



## RESISTANCE OF FLY ASH BASED GEOPOLYMER MORTARS IN SULFURIC ACID

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### ABSTRACT

The present paper reports the experimental results of a study conducted to assess the resistance of fly ash based Geopolymer mortar specimens in sulfuric acid. The program consisted immersion of geopolymer mortar samples having percentage Na<sub>2</sub>O ranging from 5% to 8% of fly ash in a 10% Sulfuric acid solution up to a period of 18 weeks and evaluation of its resistance in terms of visual appearance, residual alkalinity, changes in weight and compressive strength at regular intervals. Visual inspection of Geopolymer mortar samples did not reveal any recognizable change in colour and remained structurally intact though the exposed surface turned slightly softer. Through Optical microscope, corroded surface could be seen which increased with time of exposure. After exposure in the acid solution for 18 weeks, the Geopolymer samples almost lost its alkalinity and showed very low weight loss in the range from 0.41% to 1.23% of initial weight. Loss of weight was found higher for specimen with higher percent of Na<sub>2</sub>O. Compressive strength loss at the end of test was 52% for specimen with 5% Na<sub>2</sub>O and 28% for specimens with 8% Na<sub>2</sub>O. Results obtained in the present study indicate that Geopolymers are highly resistant to sulfuric acid.

**Keywords:** geopolymer, resistance, durability, residual alkalinity, compressive strength, weight loss.

### 1. INTRODUCTION

Though ordinary Portland cement (OPC) is the most commonly used binder in infrastructure construction, its resistance to chemical attacks such as acids and sulfates is of concern. Acid attack has not traditionally attracted much attention, even when cement composites are severely damaged by acids wherein calcium hydroxide is dissolved and the hydrated silicate and aluminium phases are decomposed. In the past few decades, geopolymer binders have emerged as one of the possible alternative to OPC binders due to their reported high early strength and resistance against acid and sulfate attack [9] apart from its environmental friendliness.

Fly ash based geopolymers are one branch in the geopolymer family and these have attracted more attention since the 1990s. As a novel binder, the performance of fly ash based geopolymers is promising; especially in some aggressive situations where Portland cement concretes are vulnerable [5]. Geopolymer binders might be a promising alternative in the development of acid resistant concrete. Since Geopolymers are a novel binder that relies on alumina-silicate rather than calcium silicate hydrate bonds for structural integrity, they have been reported as being acid resistant. Davidovits *et al.* [1] found that metakaoline based geopolymer has very low mass loss when samples were immersed in 5% sulphuric acid solutions for 4 weeks. Bakharev T. [2] studied the resistance of geopolymer materials prepared from fly ash against 5% sulfuric acid up to 5 months exposure and concluded that geopolymer materials have better resistance than ordinary cement counterparts. X.J. Song *et al.* [5] conducted an accelerated test to assess the durability of geopolymer concrete in a 10% sulfuric acid solution for 56 days and reported its good durability. S.E. Wallah and B.V. Rangan [6] have shown that geopolymer

composites possesses excellent durability properties in a study conducted to evaluate the long term properties of fly ash based geopolymers. Allahverdi Ali and Skavara [3],[4] conducted tests to study the mechanism of corrosion of geopolymer cements in high and low concentrations of sulfuric acid. The absence of standard methods to evaluate the performance of cements in acid environments has led to research in different exposure conditions and procedures by various authors making it difficult to correlate the results.

The present study is aimed at evaluating the response of different Fly ash based geopolymer mortars to sulfuric acid using physico-mechanical indicators as degree of deterioration in an accelerated test conditions. The study comprised determination of changes in weight, alkalinity, compressive strength and visual appearance as a measure of resistance against sulfuric acid. The findings of the present study shall be useful in determining the applicability of geopolymer materials for use in acid environments.

### 2. MATERIALS AND METHODS

#### 2.1 Materials

Fly ash was obtained from Kolaghat Thermal Power Plant near Kolkata and had mineral and chemical composition as shown in Table-1 and Figure-1. 75% of particles were smaller than 45 micron and Blaine specific surface was 380 m<sup>2</sup>/kg. Fine sand used was river sand having specific gravity of 2.5 and fineness modulus of 2.65. Laboratory grade Sodium hydroxide in pellet form (98 percent purity) and Sodium Silicate solution (Na<sub>2</sub>O = 8%, SiO<sub>2</sub> = 26.5% and 65.5% water) with silicate modulus ~ 3.3 and a bulk density of 1410 kg/m<sup>3</sup> was supplied by LOBA CHEMIE Ltd. A mixture of Sodium hydroxide and

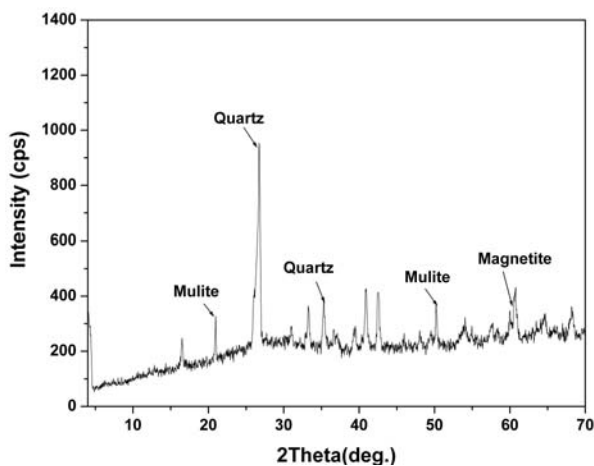


Sodium silicate solution giving Na<sub>2</sub>O in the mix as 5% to 8% of binder was used to activate the fly ash.

**Table-1.** Composition of Fly Ash (mass %).

Chemical composition	Percentage mass
SiO <sub>2</sub>	56.01%
Al <sub>2</sub> O <sub>3</sub>	29.8%
Fe <sub>2</sub> O <sub>3</sub>	3.58%
TiO <sub>2</sub>	1.75%
CaO	2.36%
MgO	0.30%
K <sub>2</sub> O	0.73%
Na <sub>2</sub> O	0.61%
SO <sub>3</sub>	Nil
P <sub>2</sub> O <sub>5</sub>	0.44%
LOI*	0.40%

\* Loss on ignition



**Figure-1.** XRD Diffractogram of fly ash.

## 2.2 Specimen preparation and test procedure

Activator solution (a combination of sodium hydroxide and sodium silicate solution) having % Na<sub>2</sub>O, 5% - 8% was used to activate the fly ash with a water to binder ratio 0.33. Mortar specimens of 50mm cubes were prepared keeping the sand to fly ash ratio as 1. Fly ash was first mixed with activator solution for 5 minutes in a Hobart mixer after which sand was slowly introduced and further mixed for another 5 minutes before being vibrated to remove any entrapped air. The samples were cured in an oven at 85°C for a period of 48 hours and specimens allowed to cool in the oven [7]. The specimens were then left at room temperature until tested.

To study the effects of exposure to acidic environment, specimens were immersed in 10 % solution of Sulfuric acid after 28 days for a period of 18 weeks, tests being carried out at regular intervals. The volume of acid solution was kept as four times the volume of specimens immersed and stirred every week. The solution was refreshed after 12 weeks. The effects of acid on the specimen were constantly monitored through visual inspection, weight change measurements and strength tests during exposure to the acid solution.

An optical microscope was used to study the surface deterioration of the specimen with time during exposure. Specimens were cut into halves using a low speed saw and a 1% Phenolphthalein solution was sprayed on the cut surface to study the residual alkalinity. Weights were measured using a digital balance in saturated surface dry conditions. Samples for weight change measurements were initially primed in water for 3 days and its saturated surface dry weight was taken as initial weight. Percent weight gain or loss has been determined based on ASTM C267. A digital compression testing machine was employed to determine the compressive strength of the specimen at regular intervals. The details of mix proportions used in the present study are given in Table-2. The 28 day compressive strength was found to be 22 MPa, 37 MPa and 40 MPa for the specimens GM1, GM2 and GM3, respectively.

**Table-2.** Mix proportion of geopolymer mortars.

Sample ID	Activator	Na <sub>2</sub> O (%)	Water/Fly ash	Curing temperature and duration
GM1	Sodium hydroxide + Sodium silicate	5%	0.33	85°C & 48 hrs
GM2		6.5%	0.33	85°C & 48 hrs
GM3		8%	0.33	85°C & 48 hrs

## 3. RESULTS AND DISCUSSIONS

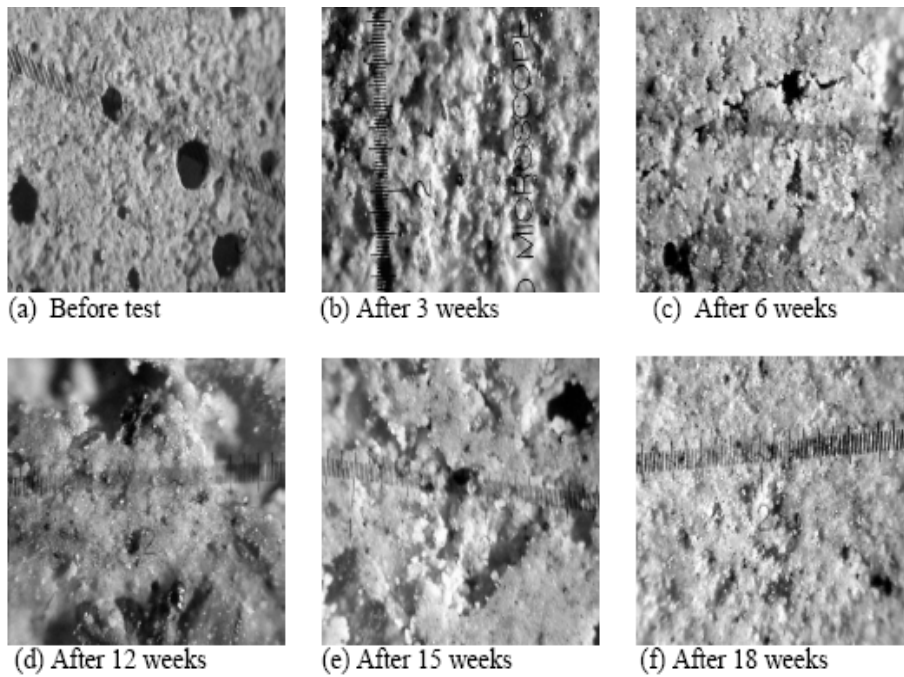
### 3.1 Visual appearance

Specimens did not exhibit any noticeable colour change and showed no visible signs of deterioration. The specimens were seen to remain structurally intact. The surface became a little softer as the duration of the test

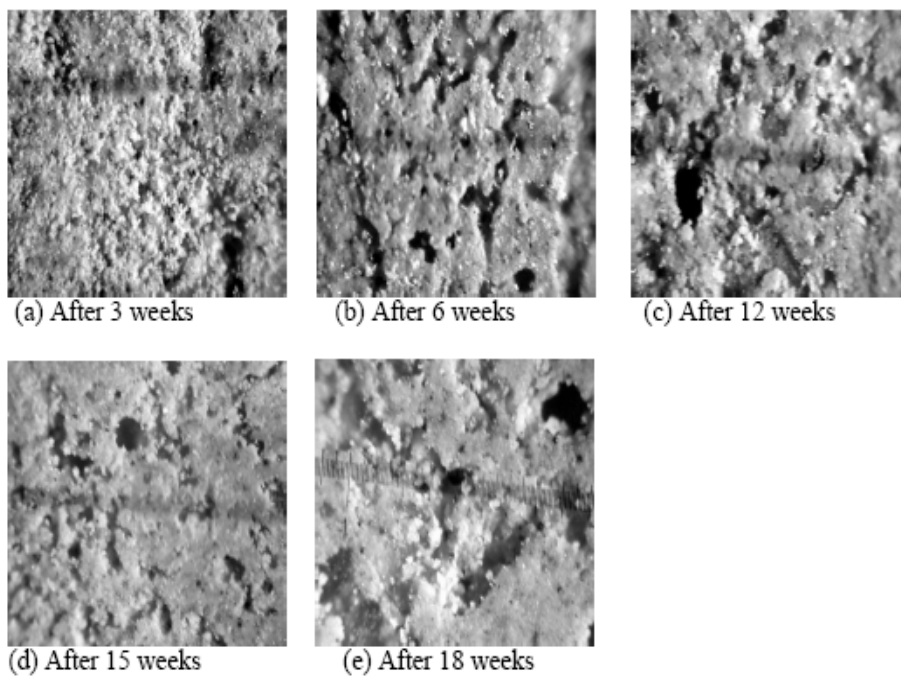
progressed but could not be easily scratched with finger nails. When seen under an optical microscope, deteriorated corroded surface could be observed. Figures 2, 3 and 4 show the corroded surfaces of specimens as seen under the microscope. The deterioration of the surface increased with time though magnitude of



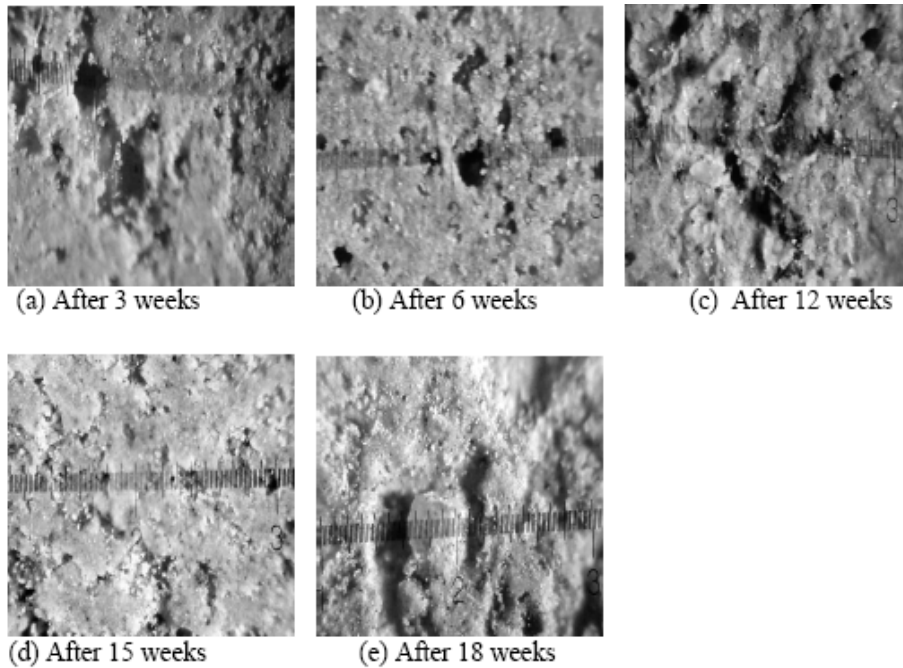
deterioration among the three series of samples could not be differentiated through visual inspection.



**Figure-2.** GM1 specimens in 10% sulfuric acid solution.



**Figure-3.** GM2 specimens in 10% sulfuric acid solution.

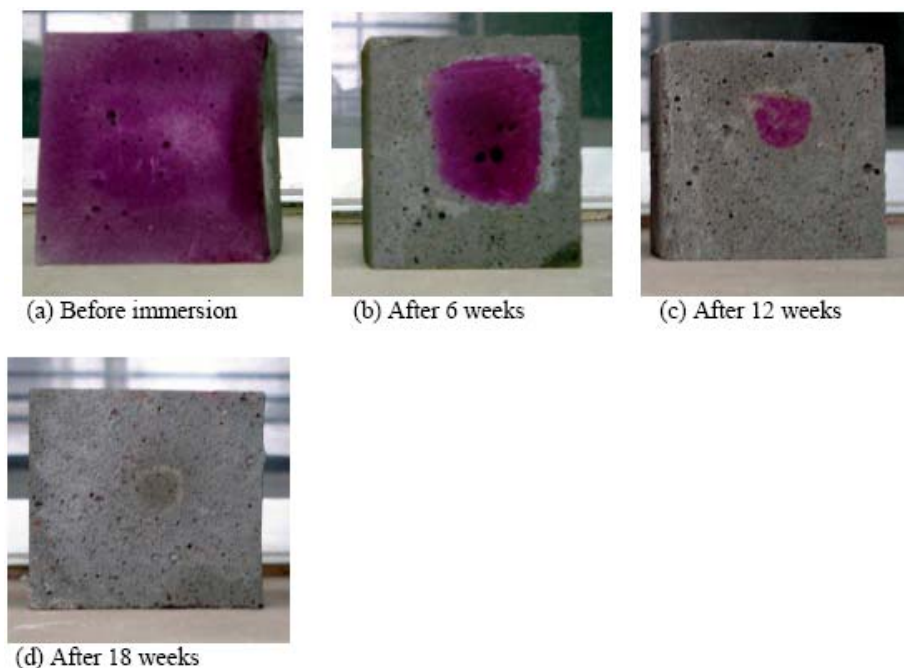


**Figure-4.** GM3 specimens in 10% sulfuric acid solution.

### 3.2 Residual alkalinity

The residual alkalinity of the geopolymer mortar specimens were examined roughly by spraying a 1% Phenolphthalein solution on the freshly cut surface [8]. On spraying, dealkalized part of specimen showed colourless while remaining part exhibited a magenta colour indicating its residual alkalinity as in Figure-5.

It was noticed that the process of dealkalization progressed inwards with time. Alkalinity were seen to have lost almost in 12 weeks, 15 weeks and 18 weeks for the specimens GM1, GM2 and GM3, respectively indicating the faster rate of dealkalization in specimen with lower content of  $\text{Na}_2\text{O}$ .



**Figure-5.** Residual alkalinity of GM3 samples in 10% sulfuric acid solution.

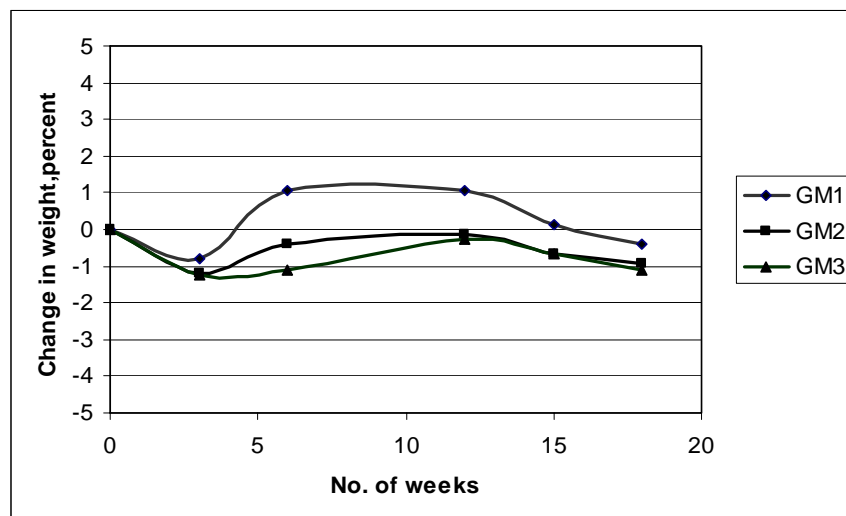


### 3.3 Change in weight

Results of the weight changes for the geopolymer mortars are presented in Table-3 and Figure-6. In all the specimens, a sudden loss of weight was noticed initially before gaining weight during 3 to 12 weeks. Beyond 12 weeks the weight dropped in the specimens. GM3 specimens with highest percent of alkali (8% Na<sub>2</sub>O) had the maximum loss of 1.23% and GM1 specimens (5% Na<sub>2</sub>O) exhibited only 0.41% loss after 18 weeks. As the Na<sub>2</sub>O content increased in the samples, weight loss also increased correspondingly.

**Table-3.** Weight changes of geopolymer mortar specimen exposed to 10% sulfuric acid after 18 weeks.

Sample ID	Weight changes (%)
GM1	-0.41
GM2	-0.92
GM3	-1.23

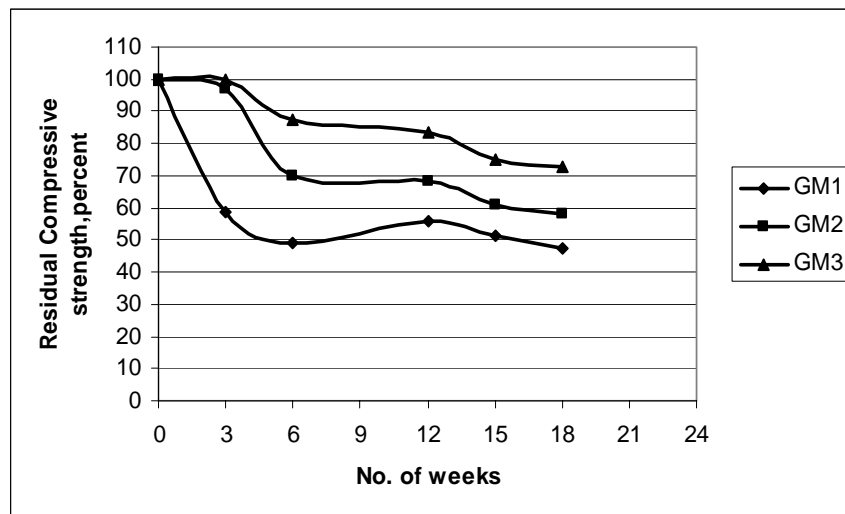


**Figure-6.** Weight changes in geopolymer mortars in 10% sulfuric acid solution.

### 3.4 Change in compressive strength

Figure-7 shows the compressive strength evolution of geopolymer mortars in the sulfuric acid environment. At regular intervals, the compressive strength was determined using a digital compression testing machine and the residual compressive strength was calculated as percentage of initial compressive strength. The three series of specimens were observed to lose weight and followed a similar trend. Geopolymer mortar specimens of higher Na<sub>2</sub>O content (6.5% and 8%) showed very little loss in strength initially. On the contrary,

specimens with lowest percentage of Na<sub>2</sub>O content (5%) exhibited large initial strength loss. Maximum loss of strength was observed in specimens of GM1 (5% Na<sub>2</sub>O) and minimum loss of strength in GM3 (8% Na<sub>2</sub>O). After exposure in 10% sulfuric acid solution for 18 weeks, strength loss was found to be 52%, 42% and 28% for specimens of GM1, GM2 and GM3, respectively. Thus geopolymer mortar specimens were observed to possess substantial residual compressive strength even when they are almost fully dealcalized at the end of 18 weeks exposure in 10% sulfuric acid solution.



**Figure-7.** Residual compressive strength of geopolymer mortars in 10% sulfuric acid solution.

#### 4. CONCLUSIONS

Based on findings during the present study, following conclusions can be drawn:

- Geopolymer mortar specimens manufactured from fly ash with alkaline activators were structurally intact and did not show any recognizable change in colour after 18 weeks exposure in 10% sulfuric acid solution;
- Exposed surfaces revealed a corroded structure when observed through an optical microscope and it progressed with time of exposure;
- Geopolymer samples almost lost its alkalinity within 15 weeks in 10% sulfuric acid solution;
- The weight loss results obtained in the study showed better performance than OPC counterparts which were reported to gain weight with heavily corroded structure. Specimens with higher alkali content were observed to lose more weight than specimens with lower alkali content; and
- Though specimens were fully dealcalized by sulfuric acid at the end of 18 weeks, it still had substantial residual compressive strength confirming its high resistance against sulfuric acid as claimed by few authors in earlier studies.

#### REFERENCES

- [1] Davidovits J. 1994. Properties of geopolymer cement Proceedings of the First International conference on Alkaline Cements and Concretes. Vol. 1, pp. 131-149. SRIBM, Kiev, Ukraine.
- [2] Bakharev T. 2005. Resistance of geopolymer materials to Acid attack. *Cem. Concr. Res.* 35: 658-670.
- [3] Allahverdi Ali, Skavara Frantisek 2001. sulfuric acid attack on hardened paste of geopolymer cements. Part 1. Mechanism of corrosion at relatively high Concentrations *Ceramics-Silikaty.* 45(3): 81-88.
- [4] Allahverdi Ali, Skavara Frantisek. 2006. sulfuric acid attack on hardened paste of geopolymer cements. Part 2. Corrosion mechanism at mild and relatively low concentrations, *Ceramics-Silikaty.* 50(1): 1-4.
- [5] Song X.J, Marosszeky M, Brungs M, Munn R. 2005. Durability of fly ash based geopolymer concrete against sulphuric acid attack. 10 DBMC International Conference on Durability of Building Materials and Components. Lyon, France. pp. 17-20.
- [6] Wallah S.E, Rangan B.V. 2006. Low Calcium Fly ash based Geopolymer Concrete: Long term properties. Research Report GC 2, Curtin University of Technology, Australia.
- [7] Thakur Ravindra N., Ghosh Somnath. 2007. Fly ash based Geopolymer composites. Proceedings of 10<sup>th</sup> NCB International seminar on cement and building materials, NEW Delhi, India. Vol. 3. pp. 442-451.
- [8] Rendell F., Jauberthie, R. 1999. The deterioration of mortar in sulphate environments. *Construction and building materials.* 13(6): 321-327.
- [9] Palomo, A. Grutzeck, M.W, Blanco, M.T. 1999. Alkali activated fly ashes cement for the future. *Cem. Concr. Res.* 29: 1323-1329.