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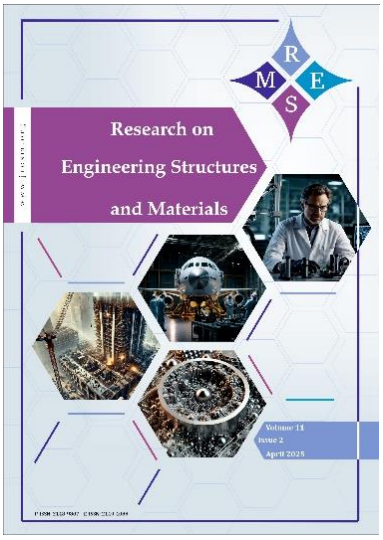

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Research Article

Enhancement of mechanical properties and sulfuric acid resistance in slag-based geopolymer mortars via metakaolin incorporation

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Abstract

This study addresses the impact of metakaolin insertion on the mechanical features and resistance of sulfuric acid of geopolymer mortars composed primarily of ground-granulated blast furnace slag. Several mortar mixtures were created by partially substituting slag with different quantities of metakaolin (0–40%) and activated using a mixture of sodium hydroxide and sodium silicate solutions. Slump flow tests were conducted on the freshly created geopolymer mortar mixtures. After 28 days, capillary water absorption and the voids ratio were determined to assess the physical features of the geopolymer mortars. The mechanical performance of the mortars was measured using flexural strength, ultrasonic pulse velocity (UPV), and compressive strength tests at 28 days, while their durability was evaluated by submerging them in a 5% sulfuric acid solution for 28 days. Weight variations, mechanical assessments, and microstructural analyses were conducted on geopolymer mortars submerged in sulfuric acid to clarify the alterations in the geopolymer matrix. The findings indicated that a 30% substitution of metakaolin augmented flexural strength, compressive strength, and enhanced acid resistance by facilitating the development of a denser and chemically stable aluminosilicate gel. Following 28 days of exposure to sulfuric acid, the compressive strength of 30MK samples increased by 73.05% in comparison to the 0MK samples. This work demonstrates that the use of metakaolin enhances the balance between mechanical features and resistance of acid in slag-based geopolymer specimens, providing a sustainable and resilient alternative to traditional binders.

1. Introduction

Geopolymer composites are binders created by amalgamating amorphous aluminosilicates and natural pozzolans, like red mud, metakaolin, and fly ash, using activators including sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) [1,2]. The geopolymer structure comprises randomly interconnected silicon tetrahedra, with some silicon cations substituted by aluminum. Geopolymers exhibit superior mechanical properties compared to Portland cement, comprising improved thermal resistance, compressive strength, and diminished water absorption, along with beneficial chemical properties, including resistance to acids and the immobilization of hazardous substances [3,4]. Unlike cement, its production does not require sintering at high temperatures procedure, resulting in diminished CO_2 emissions [5].

Recently, geopolymers have garnered heightened interest for their possible application as building materials and eco-friendly binders that may partially substitute standard Portland cement (OPC) [6,7]. Studies demonstrate that geopolymers exhibit enhanced mechanical characteristics [8], including enhanced initial strength and reduced shrinkage [9,10]. Moreover, geopolymers can

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serve as fireproof elements [11] owing to their intrinsic lightweight and thermal insulating characteristics [12,13]. Geopolymer composites can be manufactured using various sources, including byproducts. Metakaolin, slag, kaolin, and fly ash are the traditional basic substances utilized in the manufacture of geopolymer paste, mortar, and concrete [14].

Metakaolin, an exceptionally reactive pozzolanic substance, is a dehydroxylated variant of kaolinite [15]. Metakaolin functions as a valuable admixture, providing multiple benefits, including porosity, enhanced specific surface area, substantial absorbency, and robust coordinative bonding upon activation [16]. The initial response procedure and effectiveness of geopolymer based on metakaolin are impacted by the chemical makeup [17], dosing, and content of raw components [18–20], together with the curing parameters during the initial polymerization phase [21,22]. Xiao et al. [23] evaluated the synergistic effect of seawater and metakaolin on the efficacy of cementitious materials based on phosphogypsum as a sustainable substitute for Ordinary Portland Cement. Their results indicated that metakaolin improved long-term strength by facilitating the synthesis of N-A-S-H gel, providing both mechanical and environmental advantages. In addition, Guang Liang et al. [24] investigated the influence of coal-bearing metakaolin with varying specific surface areas on the sulfuric acid resistance of cement paste. Their results indicated that finer metakaolin markedly decreased porosity and deterioration while augmenting compressive strength and durability by boosting hydration product retention and restricting corrosion in acidic environments.

Alternative binders and additives have been investigated to enhance strength and durability in cementitious and various systems. Karimiazar et al. [25] revealed that the combined application of nano-silica and lime improved the strength and durability of sulfate-laden marl soil by facilitating secondary CSH gel formation and inhibiting ettringite formation. Teshnizi et al. [26] shown that cement kiln dust (CKD), an industrial by-product, enhanced the strength, stiffness, and durability of loess soil via particle aggregation and calcium-based gel formation, providing a sustainable alternative to conventional stabilizers. Karimiazar et al. [27] similarly observed that the treatment of reactive clay with 3% cement and 1% nano-silica or nano-alumina greatly enhanced the soaking CBR, improved the microstructure, and decreased swell potential, obtaining performance akin to greater cement concentrations. Mirzababaei et al. [28] discovered that the incorporation of 3% cement with nano-silica or nano-alumina improved the strength and water resistance of marl, allowing a reduction in cement consumption by augmenting CSH formation and decreasing porosity. Hashemi et al. [29] enhanced the stability of collapsing soils along the Sabzevar-Mashhad railway by using 5–10% lime and cement, noting an improvement in shear strength attributed to increased friction angles, with lime yielding superior compaction and less collapse potential compared to cement. Ezazi et al. [30] undertook experimental and computational investigations on the Chamshir tunnel lining, determining that segments reinforced with steel fibers and fiber-reinforced rebar exhibited superior strength and resistance to damage from TBM jack thrust compared to conventional rebar-reinforced concrete.

Additionally, geopolymers are employed as protective covering materials. Geopolymers offer benefits such as high strength, exceptional mechanical properties, and adjustable setting periods. In addition, geopolymers exhibit remarkable resistance to acid and alkali corrosion, as well as superior high-temperature endurance, characteristics typically lacking in traditional organic concrete protection methods. As a result, alkali-activated metakaolin-based geopolymers may possess considerable promise for applications in concrete surface protection [31,32]. Teshnizi et al. [33] examined the synergistic use of gypsum and rice husk ash for the stabilization of expanding clay soils on forest road subgrades. Their findings indicated enhanced strength, less flexibility and swelling, and the development of CSH and CAH gels, underscoring a sustainable approach to improving road stability in difficult soil conditions.

Portland cement-based materials frequently exhibit inadequate resistance to acid attack, resulting in a reduced service life for exposed structures. Structural deterioration results in various social, environmental, and physical consequences. The material obstacle can be established by placing barrier coatings on the composite surface to decrease the risk of chemical assault, thereby limiting acid infiltration into the concrete microstructure [34,35]. Nonetheless, these methods are costly

and laborious [36]. Although barrier coatings mitigate acid intrusion, they increase expenses and maintenance requirements. Geopolymers have inherent chemical resistance, providing a more cost-effective and resilient option. Recently, geopolymer binders have come to prominence as a feasible remedy to the effects of acid, owing to their microstructure. Extensive studies have been conducted on geopolymers due to their significant advantages in acid resistance. Geopolymers can produce acid-resistant materials appropriate for sewer pipelines, water tanks, and diverse applications in acidic environments. A previous study [37] investigated the acid deterioration process of fly-ash and showed a similar deteriorating using a geopolymer without of calcium. The sole distinction is that the dispersing SO_4^{2-} anions encounter the diffusing ions of calcium, resulting in the precipitation of crystals of gypsum within the infiltrating layer. Teshnizi et al. [38] assessed the impact of acid and temperature exposure on the compressive strength of geopolymer samples formulated from sustainable resources, including slag, zeolite, metakaolin, and Portland cement. The research indicated that slag-based mixes, especially those containing 8 M KOH, demonstrated enhanced resistivity and compressive strength under harsh circumstances, but alterations in alkaline activator concentration markedly affected durability. Shalan and Mohamed M. El-Gohary [39] investigated the influence of the chemical composition of geopolymers on the sulfuric acid resistance and mechanical features of the concrete. Their results indicated that elevated Al/Si and MgO concentrations, especially in conjunction with slag and metakaolin, facilitated the production of hydrotalcite and CASH, decreased porosity, and boosted resistance to acid assault. A further study [40] utilized metakaolin as an interim replacement for fly ash that is high in calcium in geopolymer materials and determined that the materials demonstrated enhanced the durability of acid impact compared to the singular material, attributable to the reduced calcium in the composites. Geopolymer compositions with various binders have varying sulfuric acid resistance, particularly for binders with and without calcium. Consequently, it is essential to evaluate the geopolymers' resistance, formulated with various binders, particularly slag and metakaolin, to sulfuric acid.

Despite increasing interest in alkali-activated materials that include slag and metakaolin, a comprehensive understanding of their performance under sulfuric acid exposure is still insufficient. The majority of the research done so far has only looked at geopolymers made from metakaolin or slag. However, there has been a dearth of research into how these two factors interact to improve mechanical performance, microstructural features, and chemical durability, especially when subjected to harsh environments like acid. Consequently, in this research, varying quantities of metakaolin were incorporated into slag at ambient temperature to construct a two-blend geopolymer mortar. The impact of metakaolin on the mechanical and microstructure characteristics of slag-based geopolymer mortar exposed to sulfuric acid was examined through various tests. Thus, this work focuses on improving the performance of slag-based geopolymers in adverse conditions by using metakaolin to create more durable composites. This fosters ecological and economic alternatives to conventional cement systems.

2. Materials and Experimental Methods

2.1. Materials Specification

This study utilized slag and metakaolin as binders, with their chemical compositions presented in Table 1. The specific gravities of slag and metakaolin were 2.9 and 2.4 g/cm^3, respectively. In addition, NaOH and Na_2SiO_3 served as activators in the geopolymer formulations, with their chemical properties presented in Tables 2 and 3. NaOH was used in the form of solid beads, while Na_2SiO_3 was provided as a commercially available solution. In addition, the fine aggregate utilized during this investigation was standard sand, according to the specifications of EN 196-1 [41]. This sand has a controlled particle size range between 0.08 mm and 2.00 mm. The fineness modulus is 2.70 and the sand was used in dry form to ensure correct mixing ratios. Moreover, sulfuric acid was employed as the acidic medium to examine the acid resistance of geopolymer mortar samples. Sulfuric acid was used at a concentration of 5% in this investigation.

Table 1. Chemical proportions of slag and metakaolin (%)

Materials	L.O.I.*	CaO	Fe_2O_3	MgO	SiO_2	Na_2O	Al_2O_3	K_2O	TiO_2
Slag	0.6	35.58	1.1	6.58	40.55	0.79	13.8	0.4	0.6
Metakaolin	1.17	0.19	0.85	0.16	56.1	0.24	40.23	0.51	0.55

*Loss of Ignition (L.O.I.)

Table 2. The chemical composition of the sodium silicate solution used (%)

SiO_2	Heavy metals	Na_2O	Fe	H_2O
27	≤ 0.005	8.2	≤ 0.005	64.8

Table 3. The chemical composition of sodium hydroxide (%)

Al (%)	Na_2CO_3	Cl (%)	NaOH	SO_4 (%)	Fe (%)
≤ 0.002	0.4	≤ 0.01	99.6	≤ 0.01	≤ 0.002

2.2. Proportions and Fabrication of Geopolymer Specimens

To make a 12M sodium hydroxide solution, 240g of sodium hydroxide was introduced to a 500 mL volumetric flask and was then filled up to the 500 mL mark with distilled water. Prior to formulating the slag-metakaolin geopolymer slurry used in the present study, a 12M sodium hydroxide solution was generated and permitted to cool at 20° C for one day. The sodium hydroxide was subsequently combined with the Na_2SiO_3 solution. Then, metakaolin was replaced with slag at rates of 0, 10, 20, 30, and 40% to form a dry binder mixture with sand. Then, the dry ingredients were mixed in a standard mortar mixer at 100 rpm for 2 min. Subsequently, the activator solution was added to this dry mixture and mixed at 150 rpm for 2 minutes to make it homogeneous. The mixtures were then placed in greased molds measuring 50×50×50mm and 40×40×160mm. The molds were then positioned on a vibrating table and compacted for 1 min at a frequency of 50 Hz to reduce trapped air and provide consistent density. The samples were subsequently extracted from the mold and preserved at 20 ± 2 °C for a duration of 28 days. Table 4 presents the quantities of the mixes utilized in this study.

Table 4. Design of geopolymer blends (kg/m^3)

Mixtures	NaOH	Na_2SiO_3	slag	aggregate	Metakaolin
0Mk	136.83	273.67	586.44	1267.69	0
10Mk	136.83	273.67	527.80	1256.94	58.64
20Mk	136.83	273.67	469.15	1246.19	117.28
30Mk	136.83	273.67	410.51	1236.42	175.93
40Mk	136.83	273.67	351.86	1223.71	234.57

2.3. Performed Tests

This study evaluated slump flow values, water absorption, UPV results, compressive strength, bending strength, mass changes, and visual assessment on geopolymer samples to evaluate the influence of metakaolin on the sulfuric acid resistance and mechanical characteristics of slag-based geopolymer mortars. In addition, scanning electron microscope (SEM) analysis was conducted to examine the microstructural properties of the samples. Slump flow values were evaluated for each mixture containing varying percentages of metakaolin to ascertain the mixture's consistency. The blends were arranged in 2 levels inside a cone apparatus on top, and the levels was tapped with hammer made of wood and copper as shown in Figure 1. After the installation process, the flat surface was thrown twenty-five times due to hand rotation. The cone was then elevated perpendicularly, then the spreading measurements in 2 distinct orientations were evaluated and averaged.

Fig. 1. Slump flow experiment conducted on fresh geopolymer mixtures

The compressive strength test was conducted by applying load to cube specimens of 50×50×50 mm using a pressing machine with a capacity of 250 kN and a loading rate of 0.6 MPa/sec in accordance with ASTM C109 [42] as illustrated in Figure 2 (a). Flexural strength tests were performed on 40 × 40 × 160 mm specimens with a loading rate of 0.469 MPa/sec to determine the flexural strength according to ASTM C348 [43] as presented in Figure 2 (b). These tests were first performed on geopolymer samples before immersion in a sulfuric acid solution. Moreover, a capillary water absorption experiment was carried out on geopolymer samples based on slag and metakaolin in accordance with ASTM C1585 [44]. Geopolymer samples consisting of slag and metakaolin prismatic specimens were subjected to drying for one day at 105°C. The samples' dry weight, subsequent to the application of Vaseline, excluding the surfaces in touch by water, was measured. Then, the dried samples were submerged in 5 mm depth of water. The samples were subsequently extracted from the water, and immersed sample weights (Ww) was recorded at different times. The findings were subsequently created, and the capillary water curves were conducted.

(a) (b)

Fig. 2. (a) Compressive strength and (b) flexural strength experiments conducted on hardened geopolymer samples

Furthermore, the sulfuric acid was diluted to a 5% concentration, and the specimens were replaced in the acid solution for 28 days at 20°C as illustrated in Figure 3. The solution was renewed every 7 days to maintain a constant concentration throughout the exposure period. This approach

ensured that the acidity level remained relatively stable. The compressive strength of the acid-exposed specimens was retested after 14 and 28 days. Similarly, flexural strength testing was performed on the geopolymer specimens after acid exposure at 14 and 28 days. In addition, mass change measurements were also performed on the cubic specimens, and a UPV test was performed on the cubic specimens before and after acid exposure after three, seven, fourteen, and twenty-eight days, following ASTM C597 [45] as presented in Figure 4.

(a) (b)

Fig. 3. Geopolymer samples immersed in sulfuric acid: (a) cubic, (b) prismatic specimens

Fig. 4. UPV experiment conducted on cubic geopolymer samples

Moreover, the specimens were categorized according to their metakaolin replacement ratio by weight to provide clarity in the presentation of the test findings. The sample codes used in the investigation are: 0Mk (0% metakaolin - 100% slag), 10Mk (10% metakaolin - 90% slag), 20Mk (20% metakaolin - 80% slag), 30Mk (30% metakaolin - 70% slag), and 40Mk (40% metakaolin - 60% slag).

3. Results

3.1. Slump Flow Values

Slump flow testing is the primary method and provides an exceptional assessment of workability. Despite their significant restrictions, workability evaluations are utilized for performance assurance and composite specifications. The creation of stable mixes with little slump flow has led to the adoption of flow testing [46]. Figure 5 depicts the median flow rates of metakaolin-substituted slag-based geopolymer mortar specimens. A peak flow of 166.7 mm was obtained from the 0MK geopolymer sample. Increasing the metakaolin concentration in geopolymer samples diminished the flow measurements of slag-based geopolymer mixes. The diameter diminishes were 0.69%, 1.08%, 1.44%, and 5.76% for 10MK, 20MK, 30MK, and 40MK, respectively, relative to the 0MK sample. The slump flow was probably reduced by the large surface area of metakaolin, which increased the water requirement, according to previous research [47,48].

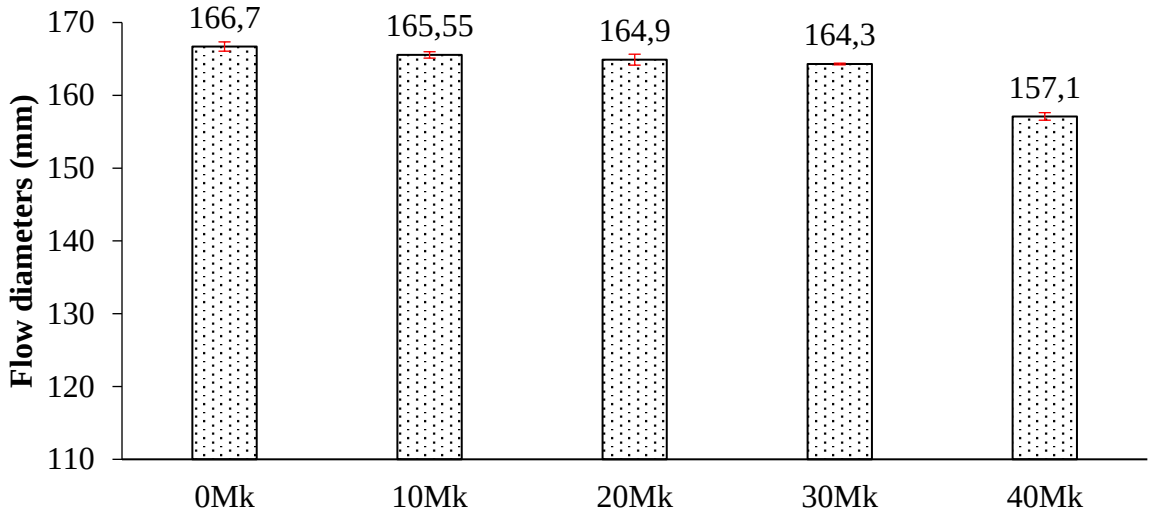


Fig. 5. Slump flow values of metakaolin substituted slag-based geopolymer mortar specimens

3.2. Water Absorption

Experiments for water absorption and capillary water absorption were conducted to evaluate the porosity of the geopolymer mortar specimens. Figure 6 depicts the void ratios and water absorption ratios. Figure 6 demonstrates that the sample containing 40Mk displayed the minimal void ratio, resulting in the smallest water absorption ratio relative to the remaining specimens. The sample void ratios decreased from 0Mk to 40Mk. The result reached is that the 40Mk sample demonstrated the greatest density. In addition, Figure 7 depicts the capillary water absorption curves of geopolymer samples with various metakaolin contents. The water absorption test produced consistent results, with 40Mk demonstrating the minimal water absorption relative to the remaining specimens. The capillary water absorption coefficients (kc) were calculated to enable a more efficient comparison, as seen in Figure 8. The number of voids in the samples decreased as the metakaolin content increased from 0Mk to 40Mk. 4.48%, 19.85%, 29.38%, and 32.63%. Metakaolin, possessing a much greater surface area than slag, improves particle packing and occupies interstitial spaces among bigger slag particles, leading to a denser microstructure and enhanced compactness [49].

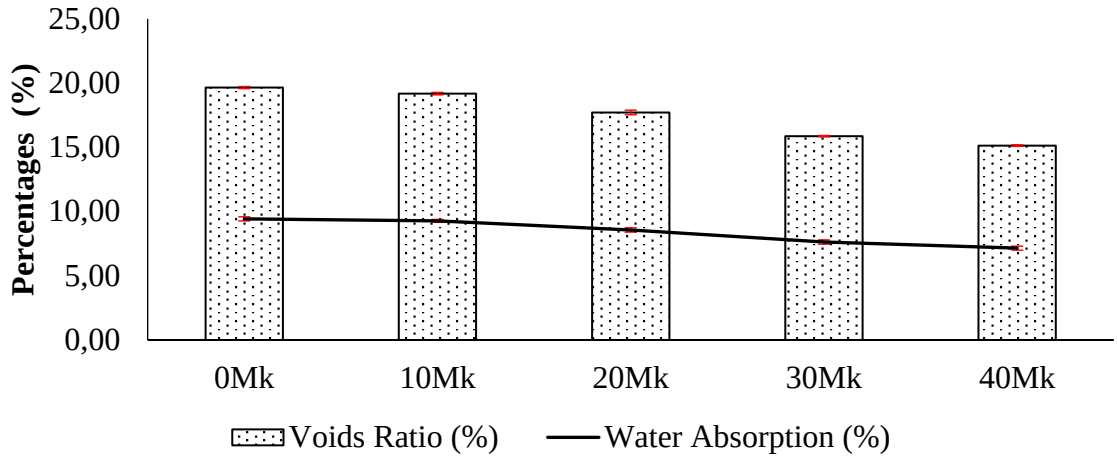


Fig.6. Water absorption and void ratios of slag-based geopolymer samples mixed with different proportions of metakaolin

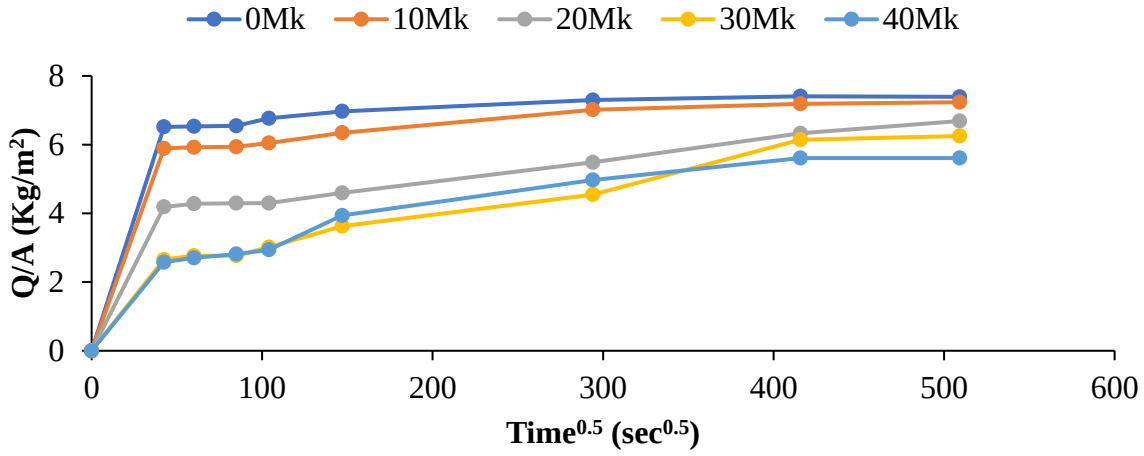


Fig. 7. The amount of water absorbed per square meter of slag-based geopolymer samples with varying metakaolin ratios

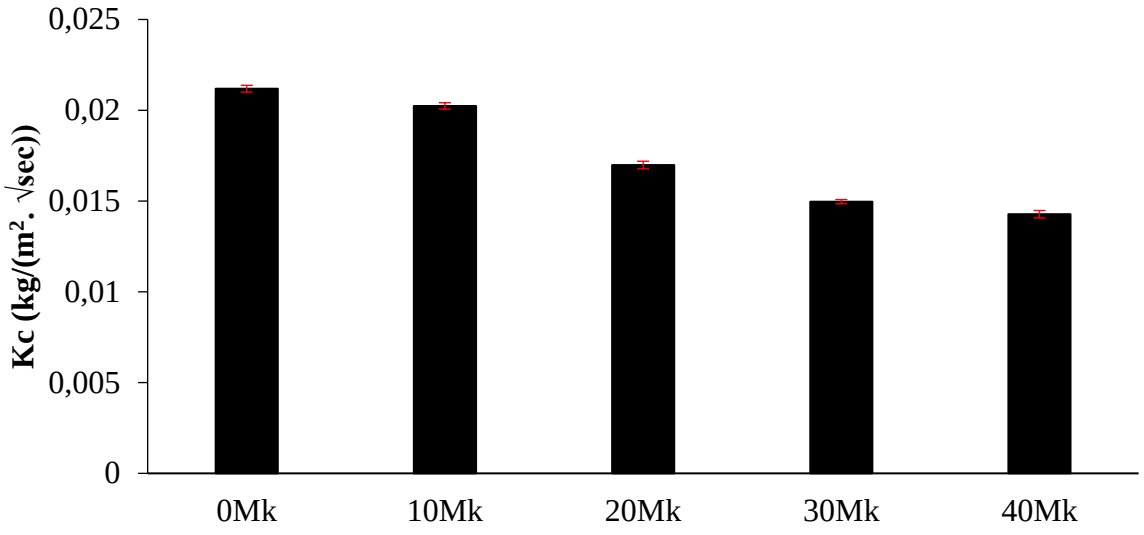


Fig. 8. Capillary water absorption coefficient of slag-based geopolymer cement with differing metakaolin ratios.

3.3. UPV Results

A UPV test was conducted on slag-based geopolymer samples with different proportions of metakaolin. This test sought to assess the impact of varied metakaolin concentrations on the specimens and to examine the consolidation of geopolymer following 28 days with differing metakaolin ratios. Figure 9 present the outcomes of the UPV experiment performed on the samples. A specimen matrix marked with a large number of voids requires a long permeation time, which reduces the permeation rate. The void ratio substantially influences the mechanical characteristics of geopolymer samples derived from slag and metakaolin. The study revealed that the highest UPV value in slag-based geopolymer samples was obtained at a concentration of 30MK after acid exposure. The UPV values of 30MK slag-based geopolymer samples increased by 30.91% compared to 0MK samples after 7 days of acid exposure. In addition, the UPV value of the 30MK sample exposed to acid until the 14th day increased by 18.29% compared to the 30MK sample that was not exposed to acid, while this percentage decreased to 16.06% after the 28th day of acid exposure. The increase in UPV values, especially in the first phase of acid exposure (7 and 14 days) observed in the 30MK samples, suggests that additional densification or filling of micro voids occurred in the geopolymer matrix. This can be explained by the fact that metakaolin forms a more chemically stable and dense aluminosilicate gel, and the N-A-S-H gel integrated with the slag-derived C-A-S-H gel forms a hybrid structure, which is less susceptible to acid-induced weathering and degradation. The 40MK samples had the greatest value prior to treatment with sulfuric acid. The void volume in the geopolymer structure exhibits an inverse correlation with the UPV values [50]. The enhancement is due to the smaller particle size and increased reactivity of metakaolin, which aids in the creation of a more chemically stable and denser aluminosilicate gel. Metakaolin facilitates the formation of N-A-S-H gel, which, when integrated with the C-A-S-H gel from slag, produces a hybrid gel structure that exhibits reduced vulnerability to acid-induced leaching and degradation [51,52]. This dual-gel technology has been shown to enhance durability and tolerance to harsh situations.

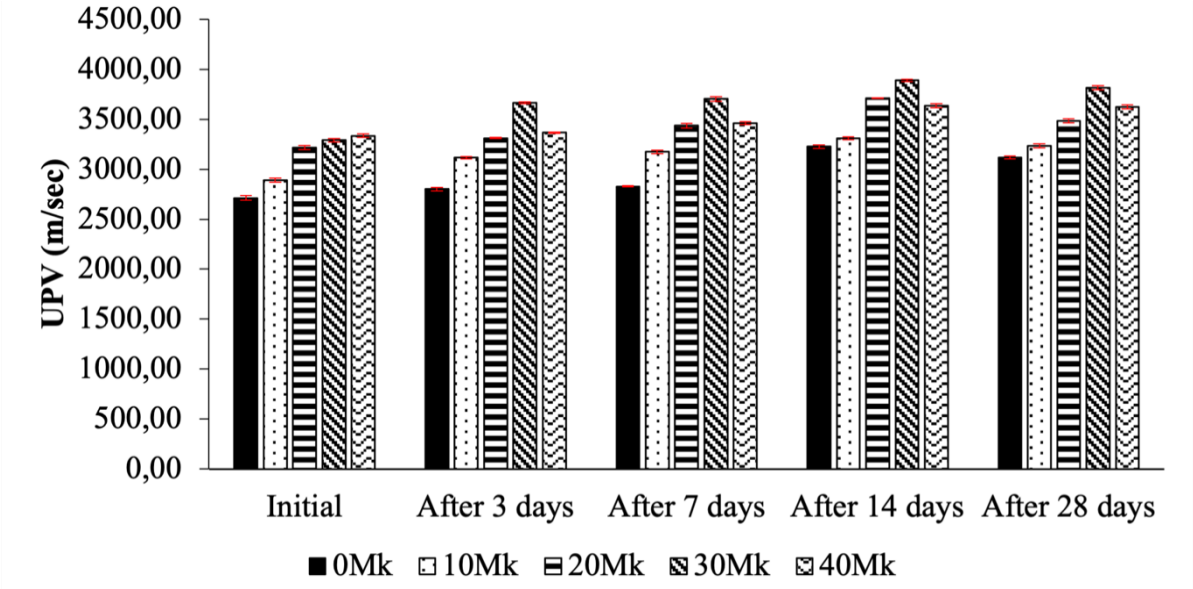


Fig. 9. UPV readings of geopolymer samples with different metakaolin ratios prior to and following exposure to sulfuric acid

3.4. Compressive Strength

This experiment aimed to investigate the mechanical properties of geopolymer samples with varying metakaolin concentrations. Three samples for each metakaolin concentration were examined prior to and following exposure to sulfuric acid for durations of 14 and 28 days. The obtained data were then averaged. Figure 10 shows the compressive strength results for geopolymer samples with varying metakaolin concentrations prior to and following fourteen and twenty-eight days of acid treatment. This work observed that the compressive strength of 40MK

exceeded the compressive strength of 0MK, 10MK, 20MK, and 30MK samples before acid exposure, while the compressive strength of 30MK exceeded the compressive strength of 0MK, 10MK, 20MK, and 40MK samples after 28 days of acid exposure. Despite the 40 MK combination demonstrating the greatest initial compressive strength, its performance declined following exposure to sulfuric acid in comparison to the 30 MK sample. This may result from the increased aluminosilicate content creating a denser gel, which improves initial mechanical strength but also adds more reactive sites susceptible to acid attack. Conversely, the 30 MK formulation seems to provide a superior equilibrium between chemical resilience and mechanical strength. Studies indicate that augmenting metakaolin concentration may elevate the Si/Al ratio; nevertheless, a larger Si/Al ratio does not always enhance acid resistance and may, in fact, diminish it owing to heightened gel depolymerization and dealumination in acidic environments [53]. In metakaolin-rich combinations, the abundant aluminosilicate structure is more vulnerable to sulfuric acid, resulting in increased strength degradation and observable deterioration relative to slag- or fly ash-based systems [54]. The data indicate that best acid resistance is attained not at maximum metakaolin concentration, but at intermediate levels when structural compactness and chemical resilience are more effectively matched.

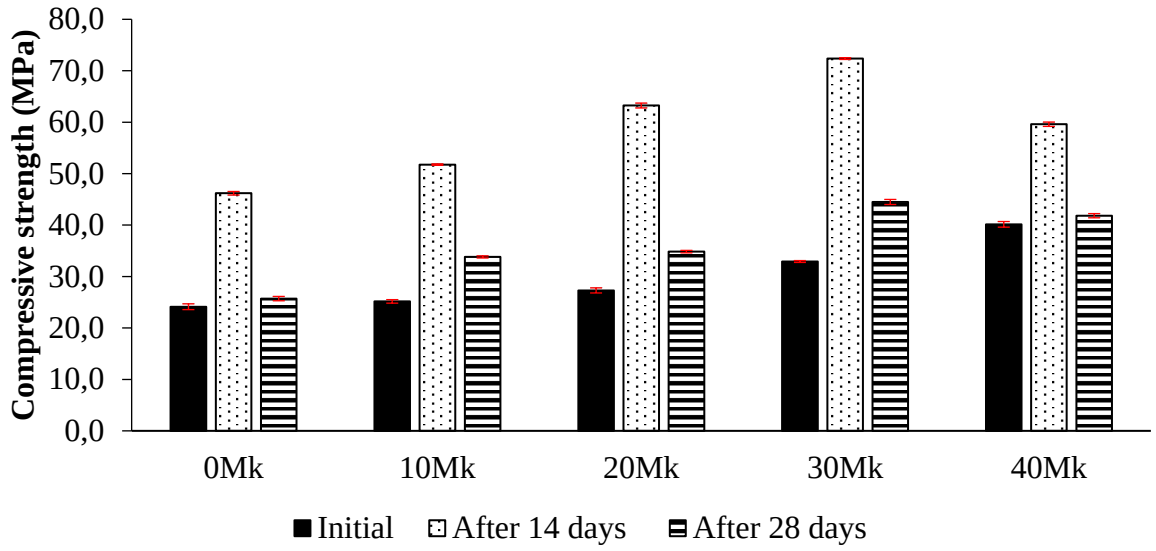


Fig. 10. Compressive strength findings of geopolymer samples with differing metakaolin proportions prior to and following 14 and 28 days of exposure to sulfuric acid

The compressive strength values of the metakaolin samples at 10MK, 20MK, 30MK, and 40MK increased compared to the samples without metakaolin before acid exposure by 4.29%, 13.12%, 36.43%, and 66.37%, respectively. This may be ascribed to the beneficial relationship between the alumina- and silica-rich metakaolin and calcium-rich slag. Metakaolin improves the polymerization process, creating C-A-S-H and N-A-S-H gels simultaneously, providing a denser and mechanically superior matrix [55]. The elevated reactivity and surface area of metakaolin promote a superior pore structure and enhanced particle packing, resulting in increased mechanical efficiency [56]. Following fourteen days of sulfuric acid treatment, the compressive strength values of the metakaolin samples at 10MK, 20MK, 30MK, and 40MK increased by 12.04%, 36.91%, 56.67%, and 29.05%, respectively, compared to the samples without metakaolin. In addition, subsequent to twenty-eight days of immersion in sulfuric acid, the compressive strength values of the 10MK, 20MK, 30MK, and 40MK samples increased by 31.51%, 35.52%, 73.05%, and 62.67%, respectively, compared with the 0MK samples. The reactive aluminosilicate properties of metakaolin form a three-dimensional geopolymer gel network (C-S-H and C-A-S-H), enhancing the density, strength, and mechanical properties [57]. After acid effect, the compression strength of the 30MK boosted by 119.82% following fourteen days compared to its initial strength before acid exposure. Also, after 28 days, the compressive strength of the 30Mk specimen reflected a 35.15% increase relative to strength before immersion in sulfuric acid. Thus, the compressive strength of the samples was

lower after 28 days of exposure to acid compared to the compressive strength after 14 days of exposure to acid. In cementitious concrete and geopolymers, sulfuric acid is believed to induce microstructural deterioration of geopolymer composites [58,59].

3.5. Flexural Strength

This experiment was conducted on geopolymer mortars. Flexural test sought to examine the mechanical properties of the mortar samples with various metakaolin concentrations. Average results were derived from the collected data. Figure 11 shows the strength results of mortar samples with various metakaolin concentrations subsequent to twenty-eight days of immersion in sulfuric acid. The research showed that the bending strength at 40MK concentration was superior to that of the metakaolin samples containing 0MK, 10MK, 20MK, and 30MK before acid exposure, while the flexural strength at 30MK concentration was superior to that of the metakaolin samples containing 0MK, 10MK, 20MK, and 40MK after acid exposure. As a result, the flexural strength enhancements of the 10MK, 20MK, 30MK, and 40MK geopolymer samples before acid treatment were 10.52%, 55.83%, 62.92%, and 90.03%, respectively, compared to the 0MK specimens. Subsequent to fourteen days of acid immersion, the flexural strength values of the 10MK, 20MK, 30MK, and 40MK geopolymer samples increased by 17.45%, 52.87%, 124.46%, and 115.71%, respectively, compared to the 0MK sample. After 28 days of exposure to sulfuric acid, the flexural strength values of geopolymer samples containing 10MK, 20MK, 30MK, and 40MK increased by 21.29%, 66.65%, 106.05%, and 103.18%, respectively, compared to the sample not exposed to 0MK. In addition, in comparison to the flexural strength of 30MK specimens that were not submersed in the acid solution, the flexural strength of 30MK specimens subjected to acid treatment for fourteen days boosted by 41.05%, while this increase decreased to 19.31% after 28 days. Thus, the flexural strength of mortar specimens submersed in sulfuric acid boosted after fourteen days, but this improvement declined significantly following twenty-eight days. This study demonstrated that metakaolin reacted with an alkaline solution to form geopolymer gels (e.g., C-S-H and C-A-S-H). Early exposure to sulfuric acid enhanced the interaction between the raw materials and the geopolymer gel, enhancing its strength for up to 14 days. Following 28 days of exposure to sulfuric acid, the geopolymer bonds began to degrade due to acid ions and the dissolution of silicates and aluminates, resulting in a decrease in their strength. Similar results were observed in a previous study, which indicated that sulfuric acid led to a decrease in strength, which is attributed to the dissolution of the aluminosilicate bonds [60].

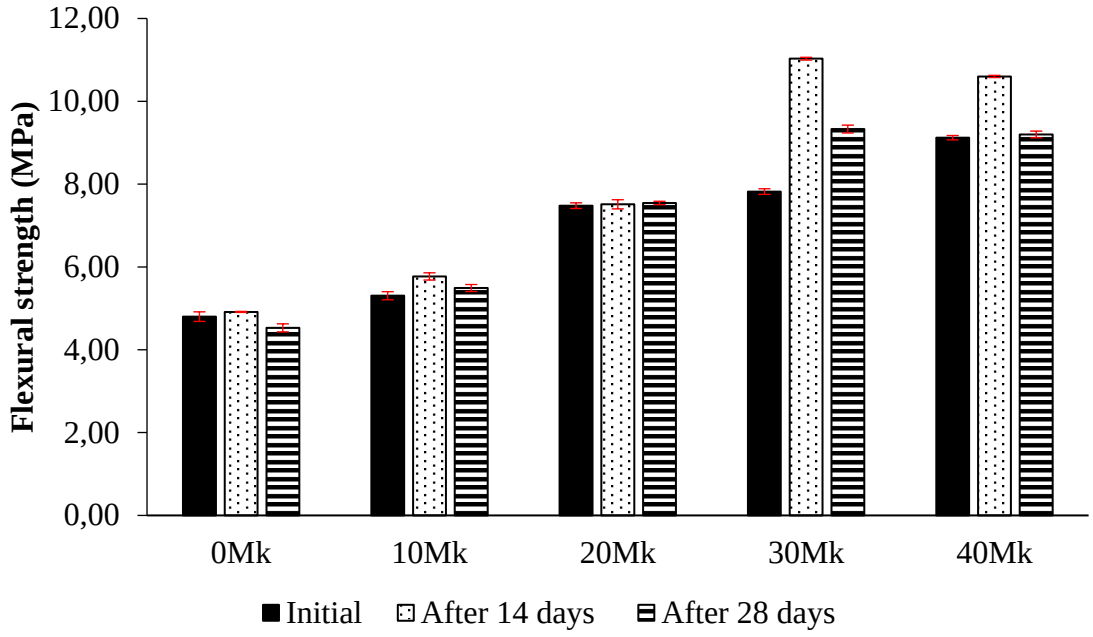


Fig. 11. Flexural strength measurements of geopolymer samples with various metakaolin fractions prior and following sulfuric acid effect

3.6. Mass Changes and Visual Assessment

A mass change test was conducted on materials immersed in 5% sulfuric acid. Figure 12 shows the increased mass change for geopolymer samples with varying metakaolin concentrations exposed to sulfuric acid over periods of 3, 7, 14, and 28 days. The mass of the 0MK geopolymer samples after 3, 7, 14, and 28 days of sulfuric acid exposure increased to 2.36%, 2.79%, 3.016%, and 1.037%, respectively, compared to the samples before exposure. Also, the increases for the 30MK sample were 4.064%, 4.617%, 5.099%, and 2.81% after 3, 7, 14, and 28 days of treatment with sulfuric acid, respectively. Furthermore, the interaction between metakaolin and calcium hydroxide produced calcium compounds that improved the density of the material and, hence, increased its mass [38]. Thus, when calcium compounds were produced, the mass of the samples increased. However, these increases reduced the mass increases of the samples containing metakaolin between 51.75%-105.37% from day 14 to day 28, while the decrease in the mass increase in the 0MK sample was 190.80%. The sharp decrease seen in the 0MK sample compared to the other samples can be attributed to the chemical reaction of sulfuric acid with the calcium content since the 0MK sample contains the highest amount of calcium [61]. Moreover, visual assessment was conducted on the geopolymer samples with varying metakaolin ratios immersed in sulfuric acid solution for 28 days, as illustrated in Figure 13. It was observed that more significant and visible damage occurred in all series after 28 days of acid treatment compared to the samples after 14 days of acid exposure.

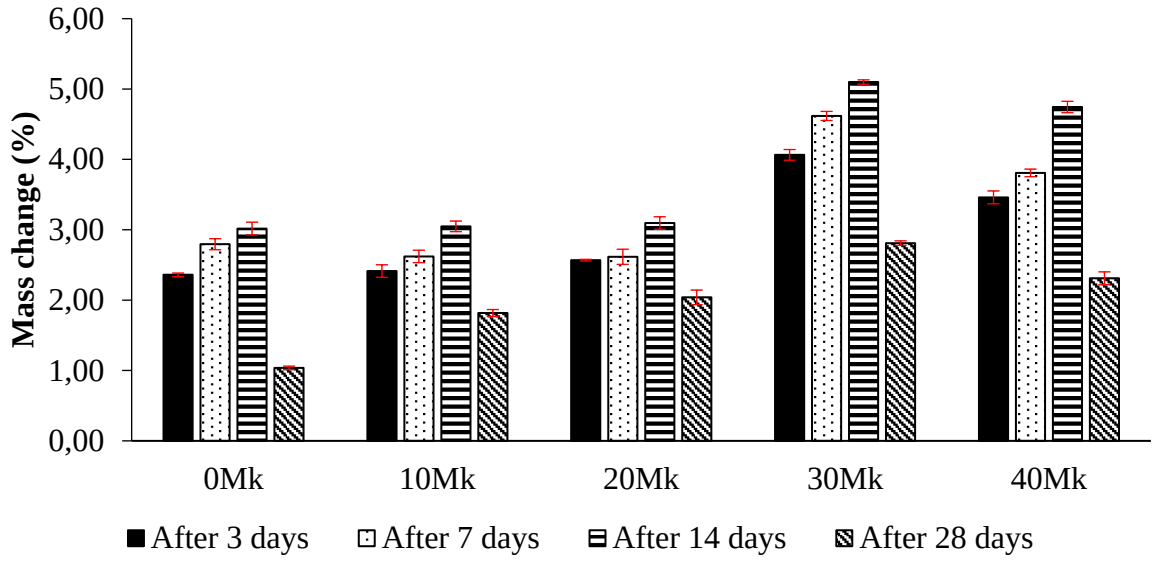


Fig.12. Mass increases of geopolymer samples with varying metakaolin specimens subjected to sulfuric acid for different durations

(a) (b) (c)

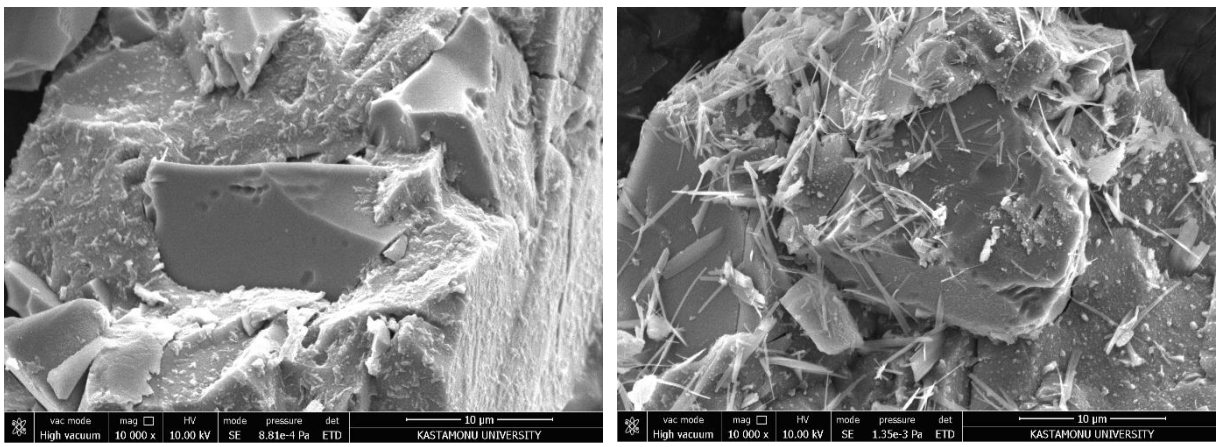
(d) (e) (f)

Fig.13. Visual assessment of (a) all series after 14 days of exposure to sulfuric acid, (b) 0MK, (c) 10MK (d) 20MK, (e) 30MK, and (f) 40MK, subsequent to 28 days of acid treatment

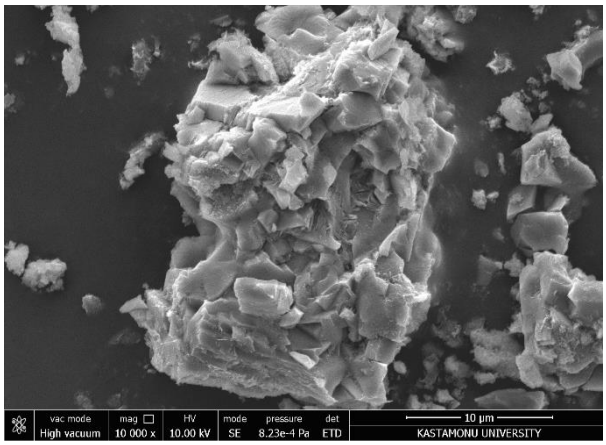
The outer shell of the samples exposed to acid for 28 days started to be damaged, and this damage caused changes in the cross-sectional dimensions of the samples and led to a decrease in their strength. In addition, it was observed that the damage was most severe in the 0MK sample, and this damage decreased with increasing metakaolin content. As the samples became more porous and absorbed more water, their strength decreased, indicating that the strength results correlate with the water absorption values [62].

3.7. Microstructural Investigation

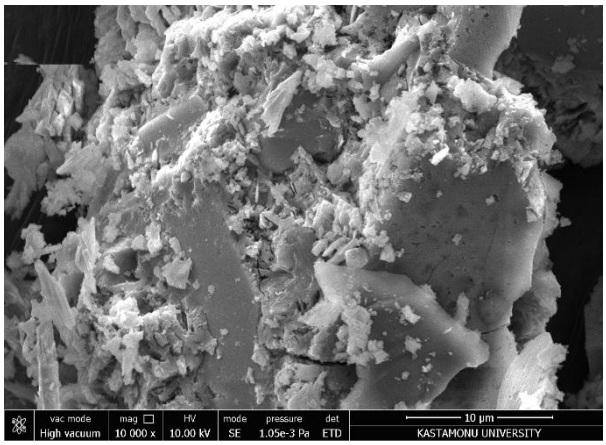
The 0MK and 40MK samples were selected for SEM analysis because they had the lowest and highest strengths before acid exposure, respectively. Furthermore, to ensure accurate comparisons, the 0MK and 40MK samples exposed to acid for 28 days were also selected. This allowed a comparison of the samples exhibiting the lowest and greatest strengths before acid exposure with those from the same series after acid exposure. Figure 14 illustrates the SEM examination of the samples prior to and following 28 days of exposure to acid solution. As illustrated in Figure 14 (a), no calcium sulfate needle crystals and structural deterioration were observed in the 0MK samples before acid exposure. Subsequent to 28 days of exposure to acid, the emergence of calcium sulfate needle crystals was noted, as shown in Figure 14 (b), potentially resulting in considerable strength degradation. This is due to the exposure of sulfuric acid, which causes $HSO4^-$ ions to assault the microstructure and recrystallize the gypsum, leading to the deterioration of the aluminosilicate gel matrix [61]. In addition, Figure 14 (c) illustrates sample 40MK before acid exposure, exhibiting a relatively compact structure because of the differences between the surface areas of metakaolin and slag [49].



(a) (b)



(c)



(d)

Fig.14. SEM microscopies of a) 0MK prior to sulfuric acid impact, b) 0MK following 28 days of sulfuric acid impact, (c) 40MK prior to sulfuric acid impact, and (d) 40MK subsequent to 28 days of exposure to acid treatment

Consequently, it was observed in Figure 14 (d) that sample 40MK had fewer calcium sulfate needle crystals than the 0MK sample following 28 days of acid treatment. This is due to metakaolin exhibiting superior resistance to acid corrosion, hence inhibiting the dissolution of ettringite-forming chemicals [63]. Djobo et al. [64] found that the sodium-rich gel in the sample enhances acid resistance, making permeability a determinant of the durability characteristics of geopolymer mortars.

4. Conclusions

- The flow test indicated that the geopolymer samples containing 40% metakaolin demonstrated the minimum flow value. Also, the augmenting of the metakaolin content decreased flow characteristics. This situation reveals the function of metakaolin in reducing workability. The reduced porosity and water absorption of samples containing elevated metakaolin content, specifically 30% and 40% MK, correlated with enhanced compressive strength. Samples exhibiting heightened porosity demonstrated diminished mechanical performance. The 30% MK geopolymer sample had the greatest UPV values, signifying superior cohesion and less porosity in the matrix. This highlights the significance of metakaolin in improving the density and stability of geopolymer structures.
- Samples with 30% and 40% metakaolin demonstrated superior compressive strength compared to those with lower proportions. Forty percent of metakaolin exhibited superior performance under initial conditions, whereas thirty percent of metakaolin demonstrated optimal performance following acid exposure. The optimal strength was linked to geopolymers due to the development of a three-dimensional geopolymer gel network (C-S-H and C-A-S-H). Strength augmented following 14 days of acid exposure, although diminished after 28 days. In addition, the flexural strength showed a decline following twenty-eight days of immersion in sulfuric acid. Moreover, immersion in sulfuric acid for 28 days led to the formation of calcium sulfate needle crystals inside the geopolymer matrix, resulting in crack propagation and a reduction in strength. Thus, 30% MK geopolymer sample was determined to be the ideal composition for acid-resistant geopolymer applications.
- This work significantly advances global sustainability objectives by demonstrating that slag-based geopolymer mortars, especially those enhanced with metakaolin, exhibit exceptional resistance to sulfuric acid while preserving robust mechanical properties. The results indicate that modifying metakaolin content improves chemical durability and facilitates more economical mix designs by minimizing excessive binder use. The use of industrial by-products like slag and metakaolin significantly reduces the carbon footprint relative to traditional Portland cement, hence enhancing the environmental benefits of geopolymer technology. Furthermore, the exhibited acid resistance establishes these materials as viable

options for enduring applications in infrastructure subjected to harsh conditions such as sewage systems, chemical storage facilities, and industrial plants where conventional protective measures are typically labor-intensive and expensive. The research emphasizes geopolymer mortars as a sustainable, durable, and cost-effective option, facilitating the development of environmentally friendly and durable building materials.

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