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Article

Geopolymer Degradation by Oxalic Acid: A Kinetic Study

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Abstract

The increasing need for sustainable construction materials has prompted research into alternatives to Ordinary Portland Cement (OPC), a major contributor to global CO₂ emissions. Geopolymers, synthesized via alkali activation of aluminosilicate precursors such as metakaolin and fly ash are a promising alternative reducing up to 80% carbon emissions. However, their long-term durability under aggressive chemical environments, particularly against organic acids, remains insufficient. While mineral and inorganic acid resistance has been studied, the impact of naturally occurring organic acids like oxalic acid (Ox) —commonly found in soils and organic-rich sediments—has received limited attention. Ox is known to chelate metal ions and alter mineral phases, potentially affecting the integrity of geopolymer matrices. This study investigates the degradation behavior of geopolymers under continuous exposure to Ox (0.2, 0.4, and 0.6 M) at 25 °C using a flow-through reactor. Mass loss over time was monitored to determine reaction kinetics, while SEM, FT-IR, XRD, and EDS analyses were conducted to evaluate microstructural and chemical changes. Results revealed significant alterations in geopolymer structure due to Ox exposure, providing key insights into their vulnerability to organic acid attack. These findings remark the importance of considering organic acid interactions in the long-term performance assessment of geopolymers.

Keywords: geopolymer; alkali-activated materials; oxalic acid; organic acid attack; durability; flow-through reactor; reaction kinetics

1. Introduction

The increasing global demand for sustainable construction materials has driven significant research into alternatives to traditional binders such as Ordinary Portland Cement (OPC), whose production accounts for approximately 6–9% of global CO₂ emissions annually [1,2]. Geopolymers, a class of inorganic polymers formed by the alkali activation of aluminosilicate-rich precursors such as metakaolin (MK), fly ash (FA), and ground granulated blast-furnace slag for instance, are a promising alternative by reducing up to 80% of CO₂ emissions compared con OPC while providing an excellent mechanical resistance tailored by the selection of the Si/Al ratio, curing conditions and water content [3–5].

Despite these advantages, the long-term durability of geopolymers remains a critical concern, particularly under aggressive chemical environments. Various studies have reported that exposure to sulfuric and acetic acid can lead to depolymerization, dealumination, leaching of alkali cations,

increase of Si/Al atom ratio, breakage of the Si-O-Si and Si-O-Al bonds, and a decrease in compressive strength [6–8].

Previous works have extensively evaluated the acid resistance of geopolymers under common acids such as sulfuric, hydrochloric, and acetic acid [6]. However, organic acids, particularly those occurring naturally or as metabolic by-products in soil and sedimentary environments [9], have received limited attention.

These organic acids are important species because they may interact with carbonates and aluminosilicates resulting in dissolution of the minerals [10] and may be responsible for the formation of secondary porosity in deep sedimentary rocks [11]. One of these organic acids is oxalic acid (Ox), which has been found in soils, peats and another organic matter-rich sediment where it forms stable calcium oxalate phases such as weddellite and whewellite [12]. This organic acid, known to chelate metal ions and alter mineral phases, plays a key role in mineral weathering and porosity formation in natural sedimentary systems [10,11].

Recent studies have explored the use of Ox and its derivatives in a wide range of applications such as improving the performance and sustainability of cementitious materials, by enhancing carbon sequestration, mechanical strength, and hydration kinetics. Such as the use of Ox, lactic and citric acid to extract aluminum from metakaolin by chemical leaching [13]. The synthesis of an iron-based construction binder by mixing fly ash, Ox, metakaolin, and calcium carbonate where it was found that the use of oxalic acid enhances the matrix densification and mechanical strength by promoting siderite (FeCO_3) and calcite (CaCO_3) formation through improved ion dissolution, whereas its absence decreases CO_2 diffusion and reduces mechanical strength [14]. The dissolution of inorganic calcium and particle size reduction in salt-loss soda residue (SLSR) to be used (the last one) in combination with fly ash as a raw material for geopolymer synthesis. When proper Ox is introduced in combination with SLSR, it contributes to higher compressive strength, reducing drying shrinkage, and increasing thermal stability due to the loss of soluble salts and the formation of calcium oxalate in the matrix [15].

Some other applications for derivatives of oxalic acid, such as sodium oxalate, have been discovered, where it has been used to form thermodynamically stable phase of calcium oxalate monohydrate ($\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$) known as whewellite when it is added to fly ash-OPC blends [16]. Moreover, the activation of basic oxygen furnace enhances the early hydration kinetics, microstructure development and heavy metals retention for ordinary Portland cement [17].

Nevertheless, to the best of our knowledge, no systematic study has investigated the degradation behavior of geopolymers under oxalic acid attack, a potential threat in environments where oxalate-producing organisms or oxalate-rich organic matter may be present. Therefore, the present study aims to address this knowledge gap by evaluating the degradation rate of geopolymers under controlled Ox exposure.

To do this, a flow-through reactor was constructed to simulate continuous acid exposure to concentration of 0.2, 0.4 and 0.6 M of Ox at 25 °C. The reaction extent was followed by assessing the weight loss through time, and the reaction kinetic was elucidated then. The effect of the attack of Ox over the geopolymer was observed by SEM, and evaluated by FT-IR, XRD, and EDS techniques. The findings offer new insights into the vulnerability of geopolymers to organic acid attack and highlight the importance of considering such interactions in long-term durability assessments of alkali-activated materials.

2. Materials and Methods

2.1. Materials

Metakaolin used in this study was sourced from BASF Corporation (USA). Its chemical composition by weight was as follows: 51.55% SiO_2 , 44.79% Al_2O_3 , 0.20% Na_2O , 0.15% K_2O , and 1.61% TiO_2 . Sodium silicate, commercially available under the trade name STIXO, was obtained from SIDESA Corporation (Mexico) with a chemical composition of 9.2 wt% SiO_2 and 29.9 wt% Na_2O .

Oxalic acid dihydrate ($\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) was purchased from Sigma-Aldrich and used without further purification. Solutions of 0.2, 0.4 and 0.6 M were prepared by dissolving 379.5, 750 and 1134 g of Ox dihydrate in 15 L of deionized water respectively.

Geopolymer synthesis was carried out according to the procedure outlined in a previous study [18]. The $\text{SiO}_2/\text{Al}_2\text{O}_3$ and $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ molar ratios were chosen based on literature data aimed at optimizing mechanical strength [19]. For this study, the chosen ratios were $\text{SiO}_2/\text{Al}_2\text{O}_3 = 3.314$ and $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3 = 0.426$. These ratios represent a hypothetical structural geopolymer expected to be exposed to oxalic acid attack.

2.2. Flow Through Reactor and Reaction Rate

Samples of around 400 mg were settled inside a flow through reactor (Figure 1) under a continuous and homogeneous fluent of Ox ($35 \text{ ml} \cdot \text{min}^{-1}$ at 25°C). To meet these conditions, a fraction of the Ox solution was introduced into the reactor, while the other was returned to a temperature-controlled reservoir. Degraded solids were retained within the reactor by filtering the effluent at the top and by gravity, which caused them to settle at the bottom.

For each time, the decay fraction of geopolymer (f) was estimated using Equation 1. The flow of Ox was then stopped to open the reactor carefully and recover the remaining solids without damaging their structure. Then, the solids were rinsed with deionized water, dried at 130°C , and weighed to measure their mass (m). For each Ox concentration, the fraction of degraded geopolymer was plotted against reaction time. Special care was taken to dry the sample (m_0) to dismiss the effect of free water during weighting. The analyses were conducted at different times, with steps of 3 minutes, for a total of 30 minutes. In each trial (conducted in triplicate), the reaction was stopped, and a new sample was introduced.

$$f = \frac{m_0 - m}{m_0} \quad (1)$$

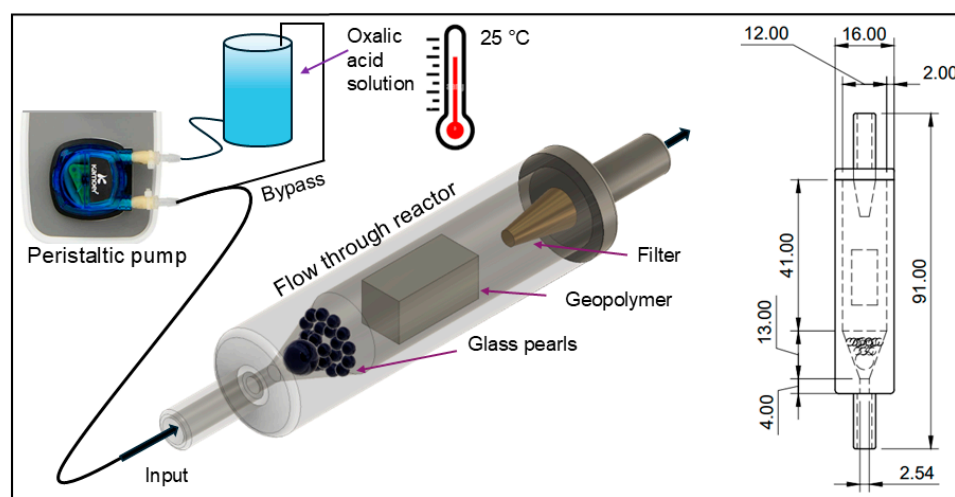


Figure 1. Scheme of set up employed to degrade geopolymers, along with the flow through reactor design.

2.3. Characterization

To analyze the chemical composition of the leachate, solid particles were removed by centrifugation (3600 rpm, 2 h), and the supernatant was concentrated at 80°C until a colloidal phase developed. Meanwhile, the remaining sediments in the reactor were collected and dried at 130°C for 2 hours for subsequent characterization of their chemical composition and morphology. The smaller particles were ground to be characterized in a RIGAKU Ultima-IV X-ray diffractometer using Cu irradiation ($\lambda = 1.5406 \text{ \AA}$, D/tex Ultra silicon strip detector) to elucidate crystalline phases. Their

molecular structure was determined by FT-IR spectroscopy (Perkin Elmer Spectrum Two) using the KBr technique (4000 to 350 cm⁻¹ with a resolution of 4 cm⁻¹). Particles larger than 10 U.S. STD. sieve were carefully handled with tweezers and observed through a Hitachi SU8230 scanning electron microscope, coupled to a Bruker Nano EDS. Chemical characterization involved X-ray diffraction and FT-IR spectroscopy, while morphological analysis included SEM imaging and EDS elemental mapping. The mean and standard deviation of the elemental composition were calculated, and statistical differences between the untreated geopolymer and those subjected to chemical attack were assessed using analysis of variance (ANOVA).

3. Results

A linear behavior of the decay fraction against reaction time was observed for each Ox concentration. This behavior suggests a zero-order reaction, where the decay rate (degradation rate of geopolymer, *r*) depends only on the Ox concentration and temperature. Therefore, the decay rate represented by Equation 2 turns into Equation 3, where the dependence of the reaction rate on a decay constant (*K*) is remarked. This decay constant is shown in Table 1, along with the linear regression parameters for each Ox concentration.

$$r = \frac{df}{dt} = \frac{d}{dt} \left[\frac{m_0 - m}{m_0} \right] = - \frac{1}{m_0} \frac{dm}{dt}$$
(2)

$$r = \frac{df}{dt} = -K$$
(3)

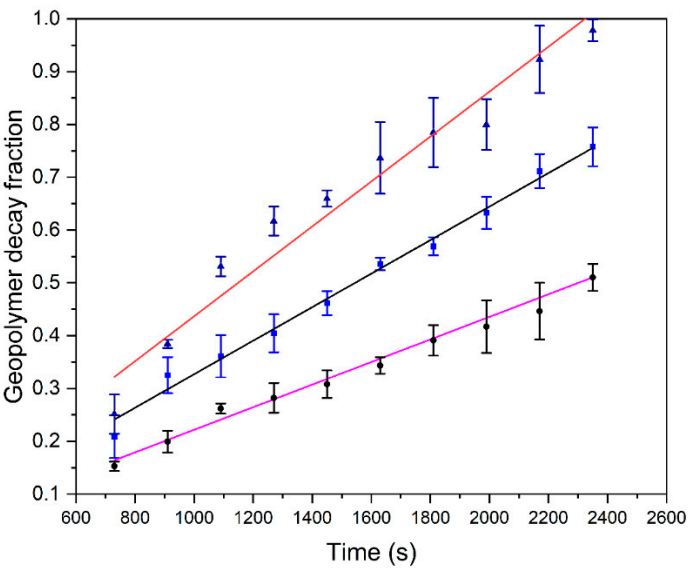


Figure 2. Mass fraction changes in geopolymers (SiO₂/Al₂O₃ = 3.314 and Na₂O/Al₂O₃ = 0.426) during exposure to Ox solutions at 25 °C with concentrations of 0.2 M (●), 0.4 M (■) and 0.6 M (▲).

Table 1. Regression equations for each Ox concentrations, along with their statistical parameters and decay constant (*K*).

Oxalic acid concentration	Intercept		Slope		Statistics	Decay constant
	Value	Standard Error	Value (s ⁻¹)	Standard Error (s ⁻¹)		
0.2 M	0.00789	0.0144	2.1383x10 ⁻⁴	1.18553x10 ⁻⁵	Adj. R-Square 0.973	K (s ⁻¹) 2.1383x10 ⁻⁴

0.4 M	0.00956	0.02172	3.17404×10^{-4}	1.30545×10^{-5}	0.98498	3.17404×10^{-4}
0.6 M	0.01125	0.03238	4.25401×10^{-4}	2.57795×10^{-5}	0.96789	4.25401×10^{-4}

On the other hand, when the geopolymer was exposed to Ox, remarkable changes occurred in its chemical structure (Figure 3). The broad band in the geopolymer spectrum, ascribed to the asymmetric stretching vibrations of Si–O–Al and Si–O–Si bonds within the range of 900–1200 cm⁻¹ and centered initially at 1100 cm⁻¹ [20], became narrower and shifted toward lower wavenumbers (989 cm⁻¹). Since this band reflects the bond lengths and angles within the silicate network [20]—and typically broadens and intensifies during geopolymerization due to the formation of Si–O–Al and Si–O–Si linkages—the observed shift of the main Si–O–Al and Si–O–Si band to lower wavenumbers suggested a lengthening of their bonds and a related reduction in bond angle. This phenomenon could be ascribed to an increase in the proportion of silicon sites containing non-bridging oxygens. An inverse process to synthesis, where every bridging oxygen atom of the original aluminosilicate is replaced by two negatively charged non-bridging oxygen atoms linked by cations (Na⁺) to yield the final geopolymer structure [21]. Additionally, in the leached was found the presence of Si-O-Si bonds (stretching 1054 cm⁻¹ and asymmetric stretching 1088 cm⁻¹ [22]) along with Ox traces (C-O stretching (1216 and 1287 cm⁻¹) [23], symmetric stretching of COO⁻ (1404 cm⁻¹) [24] and C=O stretching bands (1691 and 1719 cm⁻¹) [23] which suggest therefore a geopolymer framework fragmentation.

In all spectra, the vibration mode ascribed to free water (3440 cm⁻¹) was identified, excepting the chemically attacked geopolymer, where the free water was removed during the characterization process. And the O–H stretching and H–O–H bending vibrations modes (between 1630 and 1644 cm⁻¹) were detected in metakaolin, and the geopolymer before and after chemical attack as part of the hydroxyl groups (OH⁻) of water. Finally, the band appearing at 1436 cm⁻¹ in the geopolymer spectrum was assigned to the asymmetric stretching vibration of carbonate (CaCO₃) groups formed during geopolymer synthesis [25].

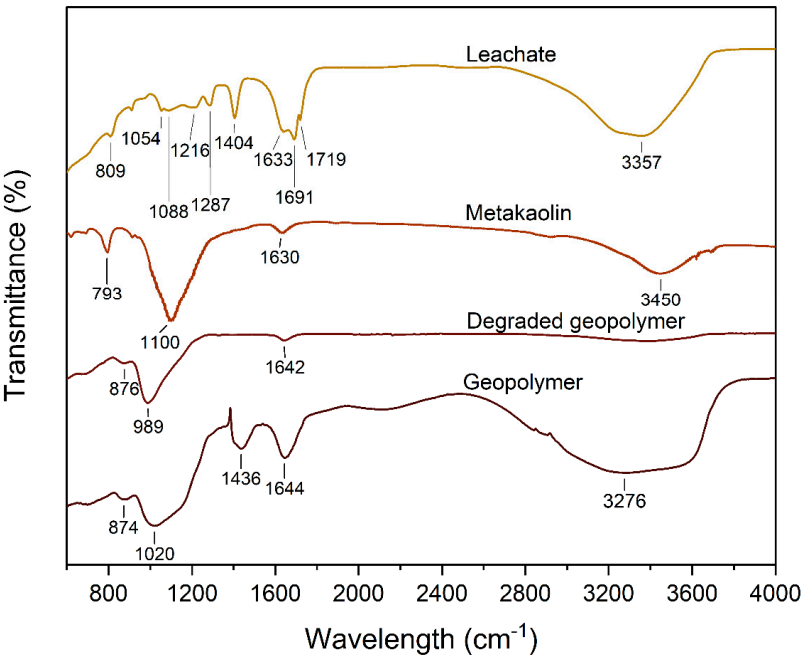


Figure 3. FT-IR spectra of metakaolin, the geopolymer before and after chemical attack by oxalic acid, and the corresponding leachate.

In the same vein, the XRD pattern of the degraded geopolymer (Figure 4) unveiled an increase in the amorphous structure. The high hump centered around $2\theta = 26^\circ$, characteristic of geopolymers and that is related to the amorphous structure, became flatter. Therefore, this phenomenon suggests the degradation of the geopolymer network, enabling the identification of anatase, aragonite, and quartz which are originally present in the geopolymer and in the metakaolin precursor. The same spectrum revealed the presence of calcite, indicative of carbonation resulting from the geopolymer's exposure to atmospheric CO_2 .

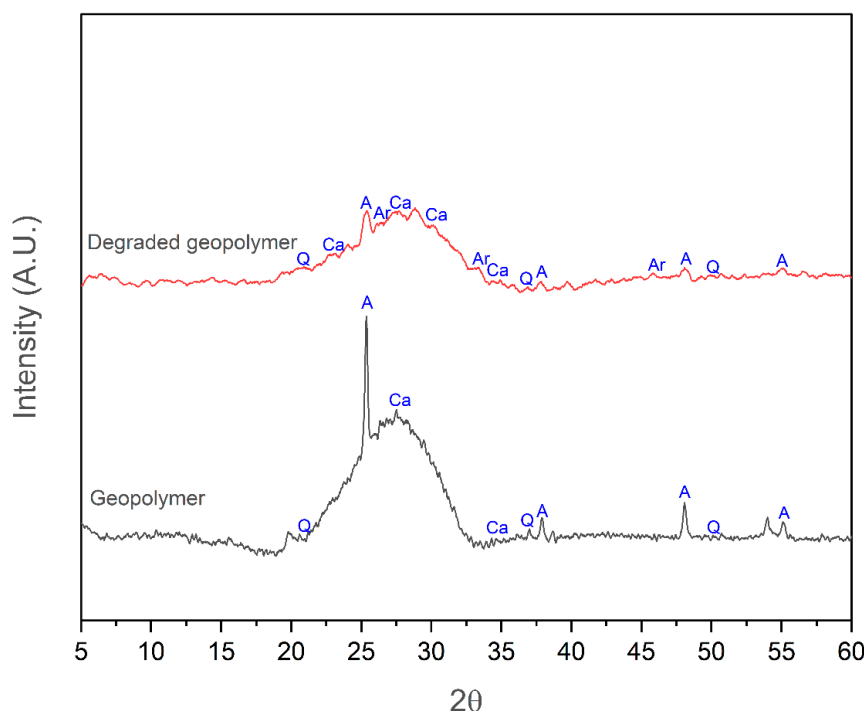


Figure 4. Comparison of X-Ray diffraction pattern between geopolymer and geopolymer once chemically attacked by Ox. It is observed that a flattening of the hump is centered at $2\theta = 26^\circ$ and is associated with the presence of crystalline phases. A. Anatase ($2\theta = 25.28^\circ, 37.81^\circ, 47.99^\circ, 53.95^\circ, 55^\circ, 62.9^\circ$ and 75° ; JCPDS-ICDD card: 21-1272), Q. Quartz ($2\theta = 20.86^\circ, 26.64^\circ, 36.54^\circ, 50.13^\circ$ and 59.94° ; JCPDS-ICDD card: 46-1045), Ar. Aragonite ($2\theta = 26.3^\circ, 33.2^\circ$ and 45.9° ; JCPDS-ICDD card: 41-1475) and Ca. Calcite ($2\theta = 23^\circ, 27.5^\circ, 30.1^\circ, 34.6^\circ, 38.3^\circ, 43.7^\circ$ and 46.2° ; JCPDS-ICDD card: 01-081-2027).

Finally, in Figure 5, the comparative analysis of chemical composition and morphology between the geopolymer before and after chemical attack by Ox is shown. In its unaltered state (Figure 5a), the geopolymer exhibited a compact and homogeneous morphology. Meanwhile, after exposure to Ox (Figure 5e), the surface became noticeably more irregular and fragmented, indicating structural degradation of the network as a consequence of the chemical interaction.

The theoretical atomic ratios of the main elements in the geopolymer were $\text{Si}/\text{Al} = 1.5$, $\text{Na}/\text{Al} = 0.7$, and $\text{Na}/\text{Si} = 0.466$. However, the values experimentally determined by energy-dispersive X-ray spectroscopy (EDS) deviated from these theoretical ratios after the chemical attack. Specifically, the Si/Al ratio increased from 1.483 ± 0.301 to 1.92 ± 0.179 , while the Na/Al and Na/Si ratios decreased from 0.778 ± 0.114 to 0.484 ± 0.096 and from 0.42 ± 0.038 to 0.249 ± 0.032 , respectively. These variations suggest a selective leaching of sodium ions from the geopolymer framework.

A detailed compositional analysis of the three main framework elements (Al, Si, and Na), supported by an analysis of variance (ANOVA) at a 0.05 significance level (Figure 5i), confirmed a statistically significant difference in the sodium content between geopolymer and the Ox-attacked geopolymer (Figures 5d and 5h). Conversely, the concentrations of aluminum and silicon remained

statistically invariant, as shown in Figures 5b, 5c, 5f, and 5g, indicating that the aluminosilicate backbone was preserved despite the sodium depletion.

Overall, these results point to a degradation mechanism primarily governed by the loss of cations (Na^+) from the geopolymer structure upon exposure to Ox, leading to damage of the aluminosilicate network. This process results in a weakening of the charge-balancing interactions between Na^+ cations and the Si-O-Al network, thereby promoting structural fragmentation and reducing the mechanical integrity of the geopolymer.

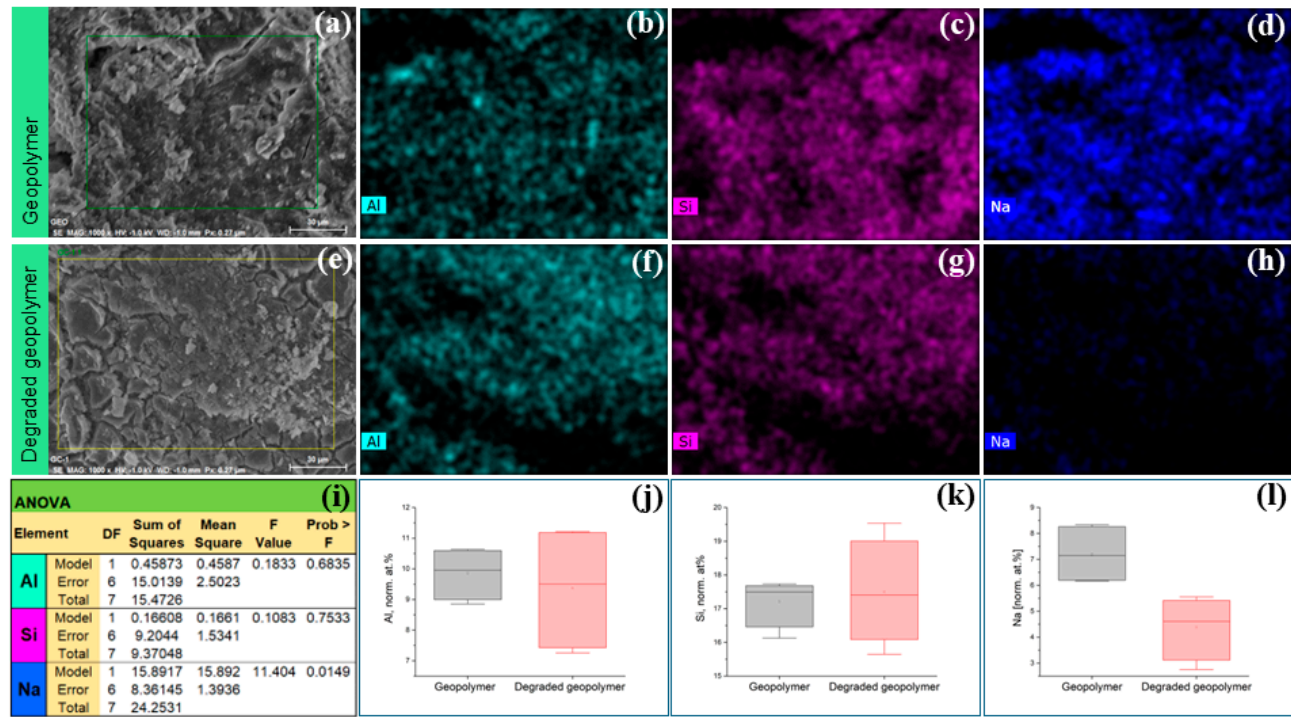


Figure 5. Geopolymer morphology and its chemical change as result of the Ox attack. Along with an analysis of variance (ANOVA) for three main framework elements (Al, Si, and Na) present in samples.

4. Discussion

Recent studies have investigated the use of oxalic acid’s role in various applications, but understanding its impact on geopolymers is crucial for materials scientists and civil engineering concerned with durability in oxalate-rich environments. Addressing this gap can inform safer, more resilient construction materials.

In this study, we evaluated how geopolymers degraded under Ox exposure. Our results clearly show that the degradation behavior follows zero-order kinetics, where the rate of degradation is independent of the remaining geopolymer concentration and primarily depends on Ox concentration, suggesting afterwards, that the chemical attack is controlled by the availability of the geopolymer surface to Ox.

Compositional analysis (EDS) and FT-IR spectroscopy of chemically attacked geopolymer revealed a significant loss of Na^+ cations, accompanied by shifts of Si-O-Al and Si-O-Si stretching vibration bands to lower wavenumbers. These spectral changes reflect increased bond lengths and relaxation of bond angles within the geopolymer network. The detection of Si-O-Si species in the leachate supports this interpretation, indicating that Ox promotes dissolution of structural units such as Si-O-Al and Si-O-Si, disrupting the framework and leading to bond distortion, network fragmentation, and ultimately a reduction in mechanical integrity.

Morphological observations and XRD analysis corroborate these findings. After Ox attack, the geopolymer surface became irregular and fragmented, and the characteristic amorphous hump at 2θ

= 26° flattened. This loss of short-range order confirms partial breakdown of the network, consistent with the chemical and spectroscopic results.

5. Conclusions

Overall, these results demonstrate that Ox exposure produces significant alterations in geopolymer structure, with Na⁺ leaching identified as the primary degradation mechanism. This process leads to progressive structural weakening and morphological fragmentation and follows zero-order kinetics, which indicates that the degradation rate depends solely on Ox concentration.

These findings highlight geopolymer's susceptibility to organic acid attack and underscore the need to consider such interactions when assessing long-term performance. Further research on cation stabilization could enhance the chemical durability of geopolymers in natural and aggressive environments.

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