	1.25	0.63	0.315	0.14	< 0.14
Sieve residue, %	14–17	35–37	26–30	14–17	2–5	2–4

Prior to testing, the slag was dried and ground to a specific surface area of 2,500–3,500 cm²/g in an MShL-1P laboratory ball mill to ensure its activation in the alkaline medium of the lime-ash system.

The silicic porous additive used was opoka from the Taskalinskoye field – a light, hard, microporous silica rock containing amorphous silicon dioxide (opal-cristobalite).

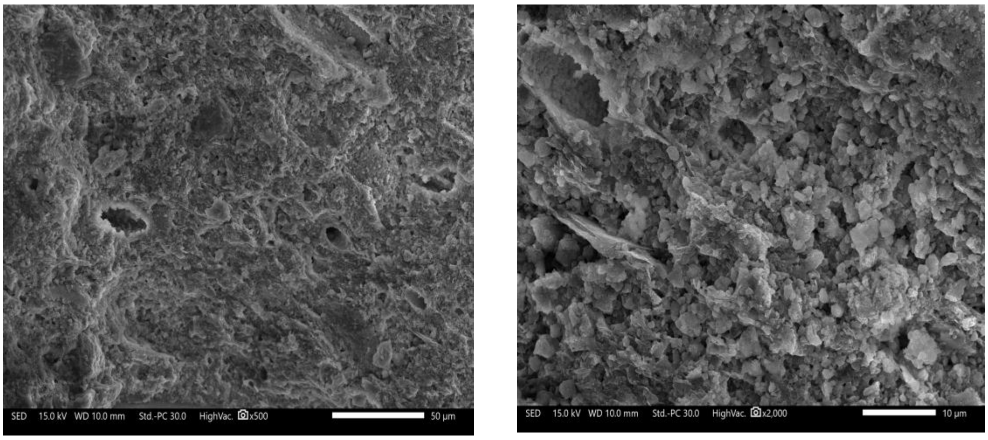
According to geological data, opoka is found in Paleogene and Cretaceous deposits, formed in marine basins as a result of the compaction and cementation of diatomites and tripoli. Their density averages at 1.3–1.5 g/cm^3. Opokas are described as white or gray, greenish light rocks with occasional remnants of diatoms, radiolaria, and sponge spicules.

The mineralogic composition of opoka from the Taskalinskoye field is described in Table 5.

Table 5. Mineralogic composition of opoka from the Taskalinskoye field.

No.	Visible	Ref. Code	Compound Name	Chemical Formula	Score	Scale Factor	SemiQuant [%]
1	True	01-070-2517	Quartz low – theoretical	SiO_2	62	0.978	-
2	True	01-073-0603	iron(III) oxide	Fe_2O_3	35	0.047	-
3	True	00-048-0476	Octadecasil	SiO_2	12	0.147	-
4	True	00-009-0478	Anorthoclas, disordered	$(Na, K)(Si_3 Al)O_8$	30	0.169	-

The morphology of the opoka surface was determined from photomicrographs taken with a JSM-IT 200 scanning electron microscope (Fig. 2).



a) b)

Fig. 2. Porous structure of the siliceous rock of opoka from the Taskalinskoye field: a – magnification x500, b – magnification x2,000 (JSM-IT 200 scanning electron microscope).

The microstructure studies established that the examined opoka is characterized by high natural porosity (55-60%).

Thus, the choice of this raw material was justified by the rock's natural porosity and lightness, the features of its chemical and mineral composition, the presence of a large deposit in the West Kazakhstan region, and the multifaceted influence of this additive at the stages of manufacturing products from highly porous materials.

To increase the reactivity of opoka and ensure its uniform distribution in the matrix, it was ground to a specific surface area of 2,500–3,500 cm²/g in an MShL-1P laboratory ball mill.

The chosen aggregate was quartz sand from the Melovye Gorky deposit (Uralsk, Kazakhstan) of medium size with a fineness modulus of 1.5–2.0.

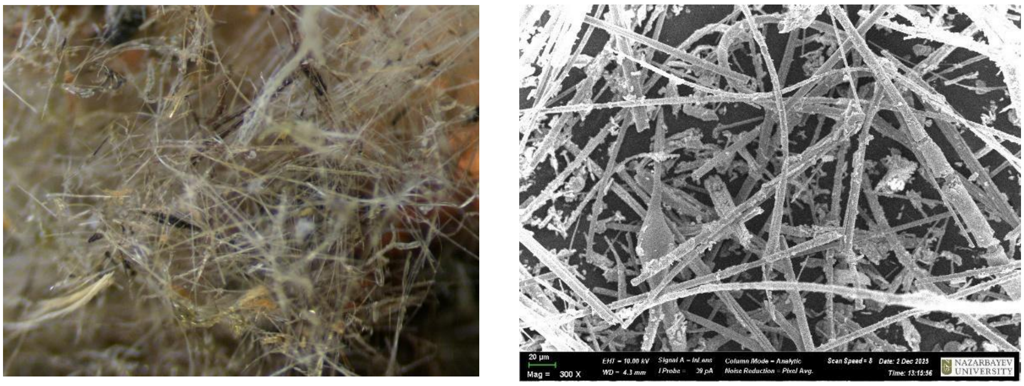
The dispersed reinforcing component was recycled basalt fiber obtained from the spent mineral wool substrate from the Green World greenhouse farm (Uralsk, Kazakhstan). Before the tests, the substrate was heat treated in a laboratory muffle furnace at a temperature of 300°C to remove organic inclusions in the form of plant roots.

Fig. 3 shows the general view of the basalt fiber substrate in its natural, heat-treated, and loosened state.

a) b) c)

Fig. 3. General view of basalt fiber substrate in the natural (a), heat-treated (b), and loosened state (c).

The structure of the fibers was examined through microscopic and electron microscopic studies of the heat-treated basalt fiber substrate (Fig. 4).



a) b)

Fig. 4. Microscopic images – a (Levenhuk 320 Series microscope) and electron microscopic images – b (JSM-IT 200 scanning electron microscope) of the heat-treated basalt fiber substrate.

The microscopic and electron microscopic studies show that despite the heat treatment of the substrate at 300°C, the basalt fiber retained its structure without any changes. The length of dispersed fibers is 0.3–1.0 mm. The obtained results thus indicate that the fiber can be used as a structure-forming component in various composites.

The mixing water used for the study conformed to requirements for technical water for the preparation of mortars.

To establish the effect of basalt fiber content on the structure and properties of mortars, we conducted a one-factor experiment in which fiber content was varied while maintaining the basic composition of the mineral matrix.

The variable parameter in the experiment was basalt fiber content, which was set at 3, 4, and 5 wt% determined by the requirements of bending strength and resistance to dynamic loads [48] (Table 6).

Table 6. Studied compositions of lime-ash mortars.

Component, wt% (dry mix)	Composite 1	Composite 2	Composite 3
Fly ash	33	32	31
Lime	12	12	12
Slag	20	20	20
Opoka	10	10	10
Sand	22	22	22
Basalt fiber	3.0	4.0	5.0
Total	100	100	100

The amount of mixing water was determined experimentally proceeding from the condition of the resulting mortar having normal density and the required mobility. The water-binder ratio was taken as (W/B) = 0.40–0.45

The process of preparing mortar mixtures consisted of the following operations: grinding opoka and slag to a specific surface area of 2,500-3,500 cm^2/g; dry mixing lime, fly ash, slag, and opoka; introducing quartz sand; gradually introducing basalt fiber to prevent agglomeration; adding water to normal density; and mixing for 3-5 minutes.

The resulting mortar mixture was placed in metal molds to make cubic samples with a size of 7 × 7 × 7 cm. The samples were molded in layers, compacting the mixture by light vibration or spading to remove air inclusions and ensure a uniform structure.

Once the molds were filled, the surface of the samples was leveled. After molding, the samples were left in molds for 24 hours at (20 ± 2)°C. Demolded samples were subjected to normal hardening in a special bath at a temperature of (20 ± 2)°C and a relative humidity of 90-95%.

The hardening time was 28 days. The normal hardening mode was used to allow the occurrence of pozzolanic reactions, the formation of hydrosilicate phases (C–S–H), and the stabilization of the pore matrix structure. Increased humidity prevented premature drying and ensured uniform development of the material structure.

3. RESULTS AND DISCUSSION

The results of the conducted studies are presented in Fig. 5-9.

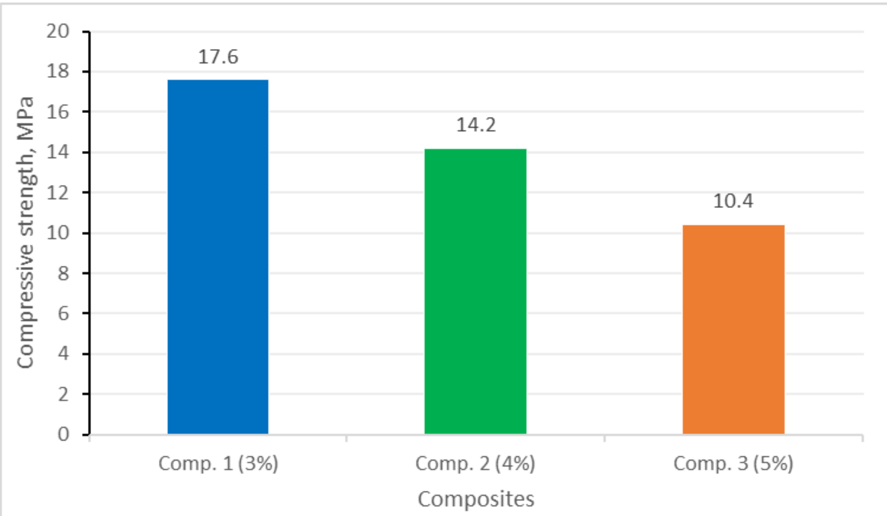


Fig. 5. Influence of basalt fiber content on the compressive strength of different mortar composites: ▨ Composite 1 (3% basalt fiber); □ Composite 2 (4% basalt fiber); □ Composite 3 (5% basalt fiber).

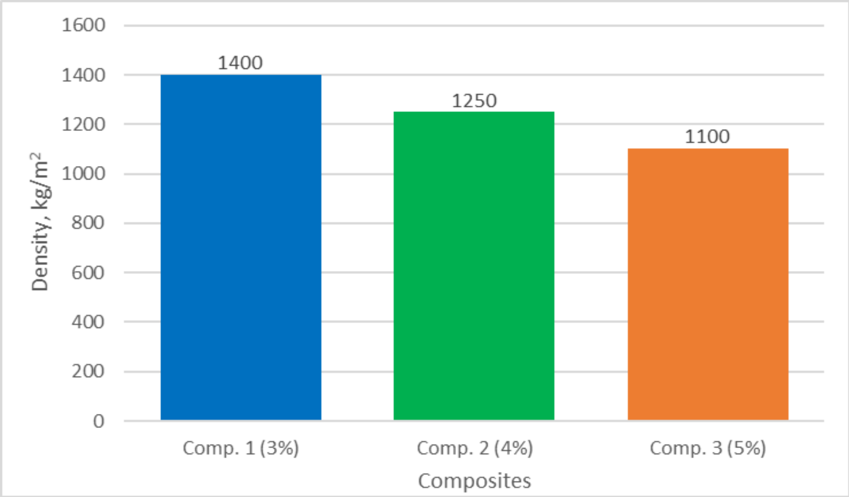


Fig. 6. Changes in the average density of mortars depending on basalt fiber content: ◍ Composite 1 (3% basalt fiber); □ Composite 2 (4% basalt fiber); □ Composite 3 (5% basalt fiber).

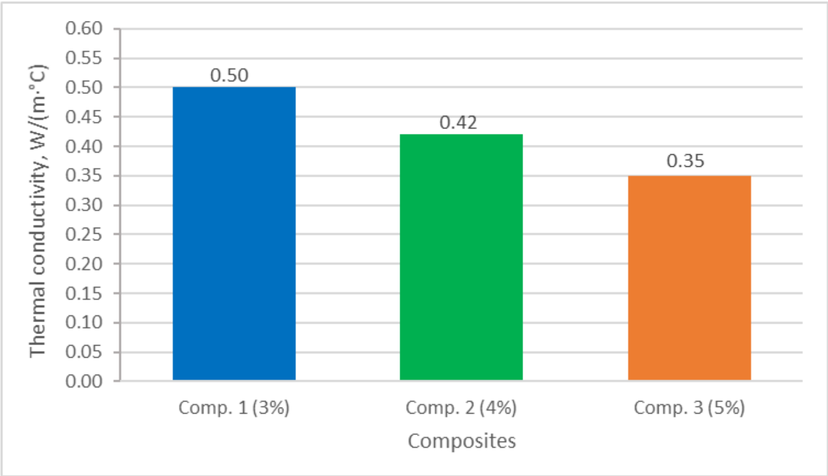


Fig. 7. Dependence of the thermal conductivity coefficient of mortars on basalt fiber content: ◍ Composite 1 (3% basalt fiber); □ Composite 2 (4% basalt fiber); □ Composite 3 (5% basalt fiber).

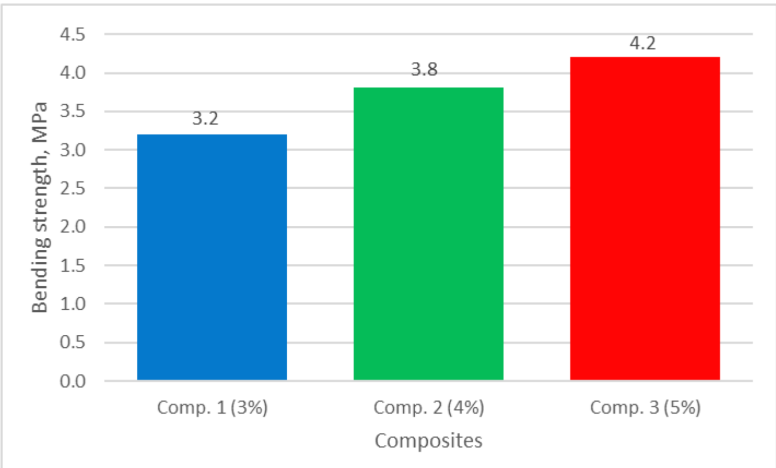


Fig. 8. Influence of basalt fiber content on the bending strength of mortars: ◍ Composite 1 (3% basalt fiber); □ Composite 2 (4% basalt fiber); ◍ Composite 3 (5% basalt fiber).

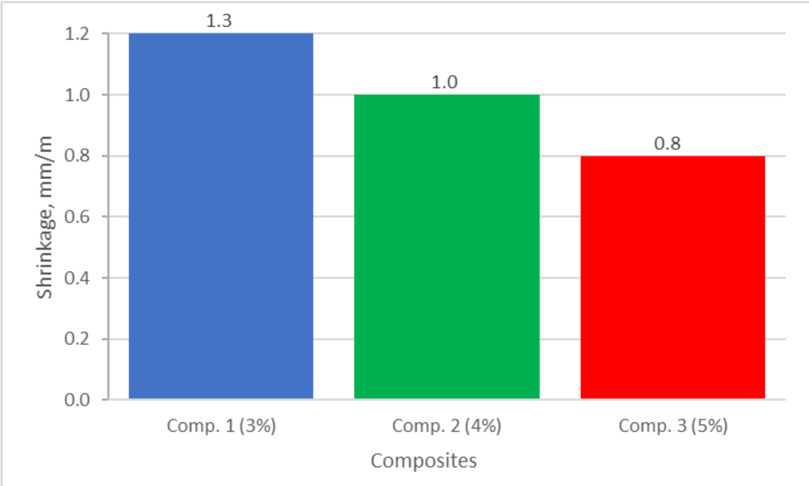


Fig. 9. Dependence of the shrinkage strain of mortars on basalt fiber content: Composite 1 (3% basalt fiber); Composite 2 (4% basalt fiber); Composite 3 (5% basalt fiber).

Analyzing the experimental data (Fig. 5-9), we can see that an increase in the basalt fiber content from 3 to 5% was accompanied by a 9-12% decrease in density and a 15-20% decrease in the thermal conductivity coefficient

The results indicate that thermal conductivity reduced not only due to a decrease in density but also as a result of changes in the structure of the pore space.

The introduced fiber increases the proportion of interphase boundaries, creates additional micropores, and increases the proportion of closed air inclusions. This is confirmed by foreign studies, which show that the decrease of thermal conductivity is directly connected with an increase in porosity and the violation of heat flux continuity [18, 50]. A study by Liu et al. [50] found that the formation of a porous structure results in the blockage of thermal channels and thus causes a decrease in the thermal conductivity of the composite. Similar findings have been obtained by Rashad [18], indicating that a decrease in density and an increase in porosity result in thermal conductivity lowering by up to 60%.

With a higher fiber content, we can observe a reduction of compressive strength and an increase in bending strength. The decrease in compressive strength results from increased porosity and reduced matrix density. On the other hand, the increase in bending strength is explained by the effect of fiber bridging, the redistribution of stresses, and the inhibition of microcrack development. These results are fully consistent with foreign studies. Yıldırım et al. [39] have shown that basalt fiber significantly raises bending strength and crack resistance by virtue of the crack bridging mechanism. Similar conclusions are presented in a study by Branston et al. [51], which observed an improvement in the bending characteristics and post-critical behavior of mortars with reinforcement with basalt fiber.

As demonstrated in Fig. 9, the increase of fiber content from 3 to 5% provided a 30-40% decrease in shrinkage. The fibers absorb tensile stresses, limit the opening of microcracks, and stabilize the structure in the early stages of hardening. These findings agree with the works of foreign researchers demonstrating that fibers significantly reduce shrinkage strain and improve the stability of the structure [39, 51]. Fine-grained concrete with various types of fibers [52].

Fly ash functions as an active silica additive and participates in pozzolanic reactions, interacts with $Ca(OH)_2$, facilitates the formation of low-base calcium hydrosilicates (C–S–H), and forms the basis of the matrix. As a result, a dispersed cementing structure is formed.

Slag acts as a hydraulically active component and is activated in an alkaline environment, participates in the formation of an additional cementing phase, and increases the strength and stability of the structure. This results in the formation of a strong matrix frame.

Opoka serves a double function. As a pozzolanic additive, it participates in the formation of C–S–H phases, and as a pore-forming component, it has a natural microporous structure and increases the proportion of closed pores. Consequently, it contributes to a decrease in density and thermal conductivity and facilitates the formation of a finely porous structure.

Basalt fiber performs micro-reinforcement functions. At the same time, it prevents the development of microcracks, redistributes stresses, and stabilizes the structure. As a result, bending strength is increased, shrinkage is reduced, and crack resistance is raised the greatest increase of strength and deformation characteristics is observed [48].

Importantly, all of the above components work not in isolation but interconnectedly: ash and opoka form a reactive matrix, slag strengthens the structure, and fiber stabilizes it.

These processes create a hierarchical pore matrix structure that includes a matrix (C–S–H phases), a system of micropores, and a reinforcing fiber network. This suggests that the properties of the material are decided not by individual components but rather by the patterns of their interaction and structure formation.

Thus, it was established that the formation of the structure of the studied lime-ash mortar shows a multifactorial dependence on its composition, which can be presented in a generalized form as: Structure = f(ash, slag, opoka, fiber).

The study has shown that the structure of the material is formed as a result of the synergistic interaction of its components, including the pozzolanic reactions of ash and opoka with calcium hydroxide, the hydraulic activation of slag, and the dispersed reinforcement of basalt fiber the active pozzolan reaction, which starts when the concrete mix is mixed with water [53]. This interaction creates a hierarchical pore matrix structure consisting of a cementing matrix (C–S–H phases), a system of evenly distributed micropores, and a spatially oriented fiber mesh.

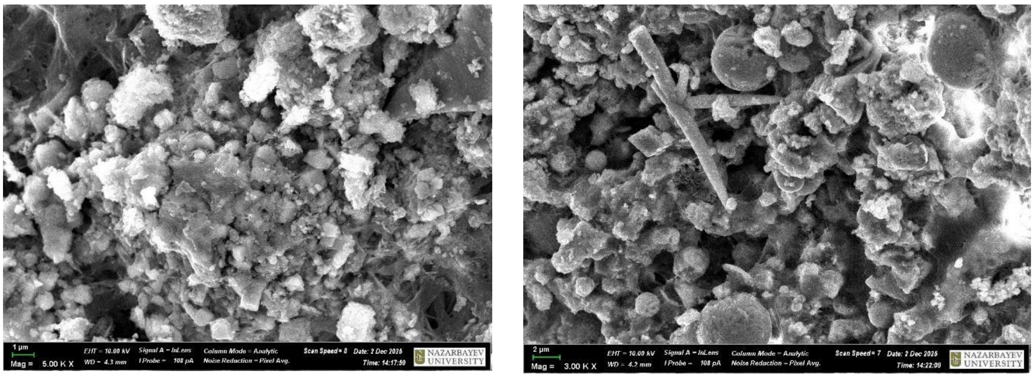
It has been established that the contribution of each component is interconnected: ash and opoka determine the development of the pore structure and thermophysical properties, slag ensures the formation of a strength frame, and fiber stabilizes the structure and prevents the development of defects.

As a result, the complex of material properties is purposefully regulated, which includes a decrease in thermal conductivity, an increase in crack resistance, and the optimization of strength characteristics.

Therefore, the proposed functional dependence reflects not additive but synergistic structure formation, which allows considering the developed composition a system with controlled structural and operational parameters. The obtained results indicate that the improvement of properties owes to the combined action of the lime-ash matrix, opoka (microporous structure + pozzolanic activity), slag (strength frame), and basalt fiber (micro reinforcement).

The formation of the structure of the tested material can be described as a function of its composition: Structure = f(ash, slag, opoka, fiber), which reflects the complex influence of pozzolanic, hydraulic, and reinforcing mechanisms on the formation of the hierarchical pore matrix structure.

These mechanisms result in the formation of a hierarchical pore matrix structure, as confirmed by electron microscopy (Fig. 10).



a) b)

Fig. 10. Hierarchical pore matrix structure of mortar samples (JSM-IT 200 scanning electron microscope): a – magnification x5,000, b – magnification x3,000.

The obtained findings are in good agreement with modern studies. The observed decrease in thermal conductivity with increasing fiber content is consistent with a study by Zhang et al. [41], where a significant decrease in thermal conductivity was noted with an increase in the content of fibers. The "density–thermal conductivity" relationship aligns with the general laws established in the works on heat-insulating composites [54]. The synergy of mineral additives and fibers is supported by studies on geopolymer and ash-slag systems [39, 46].

Thus, our study has established that the formation of the structure of the studied lime-ash mortar is subject to a multifactorial dependence on its composition, which can be presented in a generalized form as a "fly ash – granulated blast furnace slag – silica rock (opoka) – basalt fiber" system.

The study has shown that the structure of the material is formed as a result of synergistic interaction of its components, including the pozzolanic reactions of ash and opoka with calcium hydroxide, the hydraulic activation of slag, and dispersed reinforcement by basalt fiber. Such interaction leads to the formation of a hierarchical pore matrix structure consisting of a cementing matrix (C–S–H phases), a system of evenly distributed micropores, and a spatially oriented fiber mesh.

It has been established that the contribution of each component is interconnected: ash and opoka determine the degree of pore structure development and thermophysical properties, slag is responsible for the formation of a strength frame, and fiber stabilizes the structure and prevents defects. This achieves directed control of the complex of material properties, including a decrease in thermal conductivity, an increase in crack resistance, and the optimization of strength characteristics. High Strength Fiber Concrete for Industrial and Civil Engineering

In this way, the proposed functional dependence reflects not additive but synergistic formation of the structure, which makes it possible to consider the developed composition as a system with controlled structural and operational parameters [55].

For the first time, our study established that the decrease in thermal conductivity owes to the formation of a pore matrix structure stabilized by fibers and not just to the decrease of material density.

4. CONCLUSIONS

1. The study found that the introduction of 3-5% of basalt fiber into calcareous-ash mortars causes a directional change in the structure and properties of the material. With an increase in fiber content, average density decreased by 9-12% and thermal conductivity lowered by 15-20% due to the formation of a more developed microporous structure.
2. It has been shown that the decrease in thermal conductivity is determined not only by a decrease in density but also by the structural reorganization of the material, including an increased proportion of closed pores and the growth of interphase boundaries. This confirms the decisive role of pore matrix structure in the formation of thermophysical characteristics.
3. It was established that an increase in basalt fiber content is accompanied by a decrease in compressive strength with a simultaneous increase in bending strength up to 30%, which stems from the activation of the dispersed reinforcement mechanism and the effect of crack bridging.
4. The introduction of fiber was found to decrease shrinkage strain by 30-40% due to the redistribution of internal stresses and the stabilization of structure in the early stages of hardening.
5. The combined use of fly ash, granulated blast furnace slag, opoka, and basalt fiber was proven to lead to the formation of a hierarchical pore matrix structure that provides both low thermal conductivity and sufficient strength.
6. For the first time, it was established that the formation of the material structure has a functional relationship with its composition: Structure = f (ash, slag, opoka, fiber), which reflects the synergistic nature of the interaction of components and allows purposefully managing mortar properties.
7. The study identified the optimal range of basalt fiber content (3-4%) that achieves the optimal balance between the thermophysical and mechanical characteristics of the material.
8. The obtained findings confirm the prospects of using the developed composites to create energy-efficient building materials with reduced thermal conductivity and increased operational reliability.

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