كلية الهندسة بشبرا
FACULTY OF ENGINEERING AT SHOUBRA
ERJ
PRINT ISSN 3009-6049
ONLINE ISSN 3009-6022

ENGINEERING RESEARCH JOURNAL (ERJ)
Volume (54) Issue (3)
July. 2025, pp:94-101
https://erjsh.journals.ekb.eg

Sulfuric Acid Resistance of Metakaolin Geopolymers Activated with a Dealuminated Kaolin-NaOH System

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Abstract: Sulfuric acid attack is an important aspect in the long-term mechanical and durability performance of geopolymer concrete. Keeping this in view, in this study the effect of sulfuric acid attack on compressive strength was investigated, as well as the microstructural changes of geopolymer mortar mixtures. Geopolymer mortar mixes were prepared by activating metakaolin with a mixture of dealuminated kaolin (DK) and sodium hydroxide (NaOH) suspension as an alkaline activator and cured at ambient temperature. The effect of diluted sulfuric acid solution (pH=3) was investigated after 28 days of curing at ambient temperature. A comprehensive experimental program was carried out, including the evaluation of compressive strength, sorptivity, absorption, and volume of permeable voids prior to acid exposure. Over a ten-week exposure period, weight change and strength degradation were assessed. Microstructural analyses using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) were conducted before and after exposure to confirm the findings. The results of the study revealed that the formed geopolymer mortars resist the sulfuric acid (5%). It was found that about a 21.7% reduction in the compressive strength was recorded.

Keywords: Alkali-activated materials; Geopolymers; Dealuminated kaolin; Alkaline activator; Metakaolin; Sulfuric acid.

1. INTRODUCTION

The industrial sector produces substantial amounts of solid waste, creating significant challenges related to disposal and environmental management. Among the various environmental concerns, global warming remains one of the most critical issues, and the cement industry contributes considerably to this problem due to its intensive energy consumption and associated emissions of greenhouse gases such as carbon dioxide [1,2]. To promote sustainable development, numerous researchers have investigated the utilization of industrial solid wastes as raw materials for producing alternatives to Ordinary Portland Cement (OPC), aiming to address waste disposal challenges while mitigating greenhouse gas emissions and their impact on global warming.

The incorporation of solid waste in concrete applications has demonstrated significant benefits, particularly in enhancing sustainability and resource efficiency. However, such waste materials are primarily utilized as partial substitutes for Ordinary Portland Cement (OPC) [3,4,5]. Davidovits [6] pioneered the concept of geopolymers, which are synthesized through the reaction of alumino-silicate materials with alkali-activating solutions. Among these materials, metakaolin is notable for its high alumina and silica content, making it a viable precursor for geopolymer formation[7].

Geopolymer synthesis involves the activation of aluminosilicate species liberated by alkaline hydroxides with sodium or potassium silicates, which serve as alkali-activating solutions. This process results in the formation

of an alumina-silicate polymeric binding structure. Extensive studies [8,9,10] have identified several factors that influence the mechanical performance and durability of geopolymers, including the selection of source materials, particle fineness, alkali-activator concentration, and curing conditions. Unlike conventional binders that rely on calcium silicate hydrate (C–S–H) for structural integrity, geopolymer binders derive their stability from aluminosilicate gels, imparting superior early-age strength, reduced creep and shrinkage [11], and improved resistance to aggressive environments, including exposure to acids and sulfates [11,12]. Metakaolin, owing to its abundance and favorable reactivity, has been widely utilized as a primary precursor in geopolymer formulations, demonstrating effective performance in various applications.

In this study, dealuminated kaolin (DK), which is produced as a waste byproduct of aluminum extraction from calcined kaolin by sulfuric acid, was used with sodium hydroxide (NaOH) as an alkaline activator instead of traditional sodium silicate to produce metakaolin geopolymer mortars. Specimens are prepared, cured at ambient temperature, and their resistance to sulfuric acid has been examined. Sulfuric acid was selected over other acids because it is commonly encountered in practice, as concrete structures are frequently exposed to sulfuric acid in various environments such as mining, sewage, and food processing industries. Mechanical properties, including mass transport characteristics, were assessed prior to acid exposure. Sulfuric acid resistance was then examined over 2, 4, 6, 8, and 10 weeks of immersion, with parameters such as mass loss and residual compressive strength used to monitor deterioration. Furthermore, microstructural changes were analyzed using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) on both unexposed and sulfuric acid-exposed specimens after ten weeks of immersion.

2. EXPERIMENTAL WORK

2.1 Materials

Dealuminated kaolin (DK) was sourced from the Egyptian Aluminum Sulfate Factory, where it is produced as a waste by-product during the extraction of aluminum from calcined kaolin using sulfuric acid, resulting in substantial quantities that are currently stockpiled on site. Metakaolin (MK) was purchased from the local market, and the chemical composition and physical properties of both DK and MK are presented in Table 1. The fine aggregate used was clean siliceous river sand with a fineness modulus of 1.74, water absorption of 1.5%, and a specific gravity of 2.5; it was sieved through a 2.36 mm sieve, and only the fraction passing was used in the mixes. Sodium hydroxide pellets (98% purity) were dissolved in distilled water to prepare a 12 M NaOH solution, which was then mixed with DK in specified proportions to produce the alkaline activator suspension. Four different suspensions were prepared by adjusting the DK content; in each case, DK was weighed and dispersed in water before adding the NaOH solution at constant molarity. Due to the exothermic nature of the reaction, the suspensions were cooled to room temperature and stored for 24 hours prior to use.

TABLE 1. Chemical composition and physical properties of MK and DK**.**

Chemical composition (%)											
Compound	SiO_2	Cao	Al_2O_3	Fe_2O_3	MgO	TiO_2	Na_2O	K_2O	SO_3	LOI	Total
MK	55.3	0.51	33.5	1.50	0.19	2.20	0.16	0.07	0.39	4.05	99.08
DK	82.0	0.16	6.70	0.53	0.08	3.50	0.02	0.04	0.95	4.90	99.03
Physical properties											
	Specific gravity				Blaine (cm^2/g)				Color		
MK	2.5				12000				Faint rose		
DK	2.1				42000				Off-White		

TABLE 2. Mixture proportions.

Mix ID	**DK/NaOH (Activator)**	**Activator/MK**	**Water/solid**
GPM1	0.80	0.90	0.35
GPM2	0.88	0.75	0.34
GPM3	1.00	0.80	0.33
GPM4	1.13	0.85	0.32
GPM5	1.25	0.90	0.31

2.2.2 Mixing, curing and casting

In the preparation of geopolymer mortar, the metakaolin and alkaline suspension were first mixed in the specified proportion for 10 min., and sand was then added and mixed for another 5 min. The mixing time was chosen to enable the production of a mix that is homogeneous and uniform. The fresh mortars were immediately transferred to the molds, which were vibrated with a shaker for 1 min to eliminate the entrapped air. After vibrating, the molds were immediately transferred into the curing room (25 ± 2°C, 60 ± 5% RH), and the samples were covered with polyethylene sheets to prevent loss of moisture. After 24 h the samples were demolded and cured at ambient temperature up till the time of the test.

2.3 Testing of mortar specimens

2.3.1 Compressive strength

The compressive strength of ambient cured geopolymer mortar samples (50 × 50 × 50 mm) was measured according to ASTM C109/C109M [13]. Tests were conducted on a DYE-2000 electro-hydraulic pressure machine at a loading speed of 1 kN/s. The test was conducted on cubes at the ages of 7 and 28 days, and the averages were reported.

2.3.2 Mass transport properties

The mass transport properties of the geopolymer mortar were evaluated primarily through sorptivity, as the rate of chemical ingress is significantly affected by water uptake via capillary suction. The sorptivity of the specimens was evaluated as per ASTM C1585–13 [14]. Meanwhile, density, water absorption, and the volume of permeable pores were measured following ASTM C 642-13 [15]. These parameters collectively served as quantitative indicators of the potential resistance of the mortars to acid penetration.

2.3.3 Sulfuric acid exposure

Sulfuric acid resistance tests were performed as per ASTM C267 [16]. 28-day-aged samples were pre-soaked in water for 1 day before being submerged into the 5% sulfuric acid solution (pH=3) for different durations (2, 4, 6, 8, and 10 weeks). The specimens after the sulfuric acid exposure were removed from the solution and quickly dried by blotting with paper towels. After subsequent storage in the chamber room (18°C–22°C, RH = 55%–65%) for 1 h, the samples were tested for compressive strength and mass loss.

2.3.4 SEM with EDS

A comprehensive microstructural analysis was carried out to characterize the morphological features, elemental composition, and chemical structure of the geopolymer matrix before and after sulfuric acid exposure. Specimens were extracted from the surfaces of mortars cured at ambient temperature for 28 days, as well as from counterparts immersed in sulfuric acid for 10 weeks. Surface morphology and microstructural integrity were examined using scanning electron microscopy (SEM) with a TESCAN VEGA3 operated at an accelerating voltage of 20 kV and a beam current of 100 pA. To improve conductivity and imaging resolution, the specimens were sputter-coated with a thin layer of gold. Elemental composition was simultaneously analyzed using an integrated energy-dispersive X-ray spectroscopy (EDS) detector.

3. RESULTS AND DISCUSSIONS

3.1 Compressive strength of specimens before exposure.

The compressive strength of the metakaolin-based geopolymer mortars was measured after 28 days of ambient curing to establish the baseline mechanical properties prior to sulfuric acid exposure (Table 3). The results indicated that the 28-day compressive strength ranged from 39.0 MPa for GPM1 to 47.7 MPa for GPM4, reflecting a notable sensitivity to variations in the water-to-solid (W/S) ratio, DK/NaOH ratio, and Activator/MK ratio. such parameters significantly affect the degree of geopolymerization and contribute to the refinement of the matrix structure. These baseline strength values are essential for interpreting the subsequent performance under sulfuric acid attack. Generally, higher compressive strength implies a denser and less permeable microstructure, which enhances chemical durability.

TABLE 3. Compressive strength of GPM mixtures at 28 days of curing

Mixture ID	28-days compressive strength (MPa)
GPM1	39.0
GPM2	39.7
GPM3	42.5
GPM4	47.7
GPM5	40.5

As illustrated in Fig. 1, the compressive strength of the GPM mixtures increased as the W/S ratio decreased from 0.35 to 0.32, with strength improving from 39.0 MPa (GPM1) to 47.7 MPa (GPM4). This trend aligns with the well-established understanding that lower water content

leads to reduced porosity and improved particle packing, which enhances mechanical performance. However, a further decrease to 0.31 in GPM5 resulted in a slight drop in strength to 40.5 MPa, which may be attributed to insufficient workability and incomplete geopolymerization, ultimately leading to microstructural defects and less effective gel development. These findings are consistent with previous studies that reported similar adverse effects at excessively low W/S ratios [17].

It should be noted that the W/S ratio, DK/NaOH ratio, and activator-to-MK ratio were varied simultaneously across the mixtures, introducing a degree of interdependence among parameters. While this limits the ability to fully isolate the impact of each individual variable, the overall strength evolution appears to be primarily governed by the W/S ratio. The trend supports the conclusion that reducing the W/S ratio enhances compressive strength up to an optimal point; however, the slight decline observed in GPM5 suggests that excessively low water content combined with variations in the DK/NaOH and activator/MK ratios may have negatively influenced workability and gel development. These interdependencies underscore the complexity of geopolymer mix design and emphasize the need for a carefully balanced approach to activator composition and solid content to achieve optimal performance.

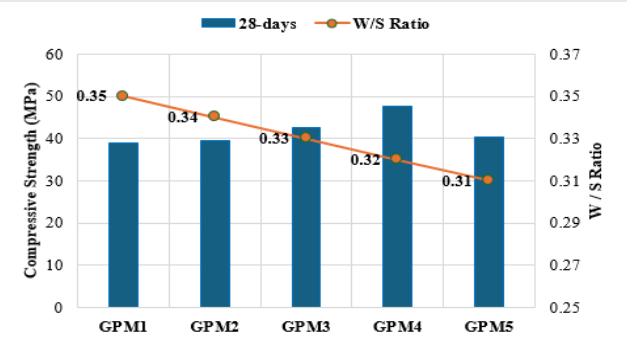


Fig 1. Effect of W/S ratio on compressive strength for GPM-mixtures

3.2 Mass transport properties

3.2.1 Sorptivity

Table 4 presents the sorptivity results for the GPM mixes, which ranged from 0.0037 to 0.0047 g/cm²·s⁰·⁵. The lowest sorptivity was observed for GPM4, indicating a denser and less permeable matrix than the other mixes, consistent with its superior compressive strength. Conversely, GPM1 recorded the highest sorptivity value (0.0047 g/cm²·s⁰·⁵), suggesting a relatively more porous microstructure that permits greater capillary water uptake. In general, lower sorptivity values reflect reduced capillary water absorption, which contributes to enhanced durability of geopolymer mortars. According to ASTM C1585 [14], sorptivity values below approximately 0.005 g/cm²·s⁰·⁵ are considered acceptable for cementitious materials with satisfactory resistance to water ingress. Therefore, all tested mixes in this study meet this criterion, and the results were in agreement with previous studies [18,19], confirming their suitability for applications that require low permeability and good durability performance.

TABLE 4. Sorptivity of GPM mixtures at 28 days of curing.

Mixture ID	Sorptivity g/cm².sec$^{-0.5}$
GPM1	0.004737
GPM2	0.004684
GPM3	0.004624
GPM4	0.003743
GPM5	0.004321

3.2.2 Density, Absorption, and Voids.

To evaluate the physical characteristics of hardened geopolymer mortars. Density, absorption, and volume of permeable voids were determined according to ASTM C642 [15] to quantify the amount of water that can be absorbed by the geopolymer mortar, which in turn indicates the connectivity and extent of its pore structure, and to provide insight into the compactness and integrity of the geopolymer mortar matrix.

Fig.2 illustrates the water absorption percentages of the GPM mixes after immersion and after immersion followed by boiling. It is evident that all mixes showed an increase in absorption values after boiling, indicating that elevated temperatures facilitate greater water ingress into the pore structure. Among the mixes, GPM1 recorded the highest absorption both after immersion and after boiling, suggesting a more open pore network. In contrast, GPM2 and GPM4 exhibited lower absorption values, implying a denser microstructure with less accessible pores. This trend confirms that mixes with optimized activator ratios and lower water-to-solid ratios tend to produce mortars with reduced water absorption, enhancing durability against moisture penetration, which aligns with similar findings reported in previous studies [20].

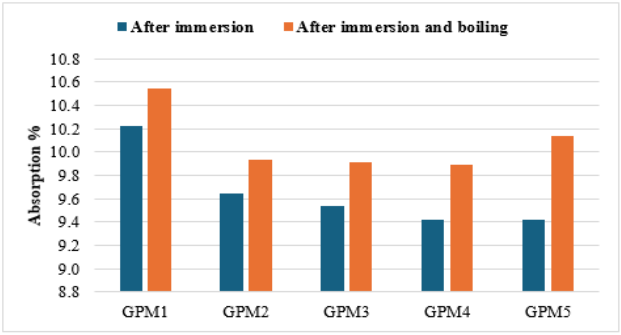


Fig 2. Absorption test results for GPM-mixtures.

As shown in Fig.3, the bulk density, bulk density after immersion and boiling, and apparent density were determined for the GPM mixtures. As expected, the apparent density values were consistently higher than the bulk density due to the exclusion of connected pore spaces. Overall, the results show moderate variations among the mixes, with average bulk density values ranging approximately between 1.9 and 1.94 g/cm³, which is typical for dense geopolymer mortars. GPM4 demonstrated the highest density across all measurement conditions, with an apparent density exceeding 2.4 g/cm³, reflecting a well-compacted matrix with minimal internal voids. This is consistent with its low absorption and low volume of permeable pores, indicating superior packing efficiency, which agrees with findings reported elsewhere [21] .On the other hand, GPM1 recorded the lowest bulk and apparent densities, dropping closer to 1.9 g/cm³, which aligns with its higher absorption capacity and greater pore volume, implying a more porous structure.

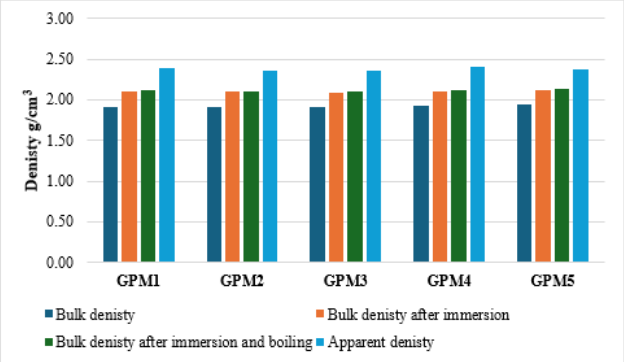


Fig 3. Bulk and Apparent Density results for GPM-mixtures.

Fig.4 depicts the inverse relationship between the compressive strength and the volume of permeable pores for the GPM series. The data indicate that mixes characterized by lower volumes of interconnected voids generally develop higher compressive strengths, which is consistent with previous findings [22,23]. For instance, GPM4 achieved the maximum compressive strength in conjunction with one of the lowest volumes of permeable pores (18.9%), demonstrating the beneficial effect of a dense, well-structured matrix in enhancing load-bearing capacity. In contrast, GPM1 exhibited the highest pore volume (20.13%) and correspondingly lower compressive strength, emphasizing the detrimental impact of excessive porosity on the mechanical performance of geopolymer mortars. This clear correlation validates that controlling pore structure through optimized mix design is essential to achieving superior mechanical and durability performance, particularly when resistance to acid attack is a critical requirement

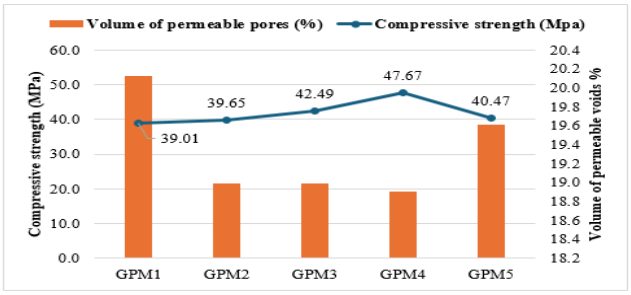


Fig 4. Volume of permeable voids and 28-days compressive strength results for GPM-mixtures.

3.3 Sulfuric acid exposure

3.3.1 Weight change

On a weekly basis, the percentage weight change (in saturated but surface-dry condition) relative to the initial saturated mass (recorded prior to acid exposure) was determined for each mix. The average weight change of three replicate specimens was calculated to ensure the reliability of the results. Fig.5 illustrates the variation in weight loss of the geopolymer mortar mixes (GPM1 to GPM5) as a function of exposure duration in a 5% sulfuric acid solution (of about pH=3). As expected, all mixes exhibited a progressive increase in weight loss with prolonged immersion, indicating continuous chemical degradation due to acid attack. Among the investigated mixes, GPM1 consistently recorded the highest weight loss throughout the test period, reaching approximately 5.5% after 10 weeks of exposure, which reflects its limited resistance to acidic environments. Conversely, GPM4 demonstrated the lowest weight loss at all immersion intervals, maintaining a total weight loss below 2% even after 10 weeks, suggesting superior chemical stability and enhanced durability against acid-induced deterioration. The remaining mixes (GPM2, GPM3, and GPM5) displayed intermediate performance, with weight loss values falling between those of GPM1 and GPM4. These findings are consistent with previous studies [24], which confirmed the adequate stability of geopolymer concrete against sulfuric acid, indicating typical mass losses of only 5–8% compared to 30–60% for ordinary Portland cement concrete under similar conditions [25]. This highlights the improved durability of geopolymer systems in aggressive acidic environments.

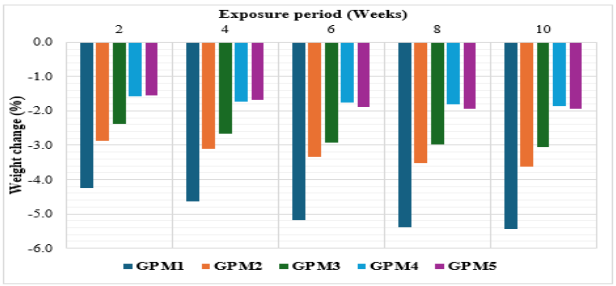


Fig5. Weight change (%) of GPM mixtures after exposure to 5% sulfuric acid solution.

3.3.2 Compressive strength degradation

The results presented in Fig.6 illustrate the compressive strength degradation relative to the original 28-day strength of each GPM mixture (GPM1–GPM5) following exposure to a 5% sulfuric acid solution for periods ranging from 2 to 10 weeks. As expected, all mixtures experienced a progressive decline in compressive strength with increasing exposure duration, reflecting the adverse effect of the acidic environment on the long-term durability of geopolymer materials. Among the investigated mixtures, GPM1 exhibited the highest level of strength deterioration throughout the test period, reaching an approximate maximum loss of 34.21% at 10 weeks, indicating limited resistance to acid attack. The reduction in strength was attributed to the breakage of aluminosilicate bonds under prolonged exposure to sulfuric acid. It has also been reported in previous studies [26,27] that the observed strength reduction is mainly due to the development of zeolitic phases and the consequent depolymerization of the geopolymer structure. In comparison, GPM2, GPM3, and GPM5 showed moderate levels of strength degradation, suggesting a relatively better but still notable susceptibility to chemical deterioration. Remarkably, GPM4 demonstrated the lowest degree of compressive strength loss with only 21.65% among all mixtures, retaining a substantial portion of its initial strength even after prolonged acid exposure. This superior performance highlights the effectiveness of its specific composition in enhancing chemical stability and resistance under aggressive acidic conditions. Accordingly, GPM4 can be considered the most durable and acid-resistant mixture within this study. Overall, compressive strength retention under acid attack strongly correlates with prior indicators of durability, including sorptivity, absorption, and volume of permeable voids.

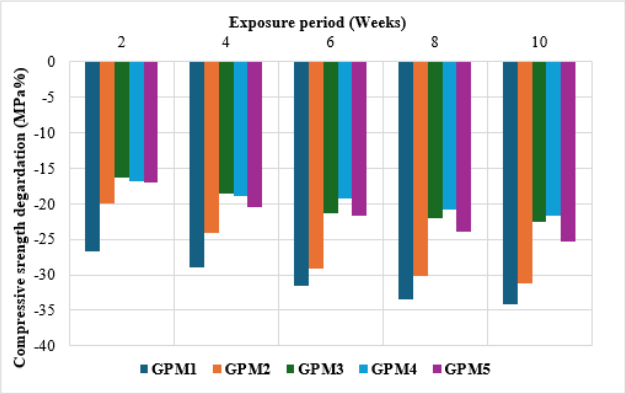


Fig 6. Compressive strength degradation (%) of GPM mixtures after exposure to sulfuric acid.

3.4 SEM with EDS

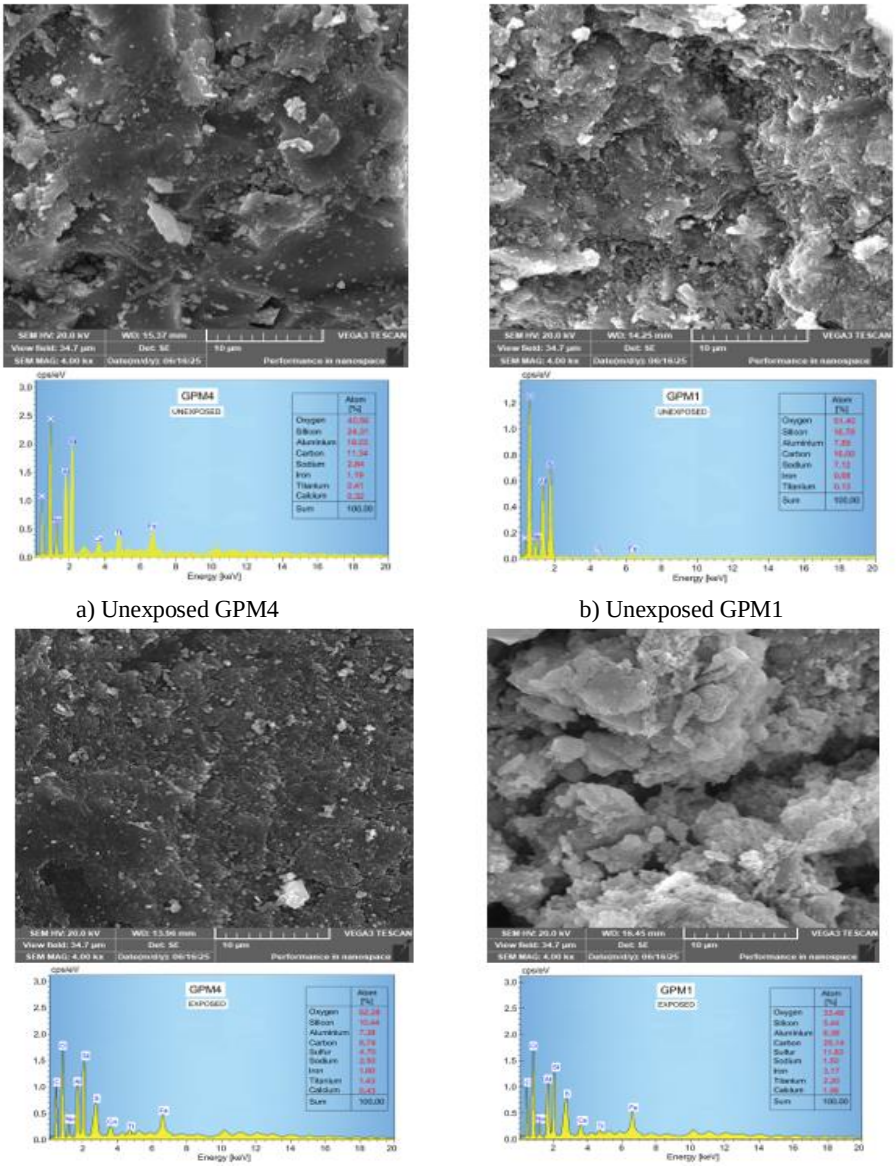
The microstructure of selected mortars before and after exposure to sulfuric acid solution was examined using SEM and EDS analysis to investigate the morphology and structural integrity of the geopolymeric gel at the microscale. From the results obtained for weight change and compressive strength degradation, for all specimens, maximum deterioration was observed at 10 weeks. Therefore, micrographs were observed for selected specimens after 10 weeks of sulfuric acid exposure and compared to the similar unexposed geopolymer mortar specimens. Similarly, EDS analysis was also performed on the exposed as well as unexposed specimens.

The primary elements detected in the EDS analysis before acid exposure were oxygen (O), aluminum (Al), silicon (Si), carbon (C), and sodium (Na), with minor traces of iron (Fe), titanium (Ti), and calcium (Ca), which originate from the raw DK and MK materials. Following sulfuric acid exposure, the same elements were observed, along with the appearance of sulfur (S), confirming the interaction of the geopolymer matrix with the acidic environment.

Fig.7 presents SEM micrographs with corresponding EDS for two representative mixes before and after exposure to sulfuric acid solution: one well-performing (GPM4) and one poorly performing (GPM1). The microstructure of GPM4, which demonstrated the highest compressive strength and lowest sorptivity, absorption, and volume of permeable voids, revealed a highly dense and homogenous gel matrix. The surface appeared compact, with minimal microcracking and an absence of unreacted or loosely bonded particles. The gel structure was continuous and well-integrated, reflecting an advanced degree of geopolymerization and matrix cohesion. The EDS spectrum confirmed the presence of Si, Al, and Na, which are typical constituents of alkali-activated aluminosilicate gel. By contrast, SEM image of GPM1 displayed a porous and discontinuous structure. The surface was rough, with visible cracks, voids, and loosely bound particulate clusters scattered throughout the matrix. The morphology suggested a poorly consolidated and weakly bonded gel, indicative of either incomplete reaction or excessive activator use, which could lead to rapid drying and structural instability. The network appeared heterogeneous and lacked the fine microstructural refinement seen in GPM4.

After 10 weeks of exposure to sulfuric acid, notable microstructural differences were evident between the two mixes. In GPM1, the matrix exhibited pronounced signs of chemical deterioration; SEM observations revealed extensive microcrack widening, surface erosion, and partial dissolution of the geopolymeric gel. These features point to sulfate-driven decalcification and acid-induced hydrolysis of the aluminosilicate network. Voids became more

pronounced and interconnected, supporting the observed increase in overall porosity and the weight loss confirmed by experimental measurements. In contrast, GPM4 displayed a considerably more stable microstructure under the same conditions. Only slight surface roughening and isolated pore formation were observed, while the overall gel network remained largely intact. No significant propagation of microcracks or binder disintegration was detected. This preserved structural integrity is consistent with the high residual compressive strength and minimal mass loss of GPM4, highlighting the effectiveness of its dense, well-crosslinked gel structure in resisting sulfuric acid ingress and degradation. In addition, the EDS results verified the incorporation of sulfur species in the geopolymer system after exposure, suggesting that available calcium interacted with the sulfuric acid to form secondary sulfate phases. These sulfur-based products were responsible for the weight loss as well as the decremented strength values for the specimens exposed to the sulfuric acid solution.



a) Unexposed GPM4 b) Unexposed GPM1

c) Exposed GPM4 d) Exposed GPM1

Fig 7. SEM with EDS micrographs of unexposed and sulfuric acid-exposed specimens: (a) GPM4 before exposure, (b) GPM1 before exposure, (c) GPM4 after exposure and (d) GPM1 after exposure.

4. CONCLUSIONS

Based on the outcomes of this investigation, the following key conclusions can be drawn:

- Geopolymer mortars can be successfully synthesized using a DK–NaOH mixture as an alternative sustainable alkaline activator, providing a viable substitute for commercial sodium silicate .

- The developed DK–NaOH suspension demonstrated good reactivity and contributed effectively to the geopolymerization process.
- The 28-day compressive strength results confirmed that careful optimization of the W/S, DK/NaOH, and Activator/MK ratios enhances matrix densification and strength development. An optimum W/S ratio of approximately 0.32 achieved the highest compressive strength before further reduction due to incomplete geopolymerization at lower ratios.
- Mortars with lower sorptivity, reduced water absorption, and fewer permeable voids exhibited higher compressive strength, emphasizing the importance of dense and well-refined microstructure in enhancing durability.
- The geopolymer mortars showed satisfactory resistance to 5% sulfuric acid exposure, with a moderate compressive strength reduction of about 21.7% after 10 weeks of immersion, indicating good chemical durability in aggressive environments.
- Microstructural analyses using SEM coupled with EDS confirmed the formation of dense gel phases in well-optimized mixes and revealed limited microstructural deterioration in acid-resistant specimens, supporting the durability test results.
- Overall, the findings demonstrate that dealuminated kaolin (DK), produced as an industrial by-product, serves as an economical and eco-friendly alkaline activator that enhances both the strength and durability of metakaolin geopolymer mortars against sulfuric acid deterioration.

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