



## RESISTANCE OF REINFORCED GEOPOLYMER CONCRETE BEAMS TO ACID AND CHLORIDE MIGRATION

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**Received:** 20 February 2012; **Accepted:** 25 June 2012

### ABSTRACT

The deterioration rate of plain geopolymer concrete specimens when exposed to aggressive environment was low and the specimens were stable when compared to plain ordinary Portland cement concrete. Attention was paid upon the durability and flexural behaviour of reinforced geopolymer concrete beams, manufactured using low calcium class F Indian fly ash, exposed to 10% concentration of sulfuric acid attack and chloride attack for a period of 180 days. 100mm×100mm cross-section and 500mm long beams with 1% tensile reinforcement were cast. Concentration of sodium hydroxide was taken as 8M for a cube compressive strength of 30N/mm<sup>2</sup>. Test results showed very little surface erosion, 3.26% and 1% weight loss, 10.64% and 4.47% decrease in ultimate moment for specimens exposed to chloride and acid attacks, respectively. This has revealed better performance of reinforced geopolymer concrete beams subjected to aggressive situation and is in line with earlier studies on plain geopolymer concretes. The erosion of surface of specimens had not led to corrosion of steel bars which underlines the geopolymer concrete as an impermeable one. X-ray diffraction (XRD), EDAX and scanning electron microscopy (SEM) analyses were done to compare results of reinforced geopolymer concrete beams, before and after immersion in aggressive liquids.

**Keywords:** Fly ash; molarity; geopolymer concrete; acid attack; chloride attack; flexural strength

### 1. INTRODUCTION

Construction of residential buildings, industries and infrastructures is gaining momentum in an upward trend worldwide. Construction industries strive hard to meet out this growing task. This ultimately leads to consumption of large volume of ordinary Portland cement

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(OPC) concrete. OPC concrete is the second largest product, next to water, being consumed in the world. The prime constituent of OPC concrete is cement. It is estimated that 2.2 **billion** tons of cement would be produced in the coming years throughout the world. This large volume of cement production emits greenhouse gas, into atmosphere. It is agreed that the emission of CO<sub>2</sub> by cement industries would be around 80% of quantity of cement produced. This huge quantity of CO<sub>2</sub> being emitted into atmosphere is increasing every year due to exponential growth in the production of cement. This emission of greenhouse gas is quite alarming and urgently requires an alternate to substitute cement in construction industry. Geopolymer and geopolymer concrete being versatile, **are** getting popular across the globe not only in research field but also in construction industry. Geopolymers are essentially amorphous[1] polymers and are synthetic minerals belonging to the family of aluminosilicates as zeolites. The silica and alumina abundantly present in the Class F flyash is activated by alkali hydroxides and alkali silicates assisted by thermal energy. This paper reports a study of durability and flexural strength of reinforced GP concrete beams manufactured using class F flyash activated by silicates and hydroxides of sodium, after immersion in 10% concentration of H<sub>2</sub>SO<sub>4</sub> solution and chloride solution.

Geopolymers are advanced mineral binders and as a consequence, Geopolymer concrete possesses physico-chemical properties which is entirely different from those of conventional OPC concrete. Yet, **an increase in number of research articles in various names is witnessed**, involving essentially alkali-activation. However, we, the civil engineers, feel alkali as danger because of the harmful alkali-aggregate-reaction. This creates a lot of confusion in people's mind, generating false granted ideas about this innovative cement-free construction material. In addition, misconception of Geopolymer concretes prevails as though geopolymer concretes have high alkalinity than OPC concrete. But in contrast to OPC concrete, Geopolymer concrete has a pH value ranging between 11.5 and 12.5.

Geopolymers are amorphous three dimensional aluminosilicate materials with ceramic-like properties that are produced and hardened **even** at ambient temperature. Under highly alkaline conditions, in the presence of alkali hydroxide and silicate solution, polymerization takes place when reactive aluminosilicates are rapidly dissolved and free SiO<sub>4</sub> and AlO<sub>4</sub> tetrahedral units are released in solution. The tetrahedral units are alternatively linked to polymeric precursors by sharing oxygen atoms thus forming amorphous geopolymers. Positive ions such as K<sup>+</sup> or Na<sup>+</sup> that are present in framework cavities, balance the negative charge [1]. For the chemical designation of geopolymers based on silico-aluminates, the term poly(sialate) that is an abbreviation for silicon-oxo-aluminate has been proposed. Poly(sialates) are chain and ring polymers with Si<sup>4+</sup> and Al<sup>3+</sup> in 4-fold coordination with oxygen. Chains and rings are formed and cross-linked together always through a sialate Si-O-Al bridge.

Even though a number of literatures **has** focused the main attributes of Geopolymer concrete such as strength and durability against aggressive environments like sulphate [2], acid [2-6], and fire, there is a handful of literatures available concerning the durability of reinforced geopolymer concrete in a seawater and sewage environments. Also the cost of this geopolymer Concrete is 10-30% cheaper than that of Portland Cement Concrete [7]. Therefore, the present research focuses on the durability of heat cured reinforced geopolymer concrete beams manufactured using low calcium class F Indian fly ash and the same is investigated with the support of SEM and XRD reports.

## 2. EXPERIMENTAL PROGRAM

### 2.1 Materials characterisation

#### 2.1.1 Source materials

Any material rich in silica and alumina in glassy powder form is apt for acting as a source material in the synthesis of Geopolymeric binder and consequently, Geopolymer concrete. In the recent past, several naturally available materials like Metakaolin clay, red soil or synthetic waste products from industries like ground granulated blast furnace slag and fly ash have been used as source material and investigations are done. Though Metakaolin showed very fast rate of dissolution in the alkaline solution, owing to its high cost, the abundantly available flyash was chosen for this work. Since high calcium Indian flyash contains excess quantity of calcium and sulfate in it, which may disturb the polymerization action, ASTM Class F low calcium fly ash has been preferred.

Table 1: Chemical composition of flyash sample

Type of chemicals	% By weight
Silica	63.53
Alumina	27.40
Iron Oxide	3.67
Calcium Oxide	1.26
Magnesium Oxide	0.35
Sodium Oxide	0.19
Sulphur Trioxide	0.01
Titanium Dioxide	1.84
Potassium Oxide	0.85

Indian flyashes are heterogeneous in nature, greyish white in colour, contain moisture less than 0.3% by weight, loss on ignition 0.9%, bulk density 1047 Kg/m<sup>3</sup>, specific gravity 2.16 and with some traces of Sulphur trioxide. An X-Ray Fluorescence (XRF) analysis done to determine the chemical composition of low calcium flyash used in this experimental program is shown in Table 1. Flyash is obtained from Tuticorin Thermal Power Station, Tamilnadu, India and contained 1.26% of CaO by weight and hence has been designated as Class F fly ash. From the composition of fly ash, it is obvious that Indian flyash contains more silica and the ratio of silica to alumina is approximately 3 whereas in Australian flyash, the ratio is approximately 2.

#### 2.1.2 Alkaline liquids

Sodium silicate liquid is purchased from local supplier. The ratio of Na<sub>2</sub>O to SiO<sub>2</sub> being 2.2 i.e Na<sub>2</sub>O being 15% and SiO<sub>2</sub>, 33%. Sodium hydroxide in flake form, soluble in water is obtained from local supplier. The weighed quantity of NaOH flakes are made to dissolve in

required quantity of water to make solution. Measured quantity of sodium silicate is then mixed with dissolved NaOH to get alkaline solution.

### 2.1.3 Fine and coarse aggregates

River sand clean from mud, silt etc is used as fine aggregate and 6 mm to 12mm size graded hard blue granite jelly obtained from local crusher is used as coarse aggregate. The fineness modulus and specific gravity of fine aggregate are 2.73 and 2.6 respectively as per IS 2388-1968 part 3 where as it is 7.12 and 2.75 for coarse aggregate. Aggregates used for ordinary Portland cement concrete is normally used to manufacture geopolymer concrete. Aggregates are made wet by sprinkling water on it which helps to avoid **absorption** of water from alkaline solution.

### 2.1.4 Super plasticizer

High range water reducing ligno-sulphonated based super plasticizer is added to improve the workability of geopolymer concrete to an extent which is, otherwise, very stiff.

### 2.1.5 Manufacture of reinforced geopolymer concrete beams

Weighed quantity of Sodium hydroxide (NaOH) in flake form to suit 8M is allowed to dissolve in potable water. Heat is generated when NaOH gets dissolved in water and the solution is stirred till it is cooled. This solution is mixed with sodium silicate solution to obtain alkaline solution. Fly ash and fine aggregate are first mixed in a 150kg capacity Pan mixer for about 2 minutes for uniform mixing. Alkaline solution is added to the dry mix of fly ash and fine aggregate and allowed to get mixed for 5 minutes. Surface wet coarse aggregate is added to the mixture and mixed for another 5 minutes[8]. Measured quantity of super plasticizer is added to the mixture for considerable workability. Oil is applied onto the surfaces of 100mm x 100mm in cross-section and 500mm long steel moulds for easy demoulding. 2 numbers, 8mm diameter rods for tensile reinforcement (1%), 2 numbers, 6mm dia hanger rods with 6mm dia two legged stirrups at 50mm centres are fabricated and placed inside steel moulds. The Longitudinal section and cross sectional details of Specimen are shown in Figure 1.

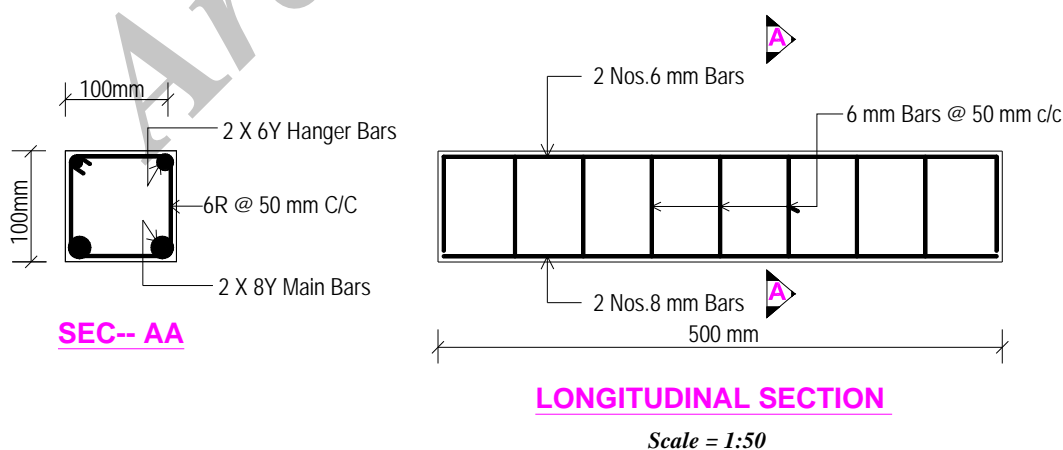


Figure 1. Sectional views of specimen (typical)

Prepared geopolymer concrete is poured into the moulds in layers and the concrete is vibrated in table vibrator for better compaction. The glassy appearance of fresh Geopolymer Concrete is shown in Figure 2.

Geopolymer concrete beams in steel moulds are placed inside heat curing chamber and cured at 70°C for 24 hours. The beams are allowed to get cooled in room temperature for one day and are demoulded. Totally 27 numbers of beams are manufactured of which 12 numbers each for sulfuric acid attack and chloride attack tests with 3 numbers being control specimens. The casting of specimen is shown in Figure 3.



Figure 2. Fresh geopolymer concrete ready to use



Figure 3. Specimen being vibrated

#### 2.1.6 Immersion in sulfuric acid and chloride solution

The concentrated sulfuric acid of 98% purity and density of 1.85 g/cc was utilised to prepare the sulfuric acid solution. 55 ml of concentrated  $H_2SO_4$  was mixed with 945 ml of potable water to reach one litre of acid solution. Sulfuric acid ( $H_2SO_4$ ) solution was prepared in plastic container to 10% concentration. For the study on chloride attack, solution was prepared by mixing 5% hydrochloric acid (HCl) and 5% sulfuric acid ( $H_2SO_4$ ) in plastic container. 12 numbers of specimens were immersed in their corresponding solution for 180 days. The volume of solution was kept in such a manner that not less than four times the volume of specimens immersed was maintained throughout this study. The solution was stirred on alternate days to avoid deposits of acid or chloride in the container. The solution was replaced with fresh one after 30 days. The specimens subjected to Chloride attack is designated as 'A' and acid attack as 'B'. The specimens put in the respective solution are shown in Figure 4.

The acidity of the solution was checked periodically with the aid of pH meter by titrating with standard alkaline solution. The effects of sulfuric acid on the geopolymer beams were regularly monitored through visual inspection, change in weight and flexure strength tests. The weight of beams before immersion is noted in a digital weighing balance and its saturated surface dry weight is considered as initial weight ( $w_1$ ). These samples were removed from the solution and weighed at specific intervals and marked as ( $w_2$ ). Samples for determining residual flexural strength were stored in room temperature for 3 days before subjected to flexure test. At the end of 26 weeks of test period, the specimens were taken out and samples were taken from the outer surface and cut into cubes of 10mm sides to study the alteration in microstructure due to acid attack and chloride attack.



Figure 4. Specimens immersed in the solutions

### 2.1.7 Testing of specimens

Two specimens were taken out after 7, 15, 30, 60, 120 and 180 days from respective solutions. All the specimens were allowed to dry in room temperature for 24 hours and then dried in heat curing chamber at 60°C for 2 hours. Visual appearance, weight loss and change in flexural strength were noted. The flexural strength test set up is shown in Figure 5. The change in microstructure due to permeability of aggressive solution was also studied by X-ray diffraction (XRD) and scanning electron microscopy (SEM), at the end of 180 days.



Figure 5. Typical flexural strength test set up before test



Figure 6. Typical flexural strength test set up after test

XRD analyses were done by using a diffractometer with the following details: PANalytical Make, X'per PRO Model, Cu K (2.2 KW Max.) source, X'celerator (Semiconductor) detector, Ni foil Beta Filter. The XRD patterns were obtained by scanning at  $2\theta$ . SEM (HITACHI S-3000H, Japan) was used for microstructural observations of the fracture surfaces, which were



coated with evaporated Copper for examination. SEM analyses were done at a maximum magnification of 300,000 x with a high resolution of 3.5nm.

### 3. RESULTS AND DISCUSSIONS

#### 3.1 Surface deterioration

Prolonged exposure of fly ash based geopolymer concrete beams in 5% HCl + 5% H<sub>2</sub>SO<sub>4</sub> solution (chloride attack) showed very little deterioration of top surface of specimens, less than 2mm, which had led to visibility of coarse aggregates.

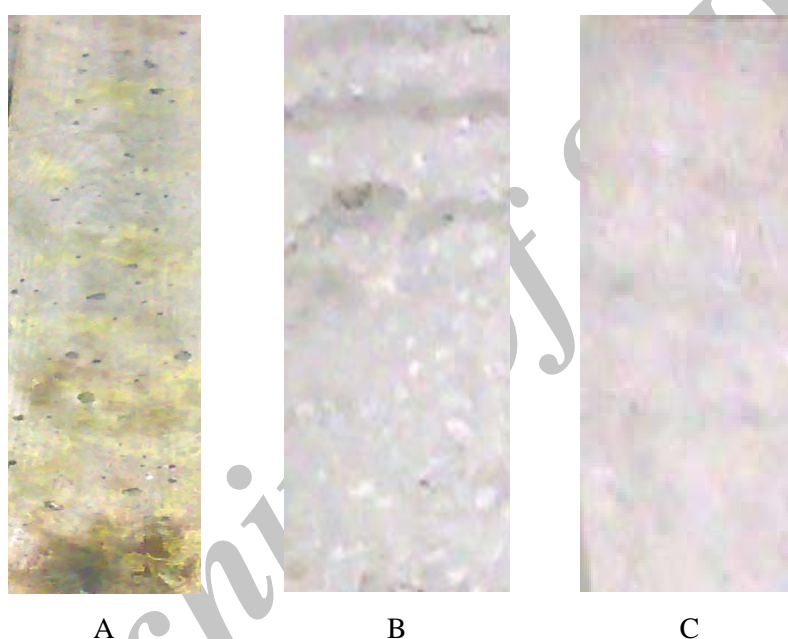


Figure 7. (A) Typical beam after 180 days of exposure to Sulphuric acid solution (B) Typical beam after 180 days of exposure to HCl+H<sub>2</sub>SO<sub>4</sub> solution (C) Typical unexposed specimen

Whereas beams immersed in 10% H<sub>2</sub>SO<sub>4</sub> solution, visible yellowish green hue patches were seen on the surfaces. This could be seen from the pictures given in Figure 7. The beam with designation 'C' represents specimen not subjected to aggressive exposure. Before exposure to acid solution, specimens possessed a fairly smooth surface and due to exposure, deterioration of the surfaces started which appeared to be very marginal, less than one mm. The specimens kept in Sulfuric acid solution showed good resistance to acid. Throughout the duration of exposure, specimens were taken from solutions periodically and checked for any noticeable changes on the surfaces. This phenomenon of acid resistance can very well be applied in manufacturing sewers, lining material in acid storing containers **such as** sewage disposing structures. There was no visible rusted surface seen in all the specimens. After 180 days of exposure to chloride, specimens 'B' showed no traces of corrosion, which is shown in Figure 8.



Figure 8. Top-uncorroded rod in specimen 'A' Bottom-uncorroded rod in specimen 'B'

### 3.2 Change in weight

The change in weight of geopolymer concrete beam specimens after exposure to acid and chloride **was** compared with the weight of control element 'C'. All the specimens except control elements recorded weight loss and it was observed to be 3.26% in specimens subjected to acid attack. Almost all the specimens retained their shape and texture and there was no noticeable weight loss in specimens subjected to chloride attack. The loss of weight in the case of specimens subjected to chloride attack was noted and found to be 1%. The results are illustrated in Figure 9. The rods were taken out from the Specimens at the end of 180 days of study and weighed. There was not even a fraction of gram weight loss noticed in both the beams.

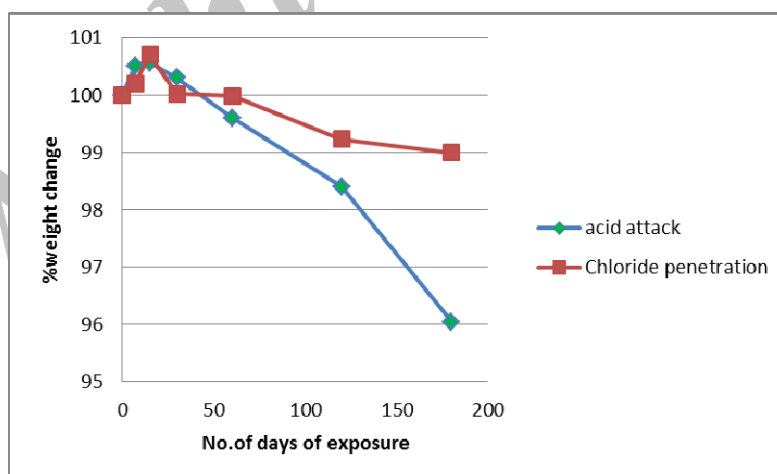


Figure 9. Change in weight (%) after exposure to acid and chloride

### 3.3 Scanning electron microscopy and EDAX

Samples for scanning electron microscopy (SEM) analysis and EDAX were taken from near the surface of specimen. SEM micrographs along with EDAX spectrum showing the images



of geopolymer concrete beams after 180 days of submergence in acid and chloride environment, which are designated as sample A Series and sample B series, illustrated in Figure 10 and Figure 11. And the unexposed specimens as sample C series is shown in Figure 12. In reference specimen C, un reacted fly ash particles could not be noticed rendering it a high denser microstructure of concrete. From EDAX spectrum of Sample C, it **could** be noted that iron oxide content was 3.7%. Also it revealed the presence of Si, Al, K, Na and C as the main elements. After 180 days of exposure to 10%  $H_2SO_4$  solution, specimens appeared to have deteriorated by the acid attack and at the same time EDAX spectrum also reported that a change in the presence of elemental traces. The iron oxide content had increased from 3.7% to 6.02% for Specimen A and the content of iron oxide for Sample B had decreased from 3.7% to 3.11%. This shows that the metal was not corroded under acid attack. From SEM images of samples A and sample B, the presence of light precipitates which might be a product of degradation was seen.

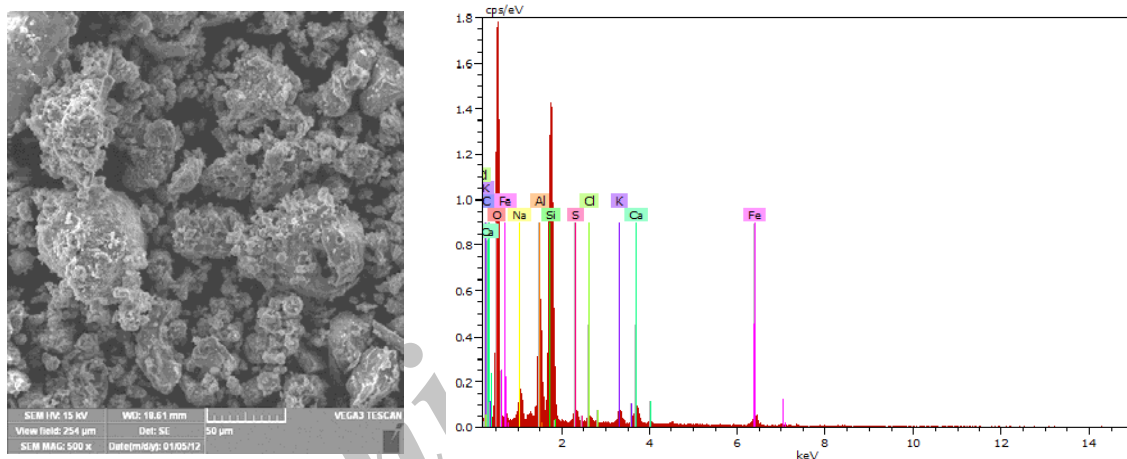


Figure 10. SEM image of specimen A and its corresponding EDAX spectrum after 180 days of 10%  $H_2SO_4$  exposure

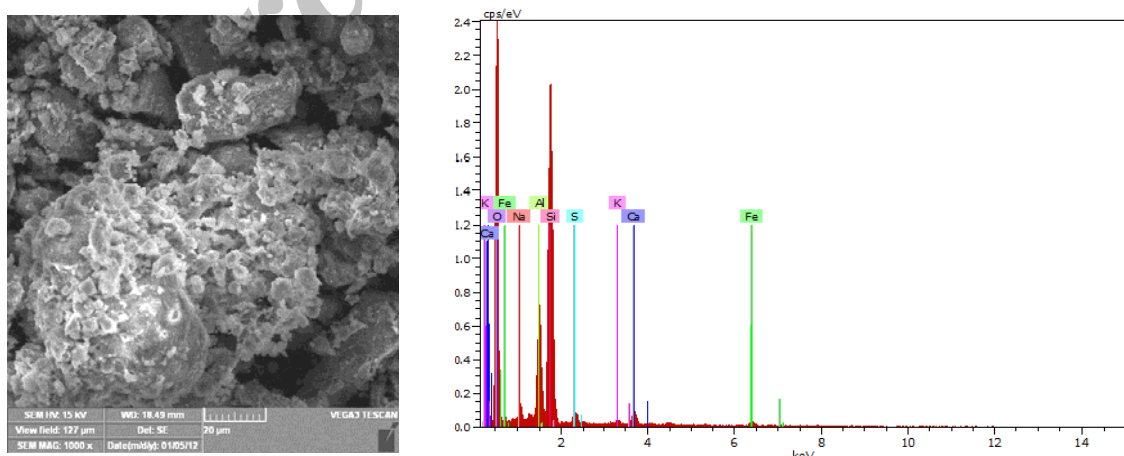


Figure 11. SEM image of specimen B and its corresponding EDAX spectrum after 180 days of 5%  $H_2SO_4$  + 5% HCl exposure

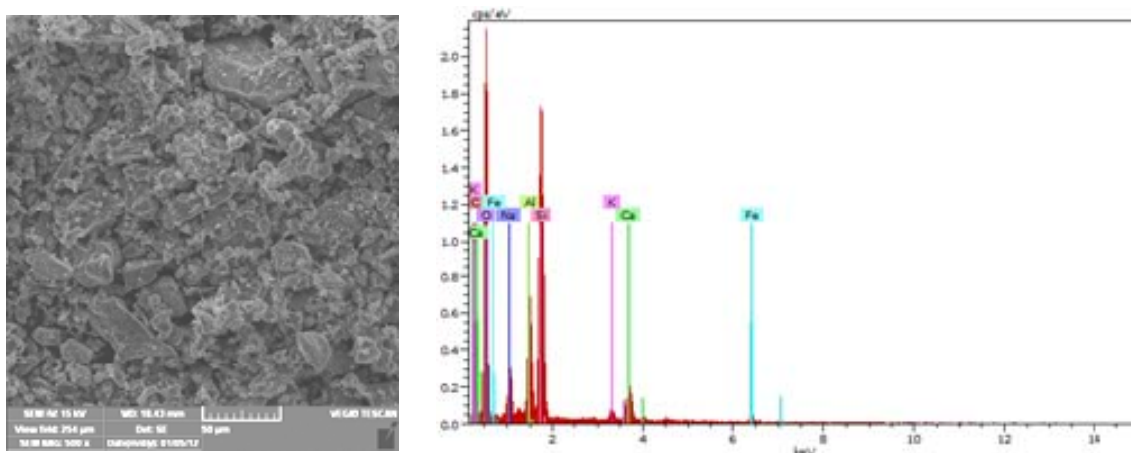


Figure 12. SEM image of specimen C and its corresponding EDAX spectrum before exposure

### 3.4 X-Ray diffraction analysis

XRD patterns of the sample 'A', sample 'B' and sample 'C' are illustrated in Figure 13, Figure 14 and Figure 15 **respectively**. From this spectrum of specimen A, we could infer that peaks for Zeolitic phases have reduced and new mullite arising from aluminosilicate gel formed between  $20$  and  $21^\circ 2\theta$  which indicates eruption by sulphur. Also new hematite crystals appeared at  $78^\circ 2\theta$  might have reduced the peak intensity seen in the  $26-29^\circ 2\theta$ .

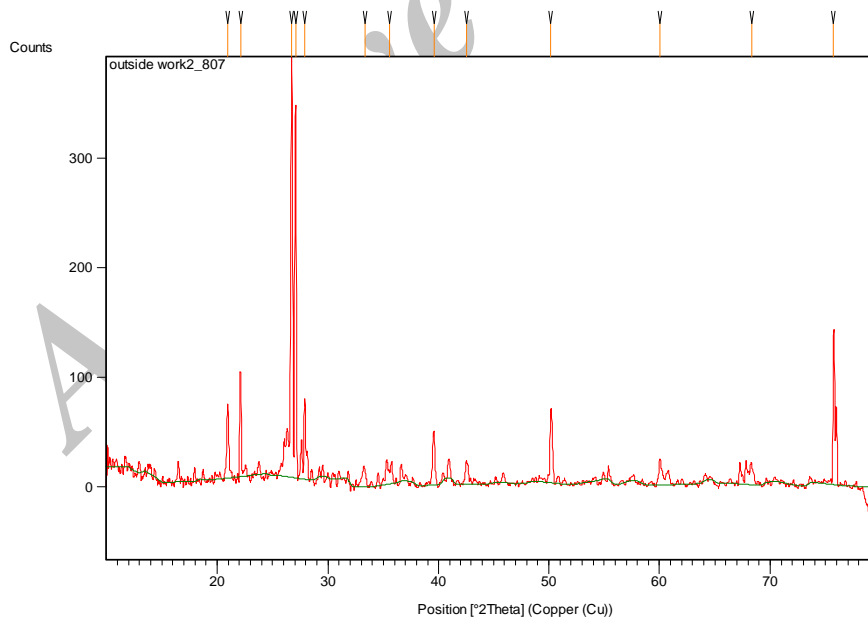


Figure 13. XRD pattern of specimen 'A'

After 180 days of exposure to Chloride solution, the peak intensity of Zeolitic phases have reduced a little, without any noticeable change in the microstructural profile. This shows the resistance to chloride. This characteristic of low calcium based geopolymer

concrete may very well be adopted in structures in coastal areas, off-shore structures, tetrapods provided on the sea shores to withstand tidal waves and erosion due to chloride attack. Interaction of geopolymers with the sulfuric acid solution **have caused** replacement of the exchangeable cations (Na) in polymers by hydrogen or hydronium ions. The observation of the mass changes of the samples exposed to acidic solutions and results obtained from SEM, EDAX and XRD analyses gave a positive sign of hypothesis test of depolymerisation of aluminosilicate polymer gel.

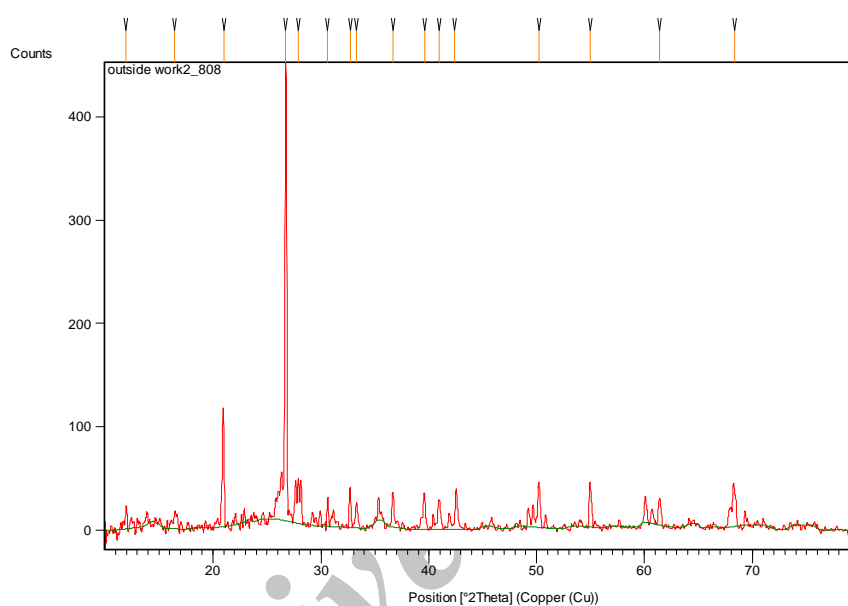


Figure 14. XRD pattern of specimen 'B'

### 3.5 Residual flexural strength

The main objective of this paper is achieved by evaluating the residual flexural strengths of Geopolymer Concrete beams after acid and chloride attack and been compared with those of Geopolymer Concrete beams unexposed to chemicals. All the beams cast **are** tested in an Universal Testing Machine of 1000kN capacity. A deflectometer is positioned at the bottom middle of each beam, to find out the deflection underwent. A monotonically increasing single point load is applied until failure, at the middle of beam by a load cell of 500kN capacity. The crack propagation, initial crack load, ultimate load and deflection are noted for further investigation. The load–deflection curves are plotted to indicate distinct events that are happening during the test. All beams behaved in a similar manner, as they are designed as under reinforced section; steel to reach its yield strength before failure. The test setup with (typical) tested beam is shown in Figure 6. As expected, flexure cracks got initiated at the bottom of the beam in the tension zone. As load is incremented, new cracks initiated in a similar way. Invariably, all the beams failed in compression mode. It is well established from the experimental studies that the load carrying capacities of Specimen 'A' and specimen 'B' are not reduced appreciably when compared to that of Specimen 'C'. The Table 2 depicts the results of this test.

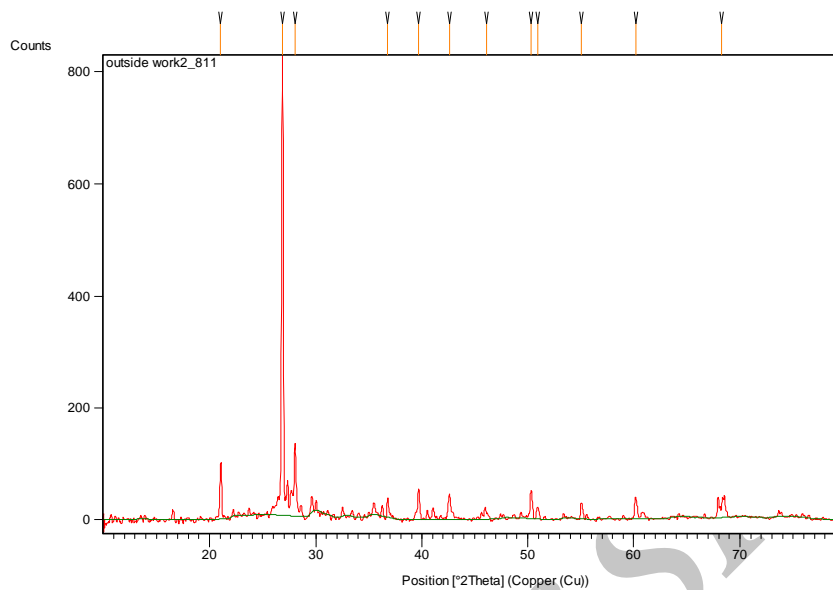


Figure 15. XRD pattern of specimen 'C'

Table 2: Flexure test results of beams

Designation of beam	No. of days of exposure	Load in kN		Mid-span deflection in mm	Cracking Moment in kNm		Ultimate Moment in kNm[9]		Failure mode
		First crack ( $P_{cr}$ )	Ultimate load ( $P_u$ )		Experimental ( $M_{cr,exp}$ )	Calculated ( $M_{cr,cal}$ )	Experimental ( $Mu_{exp}$ )	Calculated ( $Mu_{cal}$ )	
Specimen C	0	21.00	47.00	23	2.10	0.85	4.70	2.77	Compression
Specimen A	7	20.78	44.98	21	2.08	0.85	4.49	2.77	Compression
	15	19.47	42.76	21.5	1.95	0.85	4.28	2.77	Compression
	30	19.18	42.50	19	1.92	0.85	4.25	2.77	Compression
	60	19.15	42.18	22.2	1.92	0.85	4.22	2.77	Compression
	120	18.75	42.08	22.3	1.88	0.85	4.21	2.77	Compression
	180	18.35	42.00	22	1.84	0.85	4.20	2.77	Compression
Specimen B	7	20.96	45.78	23	2.10	0.85	4.58	2.77	Compression
	15	19.98	45.32	23	2.00	0.85	4.53	2.77	Compression
	30	19.25	45.25	22.65	1.93	0.85	4.53	2.77	Compression
	60	19.15	45.05	22.65	1.92	0.85	4.51	2.77	Compression
	120	19.12	44.98	22.55	1.91	0.85	4.49	2.77	Compression
	180	19.05	44.98	22.5	1.91	0.85	4.49	2.77	Compression

#### 4. CONCLUSIONS

On the basis of results obtained from the extensive experimental investigations, following conclusions were drawn:

1. Flyash based reinforced geopolymer concrete beams revealed no visible signs of surface deterioration, formation of pores on the surface and spalling of concrete after immersion in aggressive solution for 180 days. The configuration of the beams was intact. Slight yellowish green patches were seen on the surfaces of beams after 180 days of immersion in 10% H<sub>2</sub>SO<sub>4</sub> solution. Surface erosion on top of the specimens, which is less than 2mm, was observed in specimen B.
2. Though loss in weight was observed in all specimens at the end of study period, all the specimens showed an increase in weight upto 60 days of immersion period. There was a sudden loss in weight after 2 months of immersion.
3. The mass reduction at the end of 180 days noted in specimen A was 3.26% and in specimen B, 1%.
4. SEM micrographs showed change in the microstructure of binder phase after exposure to aggressive solutions, which appeared to be milder.
5. There was no trace of visible corroded particles present on the rods taken from the tested specimens. There was no loss of weight of reinforcement rods after the exposure period. This might be due to the impermeability of concrete. The depth of penetration of acids and chlorides into concrete was less than 3mm and 4mm respectively, much lesser than the cover to steel (10mm).
6. All the beams showed pure flexure cracks, invariable of type and duration of exposure. The moment resisting capacity of specimen A was reduced to 7.9 % and of specimen B, it was only 1.96 %.
7. It was evident from the microstructural analyses that no CSH gel was formed in concrete, even though flyash had a considerable amount of Calcium in them.
8. From EDAX reports, the anticorrosive nature of Geopolymer Concrete could be witnessed by noticing the constant peak of Fe ion in all the specimens.
9. The attribute of resistance to chloride salts shall be well applied in the construction of structural members even in the marine splash zone.

**Acknowledgement:** The financial support for this project received from All India Council for Technical Education (AICTE) is gratefully acknowledged. The authors of this paper express their gratitude to the Management of Thiagarajar College of Engineering. They would like to thank Prof. Vijaya Rangan, Emeritus Professor of Curtin University, Australia for his valuable guidance in presenting this paper.

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