

SYNTHESIS AND STUDY OF PROPERTIES OF GEOPOLYMER MATERIALS DEVELOPED USING LOCAL NATURAL RAW MATERIALS AND INDUSTRIAL WASTE

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Abstract. This paper describes the synthesis of geopolymer materials using thermally modified clay rocks of Georgia and metallurgical granulated blast-furnace slag; as well as the study of their physical and mechanical properties, durability, and corrosion resistance in aggressive solutions. The methods of chemical, X-ray phase, differential thermal analysis and SEM were used.

Keywords: geopolymer, thermally modified clay rock, durability, aggressive solution, corrosion resistance.

1. Introduction

Climate change and environmental issues have long been a worldwide concern, but in the recent decade, they have become particularly pressing. A number of researchers around the world have taken numerous efforts, trying to identify the possibility of applying global modern technologies, considering overriding values and principles of maintaining a clean environment.¹⁻⁴

The increase in the consumption of cement/concrete as the main building material is due to the rapid growth of the world population in the late 20th and early 21st centuries. Despite the uniqueness of ordinary Portland cement (OPC), its production is characterized by several negative properties: high consumption of energy resources and raw materials, as well as high carbon dioxide emissions (due to the decarbonization of limestone). Based on the foregoing, scientists around the world are looking for alternative sources of energy, raw materials, and the creation of new types of binders that

would become an analog of Portland cement with an improved environmental performance.⁵

The world cement industry currently contributes about 8 % of global carbon dioxide emissions from the clinker production process. In addition, the cement industry is associated with high fuel and energy costs, as it requires high-temperature firing in the range of 1673-1723 K, which has a particularly negative impact on the environment against the backdrop of global warming.

Alkaline-activated cement (AAC) is one of the most promising technological platforms for the development of energy- and resource-saving production of building materials, products, and structures. V. D. Glukhovskiy⁶ was the pioneer in the development of technology for alkali-activated cement, he was the first to discover the possibility of making binders from low-basic calcium or calcium-free aluminosilicates (clays) and solutions of alkali metals. He named these binders "soil cement" and "soil silicates" to reflect their resemblance to natural minerals.

V. D. Glukhovskiy and his collaborators,⁷ have carried out fundamental research in this direction, as a result of which a new class of alkaline or alkali-activated cement (AAC) appeared. His scientific school under the leadership of P. V. Krivenko has proposed principles for the production of these types of cement, including the ones involving by-products and industrial waste.⁸⁻¹⁰

Analysis and generalization of numerous works of various authors and his own studies of alkaline activation binders allowed the French scientist J. Davidovits^{11,12} to develop the concept of geopolymer binders – inorganic materials of a polymer structure based on thermally treated aluminosilicate materials – kaolin and feldspar rocks, as well as ash, slags and other industrial wastes that exhibit binding properties during alkaline activation. It was J. Davidowitz who said that: Geopolymers are Ceramic-Like Inorganic Polymers.

Geopolymer material (GPM), obtained by alkaline activation of aluminosilicates at ambient or slightly elevated temperature, has an amorphous or semi-crystalline polymer structure with Si⁴⁺ and Al³⁺ cations

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tetrahedrally coordinated and linked by oxygen bridges.¹³ When hydrated, the geopolymers produce a substance that is predominantly calcium silicate hydrate.

According to the studies of A. Palomo *et al.*^{14,15} there are two models of alkaline activation: the first applies to slag-alkali binders when blast-furnace slag (Si + Ca) is activated by an alkali solution of medium concentration. In this case, the main reaction products are calcium hydrosilicates – CSH. According to the second model, a concentrated alkaline solution activates compounds of aluminosilicates such as metakaolin, the main oxides of which are Al₂O₃ and SiO₂. In this case, a material of higher mechanical strength and polymer structure is obtained.

At the initial stage of obtaining geopolymer binders, metakaolin was used as an aluminosilicate material – a product of heat treatment of kaolin clays at temperatures of 1023-1123 K. When heated, the dehydration of kaolinite occurs according to the following scheme: Al₂O₃·2SiO₂·2H₂O → Al₂O₃·2SiO₂ + 2H₂O. Heat treatment increases the amount of the amorphous phase, which increases the reactivity of aluminosilicates.

However, the widespread use of kaolin clay is impossible due to the limited resources.

In the Caucasian Institute of Mineral Resources (Tbilisi State University) for a number of years, the work has been carried out to study the possibility of obtaining geopolymer binders using local raw materials. Technologies have been developed for obtaining geopolymer materials using thermally modified clay rocks of Georgia, on the basis of which materials of various compositions have been synthesized.¹⁶⁻¹⁸

Despite the fact that the processes of structure formation of geopolymers have not yet been studied well enough, these binders are considered a promising resource-efficient alternative to Portland cement. Studies have proven that geopolymers have an essential advantage in means of their high strength and density, water resistance, fire and thermal stability; corrosion resistance, and durability.¹⁹⁻²⁰ But still, presently, the main advantage of using these materials is just the opportunity of consuming the huge amount of accumulated industrial waste around the world.

The purpose of this research is the synthesis of geopolymer binding materials developed using thermally modified clay rocks of Georgia and metallurgical granulated blast-furnace slag and the study of their physical and mechanical properties, durability, and corrosion resistance in aggressive solutions.

2. Experimental

Clay rocks of Georgia were used for research: shale from Kvareli, argillite from Teleti, clay from Gardabani, as well as granulated blast-furnace slag from the Rustavi Metallurgical Plant.

An alkaline activator – NaOH, Na₂CO₃, and Na₂SiO₃ or their mixture was used as a mixing liquid.

A NETZSCH derivatograph with STA-2500 REGULUS thermogravimetric and differential thermal analyzer (TG / DTA) were used for thermogravimetric analysis. Samples were heated to 1273 K, in a ceramic crucible with the heating rate of 283 K/min. The reference substance was α-Al₂O₃.

The X-ray phase analysis was carried out using a Dron-4.0 diffractometer (“Burevestnik”, St. Petersburg, Russia) with a Cu anode and a Ni filter. U=35kv. I=20mA. The intensity was 2 degrees/min; λ=1.54178 Å.

Scanning electronic microscope (SEM) measurements were performed on a JEOL scanning electronic microscope JSM-6510LV (well-appointed by energy-disperse X-Max No.20 micro-X-ray spectral analyzer produced by Oxford Instruments). SEM measurements were carried out by means of reflected (BES) as well as secondary (SEI) electrons at an accelerating voltage (at 20 kV). The working distance was approximately 15 mm. Micrographs have been taken at the diverse enlargements.

Temperature modification of clay rocks was carried out by heating the material in a muffle furnace to a temperature of 973 K with exposure at the maximum temperature for 1 h.

Geopolymer materials were prepared as follows: granulated blast-furnace slag and modified shale, mudstone, or clay in a ratio of 80:20, were milled together in a laboratory ball mill to a specific surface area of 8000-10000 g/cm². Dry substances were added to the resulting powder in a certain amount: NaOH or Na₂CO₃, or Na₂SiO₃, or a mixture thereof, which were well mixed for 5 min. The dry mixture was mixed with water until a dough of normal density was obtained. Samples were molded with a size of 2 x 2 x 2 cm. The molds together with the samples were wrapped in a polyethylene film to prevent the binder from drying out and immediately placed in a heat treatment chamber. Heat treatment of geopolymer materials was carried out at 353 K for 20 hours. The samples were cooled spontaneously in the chamber until reaching room temperature.

To test the corrosion resistance, geopolymer materials were immersed in aggressive solutions: H₂SO₄ (concentration 2 % and 5 %), HCl (concentration 2 % and 5 %) and Na₂SO₄ (concentration 5 %).

3. Results and Discussion

Table 1 shows the chemical compositions of clay rocks.

Clay minerals (14.66 – 14.96, 7.14, 4.25, 3.66, 2.86, 2.327 Å) are noted on the X-ray diffraction patterns (Fig. 1); quartz (3.34 Å); feldspar (3.87 Å), calcium carbonate (3.03 Å).

Table 1. The chemical composition of clay rocks, mass. %

Clay rock	L.O.I.	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	FeO	Mn ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O
Shale	4.50	59.95	0.89	17.30	3.45	3.65	0.59	1.53	2.43	0.30	2.20	2.20
Argillite	7.01	47.19	-	15.90	13.36	-	0.10	6.30	4.10	1.39	2.86	1.30
Clay	10.60	52.84	-	15.07	6.47	-	-	7.06	2.49	1.36	1.19	2.17

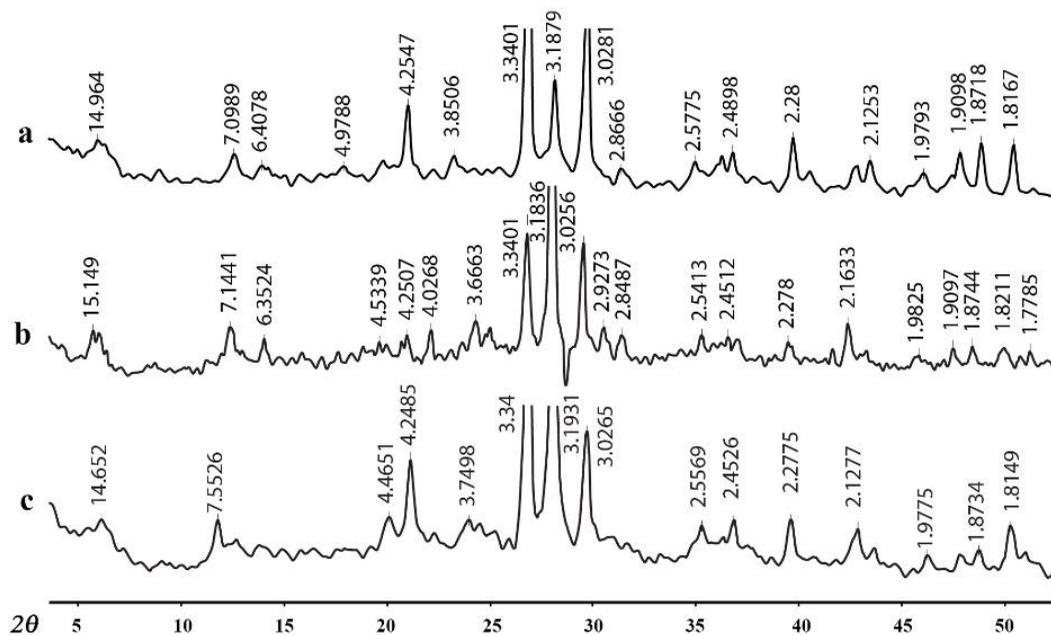


Fig. 1. X-ray diffraction patterns of clay rocks: a – shale, b – argillite, c – clay

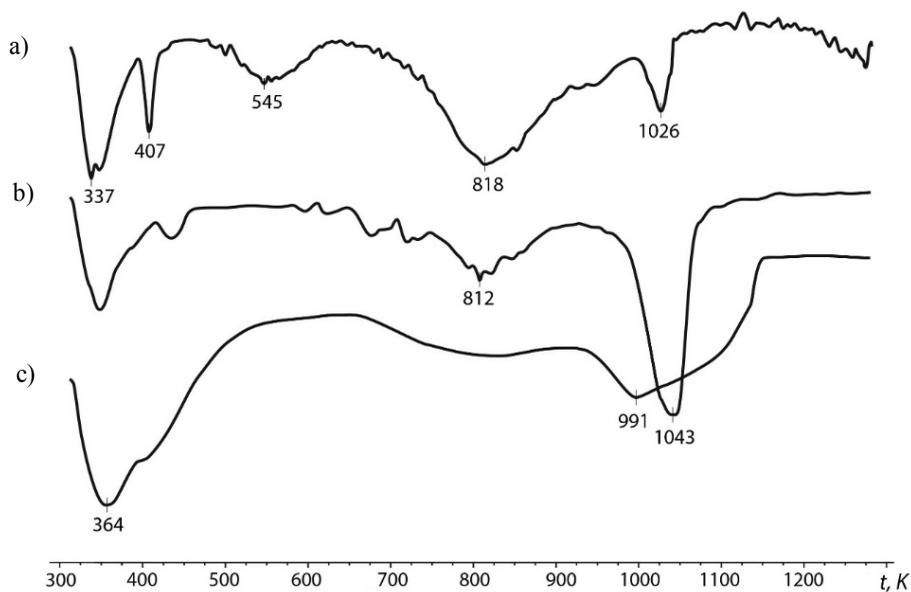


Fig. 2. DTG curves of clay rocks: a – shale, b – argillite, c – clay

According to DTA data (Fig. 2), endothermic regions within 373–423 K associated with the loss of mechanically bound water are noted on all curves. In the temperature range of 923–1123 K, endo-effects are noted, which are presumably associated with the destruction of the crystal lattice of clay minerals, and their transformation into an amorphous reactive phase (formation of metakaolin). On this basis, the temperature of 973 K was chosen for the thermal treatment of clay rocks.

As a result of our research, geopolymer binders of high strength from 80 to 100 MPa were obtained based on thermally modified local clay rocks and granulated blast-furnace slag using an alkaline activator of various compositions.

The compositions of geopolymer binders and the results of their physical and mechanical testing are presented in Table 2.

As shown by the test results (Table 2), after heat treatment (353 K for 20 hours), the strength of geopolymer binders significantly increases compared to binders that have hardened under normal conditions. Heat treatment accelerates the transition of gel-like products of

hydration of geopolymers to the crystalline phase; with increasing temperature, the solubility of Si^{4+} , Al^{3+} , and Ca^{2+} ions increases and, accordingly, the rate of pozzolanic and geopolymer reactions increases.²¹

Fig. 3 and Fig. 4 show the micrographs obtained using SEM measurements by means of reflected (BEC) as well as secondary (SEI) electrons for geopolymer binders No.6 and No.11 (Table 2), which had the best strength indicators. As can be seen in the figures, a heat treatment promotes the compaction of the material, which has a positive effect on its mechanical strength.

Thus, the possibility of obtaining geopolymer binders (with high physical and mechanical characteristics) based on thermally modified local clay rocks, granulated blast-furnace slag, and an alkaline activator of various compositions has been proved.

The assessment of the corrosion resistance of geopolymer materials was carried out according to the change in the mass and strength of the samples after 180 days of being immersed in aggressive solutions, which were 2 % and 5 % solutions of H_2SO_4 and HCl , as well as a 5 % solution of Na_2SO_4 .

Table 2. Compositions of geopolymer binders and the results of their physical and mechanical testing

No.	Components, %		Alkaline component, (dry matter above 100 %), %	Compressive strength after 28 days of curing depending on curing conditions, kg/cm^2			Compressive strength after heat treatment, kg/cm^2
				Air	Water	Air-wet	
1	Slag (80)	* Shale (20)	NaOH (10)	410	452	440	690
2	Slag (80)	* Shale (20)	Na_2CO_3 (10)	210	245	240	537
3	Slag (80)	* Shale (20)	Na_2SiO_3 (10)	187	334	212	488
4	Slag (80)	*Argillite (20)	NaOH (10)	469	480	418	695
5	Slag (80)	*Argillite (20)	Na_2CO_3 (10)	335	420	390	685
6	Slag (80)	*Argillite (20)	NaOH (2.5) + Na_2SiO_3 (7)	536	472	450	856
7	Slag (80)	* Clay (20)	NaOH (10)	460	510	478	630
8	Slag (80)	* Clay (20)	Na_2CO_3 (10)	175	223	217	575
9	Slag (80)	* Clay (20)	Na_2SiO_3 (10)	75	88	85	150
10	Slag (80)	* Clay (20)	NaOH (4) + Na_2CO_3 (6)	215	254	230	266
11	Slag (80)	* Clay (20)	NaOH (4) + Na_2SiO_3 (10)	850	940	935	1025
12	Slag (80)	* Clay (20)	Na_2CO_3 (4) + Na_2SiO_3 (10)	112	145	156	320

* Clay rocks modified at 973 K.

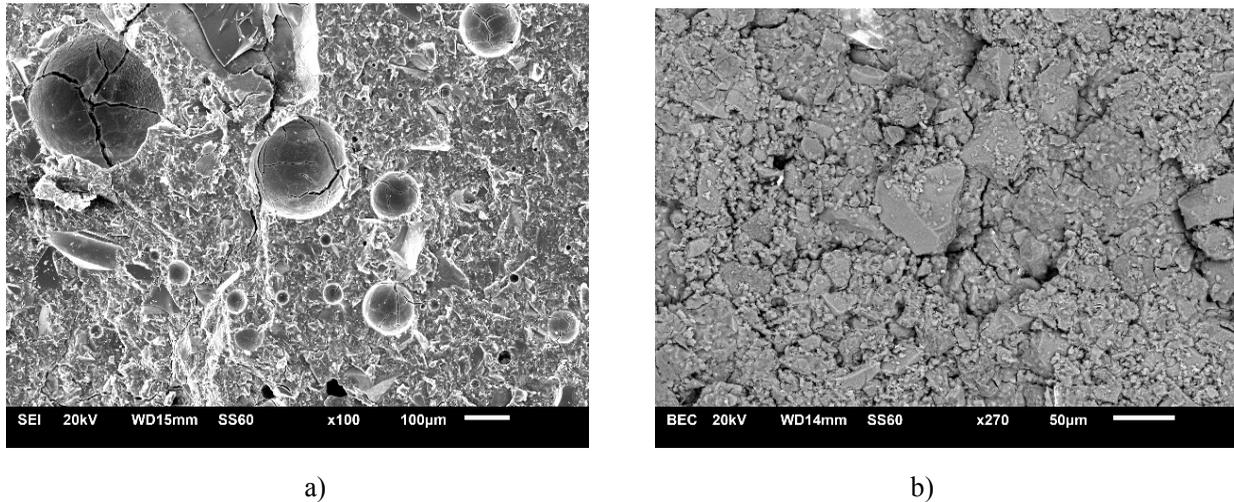


Fig. 3. SEM images of geopolymer binder No.6: a – after 28 days of curing under normal conditions, b – after heat treatment (353 K for 20 hours)

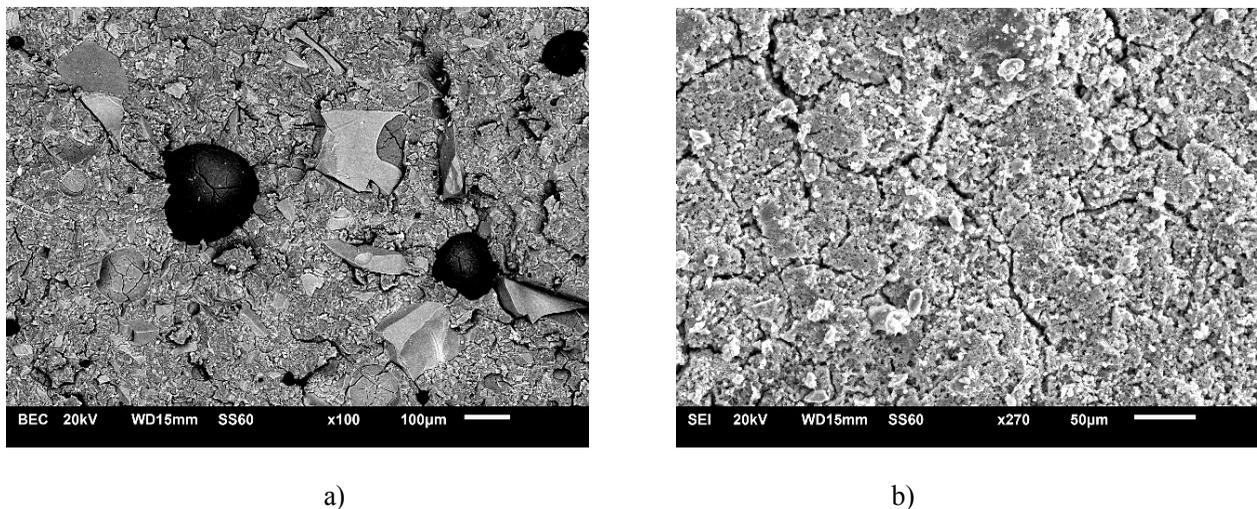


Fig. 4. SEM images of geopolymer binder No.11: a – after 28 days of curing under normal conditions, b – after heat treatment (353 K for 20 hours)

For comparison, Ordinary Portland Cement (OPC) was tested in the same aggressive solutions.

As shown by the test results (Table 3), geopolymer materials have a higher acid resistance and sulfate resistance compared to OPC, which is in good agreement with the data of other studies.²²⁻²⁴ According to the authors,²¹ the high corrosion resistance of geopolymer materials is explained by the absence of $\text{Ca}(\text{OH})_2$ in their composition, a compound that is the main cause of the destruction of Portland cement concrete.

Composition No. 27 showed lower results in corrosion resistance, which is obviously due to the fact that this composition was not subjected to heat treatment. It is known that the heat treatment of geopolymers accelerates the processes of polymerization and pozzo-

lanization,^{19,20} which increases their resistance to aggressive solutions.

Compositions No.6 and No.7 were also characterized by a lower mechanical strength and corrosion resistance since they do not include a clay component – a source of metakaolin, which provides the geopolymer structure with a high degree of polymerization.

According to the authors,^{22,25,26} the corrosion resistance of geopolymer materials is also due to the fact that there are no high-base calcium hydroaluminates in their hardening products that cause sulfate corrosion, and there is also no free lime, leaching of which occurs in soft waters. Therefore, geopolymer concretes are superior in corrosion resistance even to sulfate-resistant concretes based on Portland cement.

Table 3. Compositions of geopolymer materials and the results of their corrosion resistance

No.	Composition of the geopolymer, (%)		Alkaline activator composition, (%)	Weight loss of samples (%) after 180 days immersion in solution						Strength before testing, MPa	Loss of strength of samples (%) after 180 days immersion in solution						
				H ₂ SO ₄		HCl		Na ₂ SO ₄			H ₂ SO ₄		HCl		Na ₂ SO ₄		
				2%	5%	2%	5%	2%	5%	2%	5%	2%	5%	2%	5%	2%	5%
□ 11	Slag 80	Clay 20	NaOH (4) + Na ₂ SiO ₃ (10)	1.49	2.17	2.67	4.77	0.62	0.62	97.5	6	8	9	11	1.8		
□ 27	Slag (80)	Argillite (20)	NaOH (4) + Na ₂ SiO ₃ (10)	0.25	1.32	0.15	3.91	0.63	0.63	92.5	7	11	10	15	1.9		
□ 27	Slag (80)	Argillite (20)	NaOH (4) + Na ₂ SiO ₃ (10)	7.34	9.50	4.53	12.9	0.64	0.64	65.0	12	25	17	29	2.6		
□ 15	Slag (80)	Argillite (20)	Na ₂ CO ₃ (15)	1.15	2.49	1.72	5.5	0.22	0.22	88.0	7	12	11	17	1.5		
□ 25	Slag (80)	Argillite (20)	Na ₂ CO ₃ (7)	1.38	2.92	0.75	5.88	0.87	0.87	82.0	8	11	10	17	2.1		
□ 6	Slag (100)	-	Na ₂ SiO ₃ (10)	4.53	6.97	5.34	9.94	2.29	2.29	77.0	15	26	24	37	3.3		
□ 7	Slag (100)	-	NaOH (4) + Na ₂ SiO ₃ (10)	5.81	9.18	3.22	9.06	2.53	2.53	75.0	19	23	22	31	4.2		
	Ordinary Portland Cement (OPC)			18.11	□ □ □ □	27.7	□ □ □ □	32.8	□ □ □ □	73.0	62	□ □ □ □	70	□ □ □ □	80		

□ Curing mode: heat treatment at 353 K for 20 hours.

□ Curing mode: in the air at 293 K for 28 days.

□ Samples collapsed

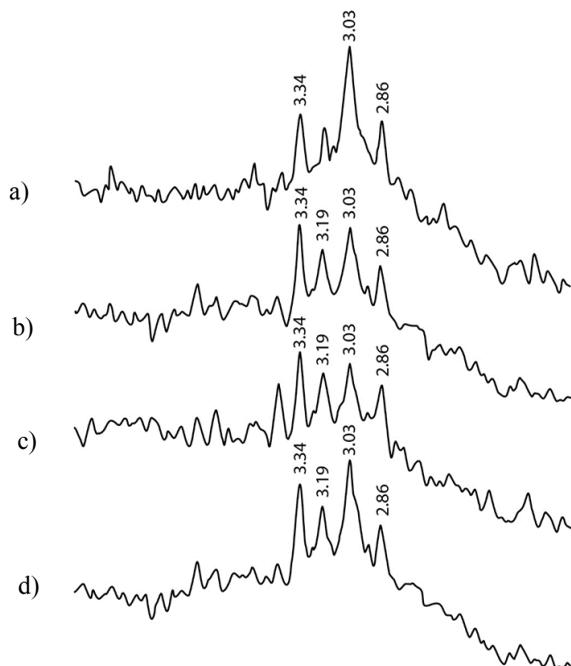


Fig. 5. X-ray patterns of sample No. 27: a – before immersion in an aggressive solution; b – after 180 days of immersion in a 5 % H_2SO_4 solution; c – after 180 days of immersion in a 5 % HCl solution; d – after 180 days of immersion in a 5 % Na_2SO_4 solution

According to the data,^{22,23} if metakaolin is mixed with a certain amount of NaOH solution or $\text{NaOH} + \text{Na}_2\text{SiO}_3$ solution, and then cured at temperatures below 100°C , it is possible to obtain a solid substance having a lattice structure of aluminosilicate resembling a zeolite material having the high mechanical strength. This is what happens when geopolymers are hardened according to the curing mode: heat treatment at 353 K for 20 hours (Table 3 – compositions № 11, 27, 15, 25).

Composition No. 27 was chosen for X-ray phase analysis; it was synthesized from 80 % slag and 20 % argillite (Table 3) and activated with a solution of $\text{NaOH} + \text{Na}_2\text{SiO}_3$. X-ray patterns are shown in Fig. 5.

The X-ray diffraction patterns of the geopolymer material demonstrate the process when the main phases of the material before being immersed in an aggressive solution (Fig. 5a) are the following: X-ray amorphous phase and diffraction lines of quartz (3.33 Å), feldspar (3.19 Å), calcite (3.03 Å) and slag (2.86 Å).

After 180 days of immersing the material in a 5 % H_2SO_4 solution (Fig. 5b), the phase composition remains the same, but the amount of the X-ray amorphous phase decreases, as does the intensity of the diffraction lines of calcite (3.03 Å). The same picture is observed after immersion of the material in a 5 % HCl solution: a decrease in the amount of the X-ray amorphous phase and calcite (Fig. 5c). Different process is observed when the material is immersed in the 5 % Na_2SO_4 solution

(Fig. 5d). In this case, the phase composition and phase ratios remain almost on par with the initial material.

The durability of the geopolymer materials was determined using the composition: slag (80 %) + argillite (20 %). The composition of the alkaline activator is $\text{NaOH} + \text{Na}_2\text{SiO}_3$. The results of tests for strength during storage in the air are given in Table 4. As the results show, the dynamics of changes in strength over time show stable indicators.

Table 4. Dynamics of changes in the strength of GPM over time

Compressive strength, MPa				
The original	After 30 days	After 90 days	After 180 days	After 360 days
92.5	95.1	89.7	93.6	91.9

As our studies have shown, geopolymer materials obtained from local raw materials have high mechanical strength, good durability, and high resistance to aggressive solutions, which is in good agreement with the data of other researchers we cited.

Geopolymers are environmentally friendly materials, and the technology for their production belongs to green technologies.

Geopolymer materials (GPM) can become an alternative to Portland cement (OPC) as carbon emissions are reduced by about 80 %. In addition, given that geopolymer concretes are generally stronger and more durable, they offer a longer service life, which reduces the need for raw materials in the future.

4. Conclusions

1. Geopolymer materials (GPM) with high mechanical strength based on thermally modified clay rocks of Georgia and metallurgical granulated blast-furnace slag were obtained.

2. Mode of temperature modification of clay rocks – heating the material to a temperature of 973 K with the exposure at the maximum temperature for 1 hour.

3. An alkaline activator – NaOH , Na_2CO_3 , Na_2SiO_3 or their mixture can be used as a mixing liquid.

4. After heat treatment (353 K for 20 hours), the mechanical strength of the GPM is significantly increased.

5. The indicators of corrosion resistance of GPM (change in mass and strength) after being immersed in various aggressive solutions correspond to changes in their phase compositions.

6. Corrosion resistance of GPM depends on the composition of the aggressive solution: a) the higher the concentration of the aggressive solution, the greater the loss of mass and strength; b) they are less stable in HCl solutions than in H_2SO_4 , and rather stable in Na_2SO_4 solution.

7. Corrosion resistance of GPM is always higher than OPC under the same conditions of aggression.

8. GPM synthesized on the basis of thermally modified clay rocks has good durability: the dynamics of changes in strength over time show stable indicators.

Acknowledgments

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СИНТЕЗ І ДОСЛІДЖЕННЯ ВЛАСТИВОСТЕЙ ГЕОПОЛІМЕРНИХ МАТЕРІАЛІВ, ОДЕРЖАНИХ З ВИКОРИСТАННЯМ МІСЦЕВОЇ ПРИРОДНОЇ СИРОВИНИ ТА ПРОМИСЛОВИХ ВІДХОДІВ

Анотація. Описано синтез геополімерних матеріалів з використанням термомодифікованих глинистих порід Грузії та металургійного гранульованого доменного шлаку, а також дослідження їхніх фізико-механічних властивостей, довговічності та корозійної стійкості в агресивних розчинах. Використано методи хімічного, рентгенофазового, диференційно-термічного аналізу та сканувальної електронної мікроскопії.

Ключові слова: геополімер, термічно модифікована глиниста порода, довговічність, агресивний розчин, корозійна стійкість.