

# Resistance of geopolymer materials to acid attack

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## Abstract

This article presents an investigation into durability of geopolymer materials manufactured using a class F fly ash (FA) and alkaline activators when exposed to 5% solutions of acetic and sulfuric acids. The main parameters studied were the evolution of weight, compressive strength, products of degradation and microstructural changes. The degradation was studied using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The performance of geopolymer materials when exposed to acid solutions was superior to ordinary Portland cement (OPC) paste. However, significant degradation of strength was observed in some geopolymer materials prepared with sodium silicate and with a mixture of sodium hydroxide and potassium hydroxide as activators. The deterioration observed was connected to depolymerisation of the aluminosilicate polymers in acidic media and formation of zeolites, which in some cases lead to a significant loss of strength. The best performance was observed in the geopolymer material prepared with sodium hydroxide and cured at elevated temperature, which was attributed to a more stable cross-linked aluminosilicate polymer structure formed in this material.

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## 1. Introduction

Acid resistance is a desirable property for structural materials used in the aggressive environment of chemical, mining, mineral processing and other industries. Basic in nature, concretes made with Portland cement and alkali-activated slag deteriorate in the acid environment [1,2]. In the case of acid attack on ordinary Portland cement (OPC) concrete, calcium salts of the attacking acid rapidly form and the concrete loses its strength and deteriorates quickly. When tested in acetic acid solution of pH 4 for 12 months, AAS concrete had about 33% strength reduction as compared to 47% reduction in OPC. Thus, AAS was found superior in durability to OPC. Its good performance was attributed to low Ca content (~ 40% CaO) compared to Portland cement (~ 65% CaO) and the glassy state of the slag, which is poorly soluble in the acid solution [2]. Geopolymer materials prepared with the class F fly ash (FA) contain very low calcium (3–4% CaO) and it could be expected that they possess high durability in the acid environment.

Geopolymers are synthetic minerals belonging to the same family of aluminosilicates as zeolites, but unlike zeolites, they are essentially amorphous polymers. The properties of these geopolymer materials prepared using FA are not well studied but there are reports of superior durability and heat-resistant properties of geopolymer materials prepared using metakaolin [3–5]. Geopolymer materials are produced by a sol–gel process utilising alumina and silica oxides activated by alkali hydroxides and/or alkali silicates. The starting materials dissolve in high pH alkaline solution and the geopolymers are precipitated; this process being facilitated by heat. In the process of the polymerisation reactions, polysialates, polysialate siloxo, and polysialate disiloxo are formed [3]. The mineral polymers have empirical formula:  $M_n[-(SiO_2)_z-AlO_2]_n \cdot wH_2O$ , where  $z$  is 1, 2 or 3;  $M$  is an alkali cation, such as potassium or sodium, and  $n$  is the degree of polymerisation [4,5]. In a previous article, the processing and characterisation of geopolymer materials made using class F FA and formed at elevated temperature were discussed [6]. This article presents a study of durability in the acid environment of three geopolymer materials utilising class F FA activated by sodium silicate, sodium hydroxide and a mixture of sodium and potassium hydroxides.

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## 2. Experimental

### 2.1. Materials

The chemical and mineral compositions of FA are shown in Table 1 and Fig. 1, respectively. FA used was sourced from Gladstone in Queensland, Australia. It is mainly glassy with some crystalline inclusions of mullite, hematite and quartz. Laboratory grade sodium silicate solution type D with Ms (ratio of silica to sodium oxide) equal to 2, and 14.7% Na<sub>2</sub>O and 29.4% SiO<sub>2</sub> was supplied by PQ Australia, while 60% w/v sodium hydroxide solution was supplied by Sigma. Potassium hydroxide pellets were supplied by Aldrich. Sodium hydroxide, potassium hydroxide, and sodium silicate solutions were used for FA activation.

### 2.2. Test procedures

FA was activated by sodium hydroxide, a mixture of sodium and potassium hydroxides and sodium silicate solutions, providing 8–9% Na in the mixtures and water/binder (w/b) ratio of 0.3. The preparation process and composition of the investigated materials were developed in a previous article [6]. Concentration of alkalis and w/b ratio in the materials was similar to corresponding numbers of geopolymer materials studied in [7–9]. Table 2 shows the details of the samples and curing conditions. The pastes were cast in plastic cylinders and sealed with the lid. The mixtures were cured for 24 h at room temperature, after which temperature was ramped to 95 °C and maintained at this level for 24 h. The materials were then cooled down to room temperature in the oven for 2 days, and initial compressive strengths was determined (8FAK, 56–58 MPa; 8FASS, 66 MPa; 8FA, 57–59.5 MPa). The samples were then exposed to immersion tests.

The resistance of materials to acid attack was studied by immersion of cylindrical specimens (Ø25 × 50 mm) in 5% solutions of acetic and sulfuric acids. The strength of acid solutions used in the test was not equal. The solution of sulfuric acid had pH 0.8 and was much stronger than solution of acetic acid, which had pH of 2.4. The choice of acids solutions and their concentrations were based on practical utilisation of concrete as a construction material in sewage pipes, mining, and food processing industries. The testing media were replaced monthly with fresh solutions. The compressive strength of cylinders (Ø25 × 50 mm) was

measured at 30, 60, 90, 120 and 150 days of exposure. Neat Portland cement paste (OPC) and Portland cement paste with 20% FA replacement (OPC+FA) with w/b ratio 0.4 were used for comparison in the tests. The specimens containing Portland cement at w/b=0.4 had the same consistency as the geopolymer specimens at w/b=0.3. Thus, the specimens were compared as having the same consistency at the time of moulding. The compressive strengths of OPC and OPC+FA specimens at the age of 2 months, when they were used in immersion tests, were 45 and 42.9 MPa, respectively.

The deterioration was studied by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). For the analysis, the samples were taken from the surface (1–2 mm depth) exposed to solutions. XRD analyses were made using a Rigaku Geigerflex D-max II automated diffractometer with the following conditions: 40 kV, 22.5 mA, Cu–K $\alpha$  radiation. The XRD patterns were obtained by scanning at 0.1° (2 $\theta$ ) per min and in steps of 0.05° (2 $\theta$ ). SEM (Hitachi S-2300, Japan) was used for microstructural observations of the fracture surfaces, which were coated with evaporated gold for examination in the secondary electron mode. FTIR was performed on the samples using a Perkin Elmer 1600 spectrometer and the KBr pellet technique (3 mg powder sample mixed with 100 mg KBr).

## 3. Results

Geopolymer specimens had very small change in appearance after 5 months of immersion in the acidic solutions. Some softening of the surface cover and insignificant lightening of the colour could be noticed in the 8FA, 8FASS and 8FAK geopolymer specimens after exposure to the solution of sulfuric acid. In the acetic acid, there was no change in appearance of 8FAK specimens and very small change in appearance of 8FASS and 8FA specimens.

Visual examination of specimens exposed to the sulfuric acid solution showed severe deterioration of the OPC and OPC+FA specimens that is consistent with high content of calcium in these samples. Within days, OPC samples had a thick layer of white paste formed on the surface. After 2 weeks, the surface layer of the samples was converted to some reaction products to a depth of 8 mm. After 1 month, OPC samples were severely deteriorated. The surface layer

Table 1  
Composition of dry materials (mass %) by XRF

Oxide	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	MnO	SO <sub>3</sub>	Loss on ignition
FA <sup>a</sup>	50.0	28.0	12.0	3.5	1.3	0.7	0.2	3.2	0.42	0.03	0.12	0.53
Portland cement <sup>b</sup>	19.9	4.62	3.97	64.27	1.73	0.57	0.15	0.23	0.12	0.06	2.56	2.9

<sup>a</sup> Pozzolanic Enterprises, Port Melbourne, Australia.

<sup>b</sup> Type I/II, Geelong, Victoria, Australia.

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