A critical review on the durability of geopolymer composites in acidic environment

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Abstract. Deterioration of concrete in acidic environments result in premature degradation in terms of microstructural alteration of phases leading to mass changes, weakening of mechanical properties, increase in porosity due to calcium leaching etc. Industries are found to dispose acidic effluents directly into the environment without proper treatments. Again, these acids can be organic as well as inorganic acids. Organic acids unlike inorganic acids are found to be weak acids due to their partly dissociative nature. The mechanism of acid attack varies based on the acid type and the characteristics of the calcium salt that are formed. Conventional concrete made with Ordinary Portland Cement (OPC) are not resistant to acids. Also, as we strive towards sustainable development, alkali activated or geopolymer concrete has started to gain attention as it is found to have better mechanical properties and durability comparing to conventional concrete. This paper reviews the damage mechanisms of sulphuric acid, citric acid, nitric acid and acetic acid on the alkali activated binders.

1. Introduction

Concrete is a handed down material which is been extensively used worldwide in construction industry. Concrete structures are exposed to aggressive environments due to growing industrial activities in urban areas. Acid attack on concrete is a wide area in which numerous researches has taken place due the continuous deterioration of concrete structures caused by these aggressive species over time. Concrete is found to be in disequilibrium with its environment due to its alkaline nature. For the hydrated compounds to be stable there should be proper defined ranges of concentration for Ca\textsuperscript{2+} and OH\textsuperscript{-} ions. As the pH of the solution decreases, the hydrated compounds undergo hydrolytic decomposition causing distortion of the cement matrix and thus leading to degradation of concrete.

The acids that attack the concrete can be either organic or inorganic in nature. Organic acid attack is found to be more complex in agro based and agricultural industries. Biogenic sulphuric acid corrosion is one of the most rapid causes of concrete degradation. Over the period from 2002 to 2022 the United States alone, has estimated $390 billion to repair existing wastewater infrastructure due to the biogenic corrosion \cite{1}. Penetration of these aggressive species can affect the pore network, physical and mechanical properties of the structure. The type of acid, its concentration, pH, solubility of salts formed are some of the major factors that influence the aggressiveness of these species. Both organic and inorganic acids have its deterioration effect on structures but with differences in its mechanism of degradation.

As of now, enormous amount of research work is carried out to replace concrete with new construction materials emphasizing on sustainability, reduced carbon footprints, durability and eco-friendliness. Alkali activated or Geopolymer concrete has started to gain attention as it is found to
have better mechanical properties and durability comparing to conventional concrete. The binders used should be rich in silica and alumina with lower calcium content. Blast furnace slag, coal derived fly ash, calcined clays and natural pozzolans are some of the binders found to give good results [2]. But more amount of research work is recommended for implementing geopolymer concrete practically due to its workability and setting time issues. Although geopolymer technology has certain disadvantages, it has potential to be a low carbon footprint material alternative to the conventional concrete. Hence, this paper exclusively depicts about the degradation mechanisms of various acids on geopolymer composites.

1.1 Sources of acid attack
Acid attack is very common in industries which use chemical acids for their various processing. Leakage and accidental spillage of acids in such industries will result in rapid degradation of concrete of acids are coming into contact with concrete components. Sulphuric acid is considered to be one of the main strong sources of concrete degradation. Generally, in a sewer collection system there is presence of aerobic bacterium which reacts with hydrogen sulphide (H$_2$S) gas to produce sulphuric acid (H$_2$SO$_4$). These acids then penetrate into the concrete causing leaching which eventually leads to deterioration of concrete. The concrete inside biogas reactors digesters are also prone to deterioration due to the production of organic acids and ammonium in the liquid phase and H$_2$S and CO$_2$ gases in the gas phase. The fertilizer manufacturing industries use nitric acid and sulphuric acid in the production of ammonium nitrate and superphosphate fertilizers [3]. Industrial emissions of sulphur and nitrogen compounds, can be later converted to sulphuric and nitric acids and lead to acid precipitations with a pH level ranging from 5.0 to 3.0. Acidic rain or mist is then another aggressive agent for concrete structures [4].

In agricultural and agro food industries, often the collection, storage and treatment systems are made of concrete. The effluents from agricultural and agro food industries are mostly the source of organic acids which can then reacts with the concrete matrix. On an average, 8–15 litres of spent wash effluent are generated for every litre of alcohol produced. India has about 620 sugar mills and about 400 molasses based distilleries with an installed capacity of 400 billion litres of alcohol and a potential to generate, on an average, 40 billion litres of spent wash annually [4]. These effluents contain a mix of organic and sulphuric acids. Liquid manure and silage effluents contain acetic acid, lactic acid and butyric acid. From the distillery and fermentation industry, we have winery wastewaters where the pH is around 4.5 and it contains acetic, lactic, citric and tartaric acids [5].

Silage acids cause most of the deterioration problems in conventional cast-in-place and precast concrete silos. Larger silos are more prone to acid deterioration than smaller silos due to the increased horizontal pressure [6]. When moist plant material is placed in a silo, it goes through the ensiling process which produces silage acids, namely lactic and acetic. These acids, on contact with concrete silo walls, react with the cement matrix, causing a decrease in strength with increase in time of exposure. Silage acids can decrease the strength of a concrete silo wall which leads to the buckling of walls and eventually leading to failure. Thus, it can be seen that acid attack is a growing durability issue which needs to be tackled by formulating a concrete mix for enhancing the service life of such structures. Alkali activated binders or geopolymer technology is a promising alternative to attain the same and is discussed in the section below.

1.2 Geopolymer composites
Geopolymers are inorganic polymers which are amorphous in nature rather than crystalline. They are considered to be a subset of alkali activated binder system. Alkali activation is the generic term which is applied to the reaction of a solid aluminosilicate (termed the ‘precursor’) under alkaline conditions (induced by the ‘alkali activator’), to produce a hardened binder which is based on a combination of hydrous alkali-aluminosilicate and/or alkali-alkali earth-aluminosilicate phases [2]. Geopolymerization is an exothermic reaction where reaction takes place between silica (Si)-alumina (Al) under alkaline condition which subsequently create three-dimensional polymeric chain of Si-O-Al-O bonds.
Geopolymerization comprises a three stage reaction i.e. Dissolution – Coagulation, Coagulation – Condensation, Condensation – Polymerization [7]. The rate of reaction is influenced by the polycondensation rather than dissolution. Ancient research works used geopolymers as coating for fire protection of cruise ships, heat resistant adhesive, thermal protection of wooden structures etc. Geopolymers differ in their reactivity and availability.

The source materials for geopolymers must be rich in aluminium and silica. The materials should be amorphous in nature as the degree of polymerization depends on it. Geopolymer precursors could be natural pozzolanic materials like volcanic ash, diatomaceous earth, shales, zeolite, kaolinite, phonolite etc. or artificial raw materials that include materials from industrial or agricultural waste such as low calcium fly ash, silica fume, brick powder, granulated blast furnace slag, sugarcane bagasse ash, rice husk ash, red mud, fluorescent lamp wastes, ceramic wastes etc. The activators used in aluminosilicate based precursors are alkaline hydroxides, alkaline silicates or blends of the two, to generate high alkalinity. Commonly used alkali activators are sodium hydroxide (NaOH) / potassium hydroxide (KOH) and sodium silicate (Na₂SiO₃) / potassium silicate (K₂SiO₃). The concentration and molarity of the solution influences the paste properties. Combination of NaOH and Na₂SiO₃ is preferred because for Na₂SiO₃ to be reactive, it has to be depolymerised first by NaOH. The rate of reaction is more influenced by polycondensation step than dissolution. Potassium ions require larger effort to penetrate through the paste due to its larger size than sodium ions and hence sodium based alkali activators are widely used [2,3]. Alkali activator solutions are difficult to handle in large amounts as it is viscous, corrosive, and hazardous. Solid NaOH is found corrosive and forms sodium carbonate when exposed to CO₂.

Sodium aluminosilicate hydrate (N-A-S-H) gel, the main reaction product of the alkali-activated aluminosilicates, differs of the aluminium-modified calcium silicate hydrate (C-A-S-H) gel of PC pastes. Fly ash and slag are found to be the most potential resources of geopolymer among the waste products. High concentration of calcium ion in class C Fly ash based geopolymers can result in higher compression strength [8]. But the use of class C fly ash based precursors was found as the causation of rapid setting. Gel composition largely affects the mechanical strength and durability. The main hydration product is a calcium silicate hydrate (C-A-S-H gel) with aluminium in its composition. The structure and composition of C-A-S-H gel and the presence of other secondary phases or compounds depend on the type and amount of activator used, slag structure and composition and the curing conditions in which the material hardens [9]. When slag was partly replaced by Ca(OH)₂, workability and compressive strength was found to be reduced [10].

Due to the formation of sodium calcium silicate hydrate slag-based geopolymer concrete had higher expansion than fly-ash based geopolymer concrete. C-A-S-H gels are formed by the alkaline activation of high silica, high-calcium materials such as slag and N-A-S-H gels are formed by alkaline activation of high-silica, high-alumina materials such as fly ash and metakaolin. The combustion of agricultural wastes produces rice hull and husk ashes as residues that are rich in high reactive silica content. China being the largest contributor of rice husk with an estimated output to be around 80 MT. One-part geopolymer rice husk mixes activated by solid sodium aluminate was found to have relatively high compressive strength (30 MPa). While these ashes sometimes contain a high amount of unburned carbon, it was shown that low-quality rice husk ashes (loss on ignition of 40%) could be successfully utilized in one-part geopolymers [10]. Vincent et al. [11] used alkali activated materials obtained from asphalt fillers and fluorescent lamps to check the suitability of using it for urban pavement. As fluorescent lamp wastes were rich in SiO₂, the bending strength of compositions were raised unlike asphalt filler wastes which had constituents of CaO and MgO. The increase in SiO₂/Al₂O₃ ratio had better positive effects on strength unlike asphalt fillers.

Alkali activated binders can be produced as either a one-part mix system or a two-part mix system. Two-part mixture is normally used for precast construction works by most industries as handling of chemicals and curative regime can be closely controlled efficiently. In two-part mix system, in addition to water, a solid aluminosilicate precursor and an alkaline activator solution is required. Figure 1 represents a one-part mix system where in addition to water to a dry solid mixture is required.
Figure 1. Preparation of one-part geopolymer [10]

From figure 1, it can be noted that the dry mixture is prepared by mixing a solid alkali-activator along with a solid aluminosilicate precursor. Also the preparation of the dry mixture can take place with or without the calcination step. The activator in a one part geopolymer mix can be any substance that provides alkali cations, raises the pH of the reaction mixture, and facilitates dissolution. For conventional geopolymers, alkaline activators can be concentrated aqueous solution of alkali hydroxide, silicate, carbonate, or sulphate [10]. Normally, one-part mix precursors used are fly ash (class F) in combination with blast furnace slag. Class C fly ash is not highly recommended as it causes rapid setting due to high amount of calcium content. In some researches where mechano chemical activation method was used in which fly ash was ball-milled with dry blended activators are found to have high strength, increased resistance to moisture and fine microstructure. One-part mixture is found to have issues related to strength i.e. their strength is found to be lower unlike two-part mixtures [2]. If issues related to these can be resolved, then one-part mix could be an industrial material as it is dry, it can be packed into bags and transported efficiently.

The geopolymerization process normally depends on the mode of curing. The method of curing adopted have significant impact on the thermal properties of geopolymer concrete, micro-structural and strength development characteristics. The curing temperatures between 40°C to 85°C is found to be almost perfect for geopolymerization. For fly ash based geopolymers, temperature curing or oven curing is generally adopted. Nuruddin et al. [12] reported that for an alkaline activated fly ash, curing temperature is very vital for achieving higher strength and the specimens subjected to higher curing temperature exhibited higher mechanical strength than those of lower temperature. They also observed that the strength increased with increase in curing duration but the increase of strength was found negligible when curing time was extended beyond 24 hours. In the case of geopolymer concrete made with fly ash and activated by a solution of sodium hydroxide and sodium silicate, the results showed that seven-day strength of the oven cured specimen is almost six times greater than that of ambient cured specimens. They have found that strength of geopolymer concrete improves at higher temperature and the optimum strength was found to be 80°C temperature for steam curing, while for water curing, the strength obtained after 28 days was less than the characteristic strength due to the low development of strength at lower temperature. Slag based geopolymer were found to exhibit lower strength at ambient temperature compared to the steam cured specimens. However, curing at advanced elevated temperature for a prolonged period causes deterioration of the specimens due to the thermoanalysis of silicate Si–O–Al–O bond [12].

2. Mechanism of acid attack
Acid attack results in the decalcification of hydration products which are highly unstable in low pH acidic solutions containing acid (H+) ions which then affects the chemical stability of the structure. The transfer of aggressive species into the concrete depends upon characteristics such as pore network,
dimensions, connectivity of capillary porosity and chemical nature of hydrated phases. The aggressiveness of the acids into the cementitious matrix depends on their solubility in water, the acid dissociation constant and mono/poly acidity of acids and the physical properties of salts. Weak acids such as organic acids dissociate only partially as compared to strong acids. The dissociation of strong acid (HA) is complete when the solution contains only H⁺ and A⁻ ions and no HA [4,13]. The extent to which dissociation occurs is translated by the acid dissociation constant pKₐ. It is observed that more negative the pKₐ of an acid, the greater its strength.

The structural integrity of geopolymer systems relies on alumina-silicate rather than calcium silicate hydrate bond. Sodium aluminosilicate hydrate (N-A-S-H) gel is the main reaction product of the alkali-activated aluminosilicates. The ion exchange reaction between cations and hydronium ions are followed by an electrophilic attack of acid protons on Si-O-Al-O bonds. On exposure to acid, the sodium ions that act as charge compensation cations of the aluminosilicate framework, are leached out and exchanged by hydronium ions from the solution. The atomic ratio of Si-to-Al is then found to increase, indicating that the tetrahedral Al from the aluminosilicate framework is dissolved completely by breaking the Si-O-Al bond [1,14].

Lodeiro et al. [9] has stated that at low pH, a fully Ca-exchanged N-A-S-H may appear to have a C-A-S-H composition, but in this case, it retains its 3D aluminosilicate framework structure and is easily distinguishable from a 2D C-A-S-H gel. The acid resistance of the strength-forming phases was linked to the binder composition. Slag based alkali activated binders include the C-A-S-H phase with an enhanced acid resistance compared to ordinary C-S-H phase. During acid exposure of alkali activated fly ash specimen, the alkalis are leached out from the pore solution as well as from the crystal structure of the N-A-S-H phases. Koenig et al. [15] has found that the acid resistance increased with a decrease in CaO content i.e. C-S-H < C-A-S-H < N-A-S-H.

3. Acid resistance of geopolymer composites

3.1 Sulphuric acid attack

The sulphuric acid first reacts with the calcium hydroxide present in concrete to produce gypsum (CaSO₄.2H₂O). Gypsum is not much soluble in acid solution and remains as a precipitate in the concrete surface thus reducing the penetration of acid in the early exposure period. This gypsum produced reacts with the calcium aluminate hydrate present in the cement to produce ettringite (3CaO.Al₂O₃.3CaSO₄.32H₂O), which is very detrimental to the hardened concrete. The substantial mass gain of all mixes on exposure to sulphuric acid, especially at the early age of exposure, was attributed to the gypsum formation around the specimens. The interfacial transition zone (ITZ) with fine aggregates of an Alkali activated (AA) sample after immersion in sulphuric acid showed similar structure in morphology from that before immersion, indicating that AA paste remained in good form under sulphuric acid attack. In the study carried out by Gu et al. [1], figure 2(a) demarcates the initial scanning electron microscopy (SEM) analysis of AA fly ash matrix consisting of a dense structure of sodium aluminosilicate hydrate (N-A-S-H) before immersion where the atomic ratio of silicon and aluminium was 1.5. The sphere like structure are the unreacted fly ash in the initial stage.
From figure 2(b), the AA fly ash matrix displayed no significant difference in its matrix after sulphuric acid attack. The dense structure remained with the presence of some micro-cracks. As N-A-S-H was dissolved on reaction with sulphuric acid, the atomic ratio of silicon and aluminium had increased to 2.3. For higher concentration of sulphuric acid, it was found to dissolve N-A-S-H gel thus reducing the compressive strength of the AA concrete. Reju [3] noticed that the process of deterioration progressed to the interior with increase in exposure time. The altered depth variation of specimens before and after acid attack is shown in figure 3. Phenolphthalein indicator was sprayed on the freshly cut surface. The pink colour region shows the unaffected regions on exposed to acids.

![Figure 2 SEM images of AA concrete sample in non-accelerated test](image)

**Figure 2** SEM images of AA concrete sample in non-accelerated test

In contrast to other acids, specimens immersed in sulphuric acid (concentration of 0.5M) shows a colourless surface indicating that they have been degraded completely due to acid attack. The fly ash based geopolymers (FGP) under visual examination showed no changes in colour or deposition of
salts on the specimens whereas slag based geopolymers (SGP) showed a change in colour. The mass reduction of FGP specimens was lesser than that of OPC indicating that the geopolymer specimens are more acid resistant than others [3]. No mass loss was found for SGP specimens exposed to 0.25M acid. FGP specimens proved to be better resistant towards sulphuric acid attack due to its better structural framework obtained after high temperature curing.

3.2 Citric acid attack

Citric acid was found to be the most aggressive among organic acids and there is rapid loss of thickness. The high concentration used, polyacidity of citric acid and the non-protective nature of precipitate formed seems to have increased the aggressiveness of citric acid. According to Koeing et al. [15], the higher damaging effect could also be due to acid buffer action exhibited by organic acids. According to Ramaswamy and Santhanam [16], the degradation kinetics in citric acid is highly dependent on its concentration. A white-coloured salt is precipitated in substantial quantities on specimens immersed in citric acid and is confirmed as calcium citrate tetrahydrate \((\text{Ca}_3(\text{C}_6\text{H}_5\text{O}_7)_2\cdot4\text{H}_2\text{O})\) by X-ray diffraction study [16]. This salt has less solubility and very high molar volume compared to portlandite and C-S-H gel and thus found to be detrimental to the cement matrix.

In the study by Reju [3], continuous exposure of FGP specimens in citric acid did not show any structural disintegration. Severe cracking of SGP paste specimens were observed due to the formation of low soluble loosely adhered white precipitate. SGP specimens from figure 3 are found to have lower rate of degradation as a pink colour is found towards the core. However, results from the microstructural analysis showed that, FGP specimens has a better performance in resisting citric acid as no expansive products were formed unlike OPC and SGP.

3.3 Nitric and hydrochloric acid attack

Nitric acid attack is a typical acidic corrosion causing volume reduction of the corroded layer due to the leaching of highly soluble calcium nitrate salt that is formed. Blast furnace slag-fly ash based geopolymers were corroded when immersed in nitric acid solution for a period of one month. This was due to ion exchange reactions between the charge compensating cations of the framework (i.e., sodium, potassium or calcium) and the H+ ions present in solution. It may also be due to the electrophilic attack of polymeric Si–O–Al bonds by acid protons, causing the ejection of tetrahedral aluminium from the aluminosilicate framework. It is observed that, all mixes had a mass loss due to progressive lixiviation of the hydrated phases because of the predominance of leaching in nitric acid attack. Provis and Deventor [17], studied the weight loss of tungsten mine waste mud binders (TMWM) and OPC specimens after exposed to different acids and is shown in figure 4.

![Figure 4 Weight loss on exposed to acids](image-url)
The worst case scenario from the above figure is when OPC specimens are subjected to hydrochloric acid as it reacts with calcium compounds (such as Portlandite and CSH gel) leading to the formation of calcium chloride, which has extremely high solubility in solution. Geopolymeric mine waste binders have low acid resistance performance when compared to geopolymeric binders based on metakaolin [17] but overall geopolymer binders had better resistance when compared to OPC.

3.4 Acetic acid attack
Acetic acids are found mainly in waste waters and it is found to be aggressive when it comes to acid attack. The corrosion process is rapid comparable to that of strong acids such as sulphuric acid but lesser aggressive compared to citric acid at equivalent concentrations. These acids produce soluble calcium salts by dissolution of calcium hydroxide in concrete. In the case of OPC specimens, orange yellow colour was observed on the specimens without any precipitation of salts. No precipitation or change in thickness was observed for geopolymer specimens even in 0.5 M or 0.25 M acetic acid solutions [3]. The altered depth variation was found to be visible in OPC and SGP specimens (figure 3).

FGP specimens showed superior performance in acid resistance with respect to mass reduction (0.8 %) in mortar specimens exposed to 0.5 M acetic acid. Mass loss for SGP specimen was found to be higher than FGP specimens. In SGP concrete, lower CH content and the C/S ratio in the hydration products and the formation of a silica (alumino) gel upon decalcification hinders the further acid ingress and contributes towards the greater acid resistance of SGP concrete. Ingression of acids are obstructed also due to the finer pore structure of SGP concrete. Koenig et al. [15] on assessment of the degradation kinetics of alkali activated concrete exposed to organic acid attack proved that the residual strength was found to decrease with an increasing Ca-content of the binder. The organic acid studied was a mix of acetic, lactic and propionic acid with a solution pH of 3. The residual compressive strength of the specimens subjected to organic acid attack is illustrated in figure 5.

There is a continuous decrease in strength over time, for all the specimens immersed in the organic acid solution, except for AAFA (alkali activated fly ash). Acid exposed alkali activated fly ash specimens displayed a steady increase in strength over time. Reju [3] also observed improved performance of fly ash based geopolymer when exposed to acetic acid solution.

4. Conclusion
Acid attack is a complex phenomenon arising in construction industries worldwide as it is responsible for the deterioration of concrete in acidic environments resulting in premature degradation in terms of
microstructural alteration of phases manifesting in the form of mass changes, weakening of mechanical properties, increase in porosity due to calcium leaching etc. This complicated mechanism of degradation by various acids is still not properly understood and further clarification is necessary in this regard. Currently, there are no codes or standards available for evaluating resistance of materials to acid attack. Further studies are necessary to investigate and clarify the mechanisms of degradation and alteration kinetics in these conditions by developing reliable test methods and thus, to design materials that perform well in these environments.

Both organic acids as well as inorganic acids differ in their mechanism of deterioration of concrete. The rate of degradation is found to be dependent on the type of acid, concentration of acid, chemical composition of binders and solubility and characteristics of the salts formed. Based on the aggressiveness of acids, sulphuric acid and citric acid are found to be the most aggressive from most of the studies. On exposure to sulphuric and citric acid, expansive salts with high molar volume were formed causing rapid degradation. Fly ash based geopolymer showed highly improved performance against sulphuric, acetic and citric acid attack. Nitric acid attack on metakaolin geopolymer did not have severe degradation on comparison to other waste binders used. Fly ash-slag based geopolymer composites cured in ambient conditions had comparable performance to that of fly ash based geopolymers cured at high temperature. The additions of small amounts of slag to fly ash based geopolymers are also found to enhance its compressive strength on exposure to acids.

The increased expansion of urban areas and proliferation of industries have led to the large scale generation of acidic media. This has led to severe degradation of concrete structures made with OPC when it gets in contact with the acidic solutions. As of now, large numbers of researches are carried out in replacing cement in concrete with new binders emphasising on sustainability, reduced carbon footprints, durability and eco-friendliness. Reinforcing the use of supplementary cementitious materials (SCMs) as alkali activated binders would be a better solution in offering a sustainable concrete (100% replacement of cement), and improved resistance against harsh aggressive environments. From most of the studies, it is evident that durability performance of geopolymers are found to be superior. Thus alkali activated binder systems can offer a sustainable and durable alternative to Portland cement based systems for exposure in aggressive acid environments. Although branded as weak acids, acetic acid could lead to rapid increase in the altered depth of specimens and the durability of various binder systems in these acidic media are not often studied in detail. Evolution of mechanism for acid attack and prediction of degraded depth for a given duration, study of effect of mixture of acids and effect of type of curing on the rate of reactions are some of the further research works that need to be looked into to understand the broader aspects of acid attack and its degradation mechanisms.

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