



INFLUENCE OF THE ACIDIC ENVIRONMENT ON THE PROPERTIES OF METAHALLOYSITE GEOPOLYMER COMPOSITES

WPŁYW ŚRODOWISKA KWAŚNEGO NA WŁAŚCIWOŚCI KOMPOZYTÓW GEOPOLIMEROWYCH Z METAHALOIZYTU

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Abstract

Geopolymers have been shown to exhibit a significantly higher degree of resistance to corrosive environments when compared with cement concrete. The present paper expounds on the impact of sulphuric, hydrochloric and acetic acid solutions on the durability of mortars with geopolymer binders composed of metahalloysite and alkali activators. An activator with sodium water glass to NaOH solution ratios of 1, 2 and 3 and NaOH solution concentrations of 4, 8 and 12 mol/dm³ was used. It was found that when increasing sodium water glass content from 1 to 3 in relation to the 8M or 12M NaOH solution in the activator, a significant reduction in the compressive strength of the mortar with this geopolymer binder was obtained after 28 days of exposure to the acid solutions. A smaller decrease in strength occurred with the acetic acid solution than with the sulphuric or hydrochloric acid solutions.

Keywords: geopolymer composite, metahalloysite, acid resistance, sodium activator.

Streszczenie

Geopolimery w porównaniu do betonu cementowego charakteryzują się wysoką odpornością na większość agresywnych środowisk korozyjnych. W artykule przedstawiono wpływ roztworów kwasu siarkowego, solnego i octowego na trwałość zaprawy ze spoiwem geopolimerowym z metahaloizytu oraz aktywatora alkalicznego. Zastosowano aktywator o stosunku szkła wodnego sodowego do roztworu NaOH wynoszącym 1, 2 i 3 oraz stężeniu roztworu NaOH 4, 8 i 12 mol/dm³. Stwierdzono, że przy zwiększeniu zawartości szkła wodnego od 1 do 3 w stosunku do roztworu 8M lub 12M NaOH w aktywatorze uzyskuje się znaczne zmniejszenie wytrzymałości na ściskanie zaprawy z tym spoiwem geopolimerowym po 28 dniach działania roztworów kwasów. Mniejszy spadek wytrzymałości występował w przypadku roztworu kwasu octowego niż siarkowego lub solnego.

Słowa kluczowe: kompozyt geopolimerowy, metahaloizyt, kwasoodporność, aktywator sodowy.

1. INTRODUCTION

The durability of materials is important factor in the lifespan of a building. Buildings must be constructed in a way that ensures sufficient structural strength and resistance to environmental factors. In comparison to

conventional building materials, geopolymers exhibit enhanced resistance to a wide range of aggressive corrosive environments [1] and demonstrate superior durability in acidic environments when compared to cement concrete [2-4].

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Geopolymers consist of three-dimensional aluminosilicate frameworks composed of $[\text{SiO}_4]^{4-}$ and $[\text{AlO}_4]^{5-}$ tetrahedra connected by oxygen atoms, with alkali cations balancing the charge [5]. Inorganic aluminosilicate polymers show very good acid resistance, as both the Si-O bond and the Al-O bond in the geopolymer network structure hardly react with acid at room temperature. The decomposition rate of geopolymer in a 5% sulphuric acid solution is only one-thirteenth of that of hardened Portland cement slurry, and the decomposition rate in 5% hydrochloric acid is only one-twelfth of that of Portland cement slurry [6].

Geopolymers, due to their high durability, low shrinkage during drying and high adhesion to the substrate, can be used as a coating to provide corrosion protection for other materials that are less durable under these conditions [7]. The findings demonstrated that coating cement concrete with the application of geopolymer mortar resulted in a substantial decrease in the rate of mass loss of the specimens. Cement concrete placed in 10% inorganic acid solutions (HCl , HNO_3) reduced its mass by about 70%, while when it was covered with a 2 mm layer of geopolymer mortar there was a mass loss of only up to 1% after 28 days of exposure to inorganic acids, and for organic acids (CH_3COOH , $\text{C}_3\text{H}_6\text{O}_3$) the mass loss was no greater than 2% [1]. A similar value for the loss of cement mortars after immersion in sulphuric acid of 68% for 6 months was observed by Vafaei M. and Allahverdi [8], while for geopolymer mortars the mass loss was 32% after immersion in a 5% hydrochloric acid solution. Geopolymer mortars contained a significantly lower amount of calcium compounds when compared to cement mortars. The reaction of calcium compounds with sulphuric acid has been shown to result in the formation of gypsum crystals. This, in turn, has been demonstrated to cause internal stresses, which can lead to a range of undesirable outcomes, including cracking, spalling and accelerated material deterioration [8]. An expansive degradation mechanism can also occur through the formation of ettringite ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$) [9].

Geopolymer materials manufactured from class F fly ash, which contains low calcium content (3-4% CaO) exhibit enhanced durability in acidic environments [10]. Ganesan et al. [11] conducted a study on the impact of sulphuric acid 3% solution (H_2SO_4) and sodium sulphate 3% solution (Na_2SO_4) on fly ash geopolymer activated with 10M NaOH and sodium silicate solution with steel fibre. Samples that

had been subjected to curing for 28 days and then placed in solutions for a further 180 days exhibited minimal change in appearance. The maximum mass loss of the geopolymer was 2.2%, and the reduction in compressive strength was 20%. By contrast, for the cement concrete specimens, the mass loss was 27 per cent, and the strength decreased by 41%.

Alibitar [3] tested the effects of a solution of 5% sodium chloride, 5% sodium sulphate and magnesium sulphate and 5% sulphuric acid on geopolymer concrete made from class F fly ash and granulated lead smelting slag activated with sodium silicate and 14M NaOH at a ratio of 1.5. The geopolymer concrete demonstrated enhanced durability in comparison to Portland cement concrete when subjected to the same solutions. Following a nine-month exposure period, the geopolymer demonstrated the least resistance to sodium sulphate solution, a phenomenon attributed to sodium hydroxide leaching from the solution. The author recommends the utilisation of an alternative activator in order to enhance the durability of the material when exposed to sodium sulphate.

Kwasny et al. [4] investigated the resistance of a 28-day cured kaolinite-based geopolymer to sodium and magnesium sulphate solutions, as well as sulphuric and hydrochloric acid. The microstructure of the geopolymer was not affected during the sulphate exposure, while samples immersed in a 1% to 5% sulphuric acid solution showed greater mass loss (a maximum of about 8% after 8 weeks of immersion) than samples placed in hydrochloric acid with concentrations ranging from 0.37% to 1.86% (a loss of about 5%).

Mehta and Siddique [12] found that a fly ash-based geopolymer with up to 30% Portland cement, immersed in a 2% sulphuric acid solution for 356 days, exhibited the highest mass loss for a 30% Portland cement content. An increase in the calcium hydroxide content of Portland cement results in the formation of a geopolymer with reduced resistance to sulphuric acid, due to the formation of calcium sulphate.

Deb et al. [13], after adding optimally 2% nanosilica to a fly ash geopolymer activated with a sodium silicate solution and 8M NaOH , obtained a material with a more compact microstructure and higher resistance to a 3% sulfuric acid solution. The mass loss after 90 days of immersion decreased from 6.0% to 1.9%, and a significant reduction in compressive strength loss was observed. In addition, Elyamany et al. [14] conducted a study on the durability of geopolymer made from slag, fly ash and with silica dust added. The geopolymer concrete, manufactured

from blast furnace slag, demonstrated the highest level of resistance to sulphuric acid.

Sata et al. [15] conducted a study on the durability of geopolymer mortars composed of fly ash and lignite ash, which were activated with sodium silicate in conjunction with 10M NaOH in 3% sulphuric acid solution and 5% sodium sulphate solution. The geopolymer mortars exhibited reduced mass loss and augmented compressive strength in comparison to the Portland cement samples. In mortars where the aluminosilicate was more finely ground, higher strengths were observed. However, greater mass loss under solution was exhibited by these mortars in comparison to geopolymer mortars made from coarser-grained ash.

In the study by Ribeiro et al. [16], the resistance of a metakaolinite geopolymer reinforced with bamboo fibre to sulphuric and hydrochloric acids at concentrations of up to 15% over a period of 112 days was investigated. The findings indicated that the geopolymer exhibited adequate stability and durability when exposed to these corrosive substances, making it a promising material for structural and drainage applications subjected to such environments.

The objective of this study was to examine the impact of 5% hydrochloric acid, sulphuric acid and acetic acid solutions on the mechanical properties of metahalloysite geopolymer mortars. The geopolymer mortar contained an activator with a ratio of sodium water glass to NaOH solution of 1, 2, 3 and sodium hydroxide solution of 4M, 8M and 12M. To date, there is a paucity of information regarding the results of studies of the effect of the activator composition on the physico-mechanical properties of the metahalloysite geopolymer in an acidic environment.

2. MATERIALS AND METHODS

2.1. Materials

Halloysite in powder form with a grain size of 0-100 μm came from the Dunino mine near Legnica, Poland. Metahalloysite obtained by roasting halloysite for 2 hours at 750°C in a muffle furnace was used to produce the geopolymer. The specific surface area of metahalloysite determined by the BET method was 44.64 m^2/g . The chemical composition of halloysite determined by X-ray fluorescence (XRF) has been shown in Table 1.

Table 1. Chemical composition of halloysite

Component	SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	MgO	Mn ₂ O ₃	Na ₂ O	P ₂ O ₅	TiO ₂	LOI*
Content, %	24.17	0.89	19.49	28.63	0.09	3.74	0.29	0.13	0.83	5.18	16.41

* – roasting losses performed for 1 h at 1050°C

Geopolymer mortars were made from EN 196-1 standard sand with a granulation <4 mm and a binder containing metahalloysite with a room-temperature activator that is a mixture of a sodium silicate solution with a molar modulus of SiO₂/Na₂O in the range 2.4-2.6 with a sodium hydroxide solution of 4, 8 and 12 mol/dm³, with a mass ratio of 1 to 3. The weight ratio of sand to metahalloysite was 3, and the ratio of activator to metahalloysite was 0.93. The dry mortar components were mixed for a duration of 10 seconds, then the activator solution was added, and the whole was stirred for 3 minutes. The geopolymer mortar was subjected to a process of compaction on a vibrating table for a duration of one minute. Thereafter, the mortar was transferred into moulds with dimensions of 40 × 40 × 160 mm. The moulds were then covered with plastic sheeting. After 24 hours it was unmoulded and the specimen was stored for a period of 28 days at a temperature of 20°C ±2°C.

2.2. Methods

Compressive strength tests were carried out on a 40 × 40 × 160 mm beam half (compressed area 40 × 40 mm) using a Hydraulic press after 28 days of curing at an ambient temperature of 20°C ±2°C in a sealed container and after a further 28 days of immersion in 5% acid solutions. The rate of force build-up for the compressive strength test was 2400 N/s.

The acid resistance of the mortars was assessed by checking the mass loss after 7, 14 and 28 days for three specimens of each type of composite immersed in 5% solutions of sulphuric, hydrochloric and acetic acid. After 28 days of curing, the mortar bars were soaked in water and placed in acid solutions; after a set time, the specimens were removed from the solution, dried with a damp cloth and weighed before being placed back in their respective solutions.

The specific surface areas of metahalloysite and selected geopolymer mortars after 28 days of curing were determined by BET at a relative pressure p/p_0 in the range 0.05-0.3 using Quantachrome's Autosorb iQ analyser from a nitrogen adsorption isotherm at 77K. Prior to testing, the samples were subjected to a pre-drying process at a temperature of 50°C. The average pore size was determined from the nitrogen adsorption curve at 77K using the density functional

method (DFT). The total porosity of the tested geopolymer mortars was determined in the work [20].

The chemical composition of halloysite was determined on a Bruker S8 TIGER spectrometer.

3. RESULTS AND DISCUSSION

The compositions of the activators in the geopolymer binders are shown in Table 2.

Table 2. Composition of activator in geopolymer binder

Mortar designation	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Molar concentration of NaOH solution [mol/dm ³]	4	8	12	4	8	12	4	8	12
Mass ratio sodium water glass to NaOH solution	1	1	1	2	2	2	3	3	3

Figures 1-3 show the mass loss of geopolymer mortar samples after 7, 14 and 28 days of storage in 5% sulphuric, hydrochloric and acetic acid solutions.

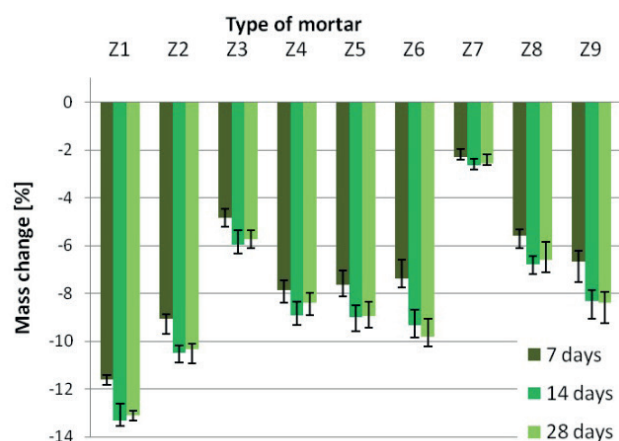


Fig. 1. Geopolymer mortar mass loss in 5% H₂SO₄ solution

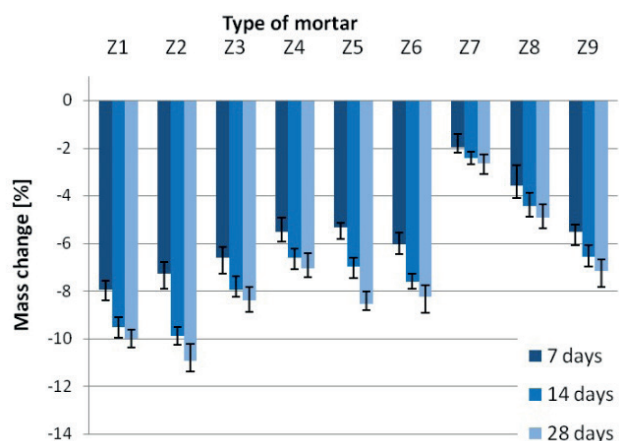


Fig. 2. Geopolymer mortar mass loss in 5 % HCl solution

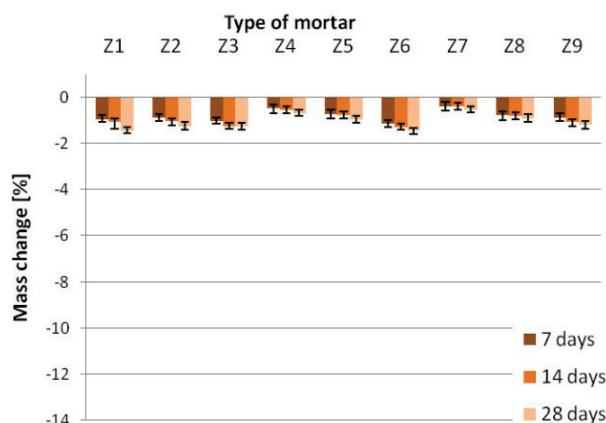


Fig. 3. Geopolymer mortar mass loss in 5% CH₃COOH solution

The most significant mass losses, amounting to several percent (Z1), were observed in mortars stored in sulphuric acid solution, while the least substantial losses were recorded in samples treated with an acetic acid solution of less than 2%. In the present study, geopolymer mortar samples Z7, Z8 and Z9, for which an activator was applied at a ratio of sodium water glass to NaOH solution of 3, were characterised by the lowest mass losses. It was established that an increase in the quantity of sodium silicate solution, from 1 to 3 relative to the NaOH solution of 4, and 8 mol/dm³ in the activator solution, resulted in a decrease in the mass loss of the samples. According to Nguyen et al. [19], the acid resistance of geopolymer concrete is much better because sodium silicate or sodium water glass prevents acid penetration. Different results were shown by Bakharev T. [10] for FA fly ash geopolymer activated with sodium hydroxide obtained lower mass losses compared to samples immersed in sulfuric acid solution, where an activator in the form of sodium silicate solution or NaOH in combination with KOH was used.

Significantly lower losses after 28 days of 0.5% to 1.4% were recorded for samples stored in acetic acid solution than in sulphuric acid solutions, where mass losses ranging from 2.5% to 13.1% were recorded. Similar results were obtained by other researchers. Bakharev [10] for a solution of 5% sulphuric acid for 2 months, the mass loss of the sample was 12.43% and 1.15% when treated with a solution of 5% acetic acid when the fly ash was activated with a NaOH solution. In the study conducted by Kwasny et al. [4], kaolinit clay geopolymer samples were activated using a sodium silicate solution and then curing for 28 days. Following this, the samples were immersed in a 5% sulphuric acid solution for a period of eight weeks.

The results of this experiment demonstrated that there was a mass loss of approximately 8% for the samples of geopolymer that were immersed in solution. In comparison, the samples that were immersed in a 1.86% hydrochloric acid solution experienced a mass loss of around 5%. Similarly, according to Vafaei M. and Allahverdi [8], the failure rate of geopolymer mortar formed from waste glass powder and calcium-aluminium cement activated with a solution of sodium silicate and sodium hydroxide, in sulphuric acid is higher than in hydrochloric acid [8].

Following a 28-day immersion in a 5% sulphuric acid solution, a mass increase was observed in the samples in comparison to the loss that occurred after a 14-day exposure to the acid. Silica gel precipitation was observed in the container. According to Iller [17], in an acidic environment silicic acid can precipitate from silicates that contain unpolymerised SiO_4^{4-} tetrahedra.

An increase in mass and compressive strength of geopolymer exposed to Na_2SO_4 and MgSO_4 concentrations of 3%, 5% and 7% after 180 days was observed by Farhan et al. [18]. The primary cause of the observed mass increase was the accumulation of sulphate particles that resulted in the formation of reaction products, such as gypsum and ettringite, within the voids of the geopolymer. It has been demonstrated that exposure of geopolymer composites to sulphate or seawater results in an enhancement of strength due to the crystallisation of reaction products, leading to a thickening of the microstructure [18].

Figures 4-6 show the results of strength tests on geopolymer mortars stored in 5% sulphuric, hydrochloric and acetic acid solutions and the results of strength tests on geopolymer mortar samples matured for 28 days under laboratory conditions.

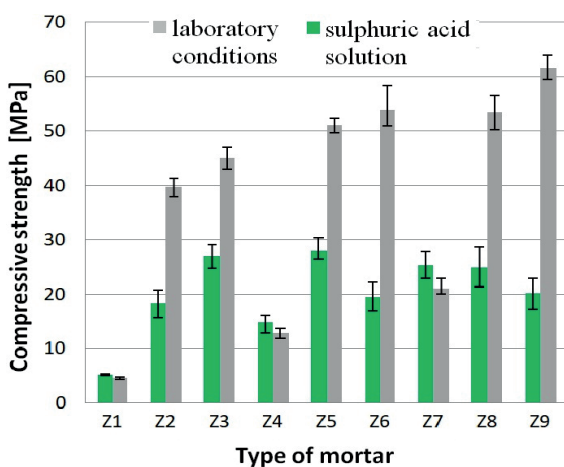


Fig. 4. Change in compressive strength geopolymer mortar in a 5% H_2SO_4 solution

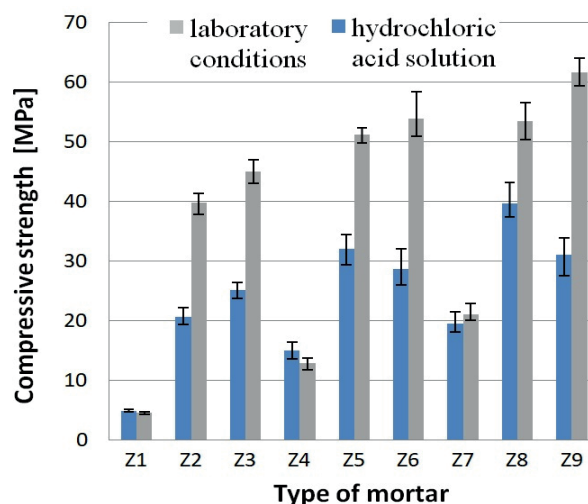


Fig. 5. Change in compressive strength geopolymer mortar in a 5% HCl solution

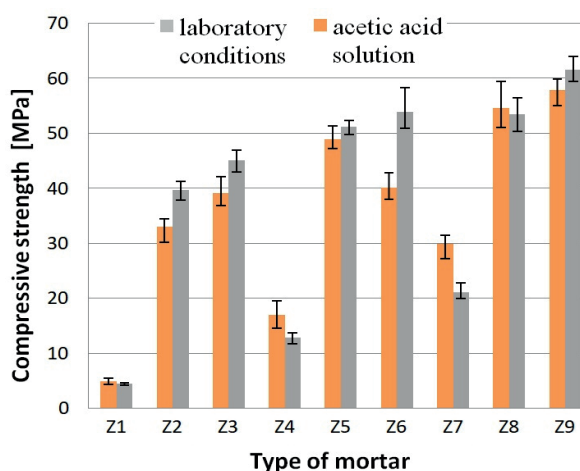


Fig. 6. Change in compressive strength geopolymer mortar in a 5% CH_3COOH solution

The compressive strength of geopolymers placed in a 5% sulphuric acid solution decreased for samples with 8M and 12M sodium hydroxide solution as the activator, while geopolymer samples Z1, Z4, Z7 with 4M NaOH increased their compressive strength by 13.5%, 14.8%, 19.7%, respectively, despite a decrease in sample weight. Similar increases in compressive strength for mortars Z1, Z4 and Z7 occurred when the samples were immersed in hydrochloric and acetic acid solutions. The increased or comparable compressive strength of the specimens stored in acid solutions compared to the reference specimens may have been due to the fact that the reference specimens were tested after 28 days of curing rather than 56 days. The effect of geopolymer binder composition on changes in the strength of the mortars in acidic environments, in turn, requires further research.

In the instance of utilising a 12M NaOH solution within the activator, a marked decline in compressive strength was observed as the sodium silicate solution content increased in comparison to the NaOH solution. Similarly, Bakharev T. [10] observed that geopolymers made from fly ash activated with a 60% sodium hydroxide solution exhibited enhanced durability in a 5% sulphuric acid solution when compared to samples with a sodium silicate solution as an activator. Degradation of geopolymer materials in acidic environments occurs as a result of depolymerisation of aluminosilicate polymers and release of silicic acid [10].

Geopolymer mortars subjected to a 28-day immersion in a 5% hydrochloric acid solution exhibited a decline in strength, exhibiting a comparable trend to that observed in samples stored in a 5% sulphuric acid solution. As posited by Nguyen et al. [19], the residual NaOH resulting from geopolymerisation has the capacity to undergo a reaction with hydrochloric acid, thereby yielding sodium chloride. The formation of NaCl demonstrates the resistance of the geopolymer concrete. Sodium hydroxide has been demonstrated to attenuate the efficacy of hydrochloric acid [19]. In the case of the analysed samples of geopolymer mortars made of metahalloysite, no deterioration in the durability of the material was observed as a result of a 5% HCl solution together with an increased amount of NaOH in the activator solution.

The lowest recorded reduction in compressive strength, ranging from 4.3% to 25.6%, was observed for geopolymers placed in acetic acid solution. In contrast, mortars Z1, Z4, Z7 and Z8 exhibited an increase in strength of 8.4%, 31.8%, 41.8% and 2%, respectively. Geopolymer mortars Z1, Z4 and Z7, which had 4M NaOH solution in the activator, exhibited the lowest 28-day compressive strength and the smallest mass loss of the samples following 28 days of acid immersion.

The stability of geopolymer materials in acidic environments is contingent on the internal ordering of the material structure [10]. The enhanced stability of the cross-linked aluminosilicate polymer structure relative to that of Portland cement specimens is a salient factor contributing to the superior resistance of this material to sulphate solutions and sulphuric acid [15].

Table 3 shows the results of the specific surface area determined by the BET method for selected geopolymer mortars.

Table 3. Results of analysis of nitrogen adsorption in metahalloysite geopolymer mortars

Sample	Specific surface area (BET) [m ² /g]	Average pore diameter [nm]	Total porosity [%]
Z1	13.62	12	24.5
Z3	14.24	7	19.0
Z4	13.82	10	23.3
Z6	22.62	9	21.0
Z7	17.58	16	22.2
Z9	26.27	7	20.6

The geopolymer mortars Z6 and Z9 with the highest specific surface area and small average pore diameter had the highest 28-day compressive strength, but also the highest percentage reduction in strength after 28 days of immersion in 5% acid solutions. As the content of sodium water glass in the activator solution increases, the specific surface area determined by the BET method increases. When the molar concentration in the activator solution is increased from 4 mol/dm³ to 12 mol/dm³, the specific surface area of the geopolymer mortar increases and the average pore size decreases.

The geopolymer mortars Z1, Z4 and Z7, in conjunction with an increase in the content of sodium water glass relative to 4M NaOH in the activator solution, exhibited a higher specific surface area and higher compressive strength after 28 days in sulphuric and acetic acid solution than mortars that had matured for 28 days in air.

It has been demonstrated that a more compact structure with lower porosity exhibits greater resistance to acids. A reduction in contact with the matrix has been shown to result in a decrease in mass loss and compressive strength loss [13]. Bakharev [10] posited that the strength properties of geopolymer materials are contingent on the pore structure of these materials, rather than on the total porosity. The sodium hydroxide-activated geopolymer, despite exhibiting the highest porosity, demonstrated the least compressive strength loss following immersion in a sulphuric acid solution. The author observed that the smallest average pore size of the geopolymer resulted in enhanced resistance to the sulphuric acid solution. In contrast, no such relationship was observed in the case of the analysed geopolymer mortar compositions made of metahalloysite.

4. CONCLUSIONS

The results of the study showed that:

- Geopolymer mortars with a metahalloysite binder that has been activated by a sodium silicate with sodium hydroxide solution demonstrate reduced mass loss when exposed to a 5% acetic acid solution in comparison to placed in sulphuric or hydrochloric acid solutions.
- Increasing the ratio of the sodium silicate solution to the 4M and 8M NaOH solution in the activator from 1 to 3 in the binder results in a decrease in mortar mass loss.
- It was revealed that the compressive strength of geopolymer mortars with a binder containing a 4 mol/dm³ NaOH solution increased after 28 days of exposure to 5% sulphuric, hydrochloric or acetic acid solutions.
- Increasing the ratio of sodium silicate solution to 4M NaOH solution in the activator from 1 to 3 in the binder increases the compressive strength of mortars stored in sulphuric and acetic acid solutions for 28 days compared to mortars stored under laboratory conditions.
- The percentage loss in compressive strength of geopolymer mortars with a binder of metahalloysite with 12M NaOH and a sodium silicate solution immersed in 5% acid solutions for 28 days increases with increasing specific surface area.
- It has been demonstrated that increasing the molar concentration of the sodium hydroxide solution from 4 to 12 mol/dm³ results in an augmentation of the specific surface area of the geopolymer mortar, concomitant with a reduction in the average pore size.
- A geopolymer mortar with a binder of metahalloysite with 12M NaOH and a sodium silicate solution immersed for 28 days in 5% acid solutions characterised by an increased specific surface area

shows reduced compressive strength.

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