

# Strength And Durability Of Fly Ash Geopolymer Blended With Lime Stone Dust

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## Abstract

*This paper presents results of an experimental study performed to investigate effect of incorporating Lime stone dust on physico-mechanical properties and durability of resulting fly ash geopolymer Paste sample. In this program geopolymer samples having percentage Lime stone dust ranging from 10 to 15% of fly ash were immersed in a 10% magnesium sulfate solution upto a period of 15 weeks and evaluation of its resistance in terms visual appearance, change in weight and compressive strength at regular interval. Addition of Lime stone dust significantly improved durability and physico-mechanical properties.*

## 1. Introduction

Geopolymeric binders [1,2,3] are latest types of inorganic binding means whose regular aspect is alkaline activation of clinker or substances with latent hydraulic properties, such as slag or fly ash. Geopolymer is promising especially in some aggressive situations where Portland cement concretes are vulnerable [4]. The presence of zeolite-type substances is responsible for modifying the properties of the alkali-activated binders, for instance by increasing their resistance to acids [5] or improving their ability to immobilise heavy metals [6, 7]. Its resistance to chemical attacks such as sulfate is of concern. In the recent years, interest in geopolymer is increasing manifold due to their reported advantages over ordinary Portland cements. Geopolymer materials are accounted to exhibit high early strength, better durability and have just about no alkali-aggregate reaction [8]. These materials are therefore anticipated to be cement for the future [9]. In recent years, fly ash based geopolymers have received tremendous attention for its various reported superior properties and also for its abundant availability as wastes from thermal power plants. Low calcium fly ash based geopolymer prepared with various activators have exposed high compressive strengths and outstanding performance when subjected

to different acid and sulphate solutions [10-16]. Again geopolymer has also been stated to be highly resistant to elevated temperatures [17-19]. Geopolymer activated by a mixture of sodium hydroxide and sodium silicate solution capitulate higher compressive strength [10, 16, 17]. Moreover, the microstructure progress depends on alkali content of activating solution. Addition of moderate amount of minerals to a geopolymer can have considerable enhancement on the geopolymer structure and properties. Temuujin et al. [20] recommended that the addition of calcium compounds CaO and Ca(OH)<sub>2</sub> improve mechanical properties of the fly ash-based geopolymers cured at ambient temperature again.

Lime stone dust was used as a source material of CaCO<sub>3</sub> a strong inter-particle bond may be developed due to the pozzolanic reaction of lime present in Lime stone dust with the amorphous silica and Alumina [21]. Favourable effect in conventional cement concrete by adding calcium has already been studied [22].

The objective of the experimental investigation was to study the effect of addition of Lime stone dust as a supplementary material on physico-mechanical properties and durability of fly ash based geopolymer Pastes. Up to 15% of Lime stone dust by weight was added in increments to the fly ash while manufacturing geopolymer paste. Durability of geopolymer materials was assessed by regular monitoring of its physical appearance, weight changes and compressive strength changes on exposure to 10% magnesium sulphate solution. In addition, effects of apparent porosity and water sorptivity on weight and strength changes are studied.

## 2. Experimental

### 2.1 Materials

The lime stone dust is a solid composite having specific gravity 2.6 ,bulk density 1545 kg/m<sup>3</sup> ,consisting of 95.8% CaCO<sub>3</sub> .It has a average particle size of 12 micron while particle size varies between a range of 2 $\mu$  to 25 $\mu$ . The chemical composition has been provided in table 2. Low calcium Class F fly ash

used in the present research work was collected from Kolaghat Thermal Power Plant near Kolkata, India. It had chemical composition as given in Table-1. About 85% of particles were finer than 45 micron and Blaine's specific surface was 380m<sup>2</sup>/kg. 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,India. The alkaline activating solution was prepared by dissolving required quantity of sodium hydroxide pellets directly into predetermined quantity of sodium silicate solution. It had Na<sub>2</sub>O content and SiO<sub>2</sub> content as 8.0% of fly ash, thereby making SiO<sub>2</sub>/Na<sub>2</sub>O ratio of 1. Water to Fly ash ratio was of 0.33. The activator solution was left at room temperature overnight before being used to manufacture geopolymer specimens. In Fig 1 and Fig 2 the particle size of Fly Ash and Lime Stone Dust have been examined through Scanning Electron Microscopy respectively. Figure 3 and Figure 4 reveal the existence of typical elements in Fly Ash and Lime Stone Dust respectively.

Table 1. Chemical composition of Fly ash

Chemical composition	Fly ash
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
Loss on ignition	0.40

Table 2. Chemical composition of Lime stone dust

Chemical Composition	Percentage
CaO	51.01
MgO	0.28
Fe <sub>2</sub> O <sub>3</sub>	0.36
Al <sub>2</sub> O <sub>3</sub>	2.74
SiO <sub>2</sub>	3.92
K <sub>2</sub> O	0.04
Na <sub>2</sub> O	Nil

TiO <sub>2</sub>	0.09
Loss on ignition	41.56

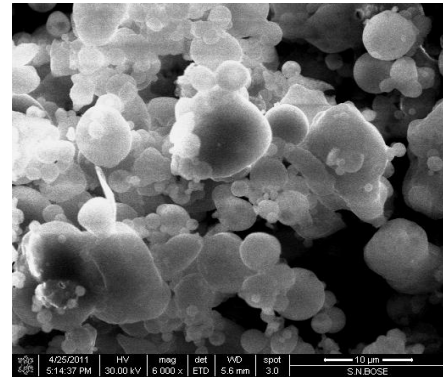


Fig 1: SEM Image of Fly Ash

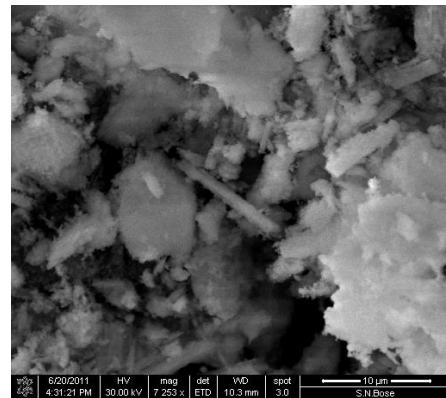


Fig 2: SEM Image of Lime stone dust

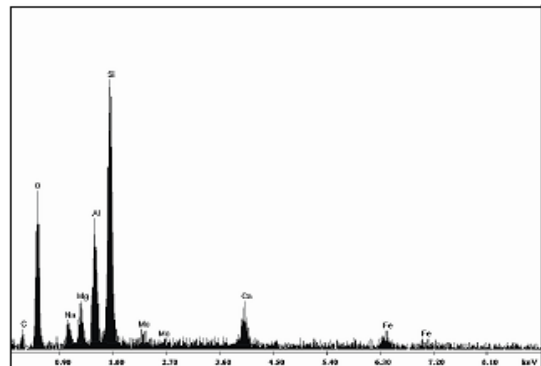


Fig 3: EDAX SPECTRA OF FLY ASH

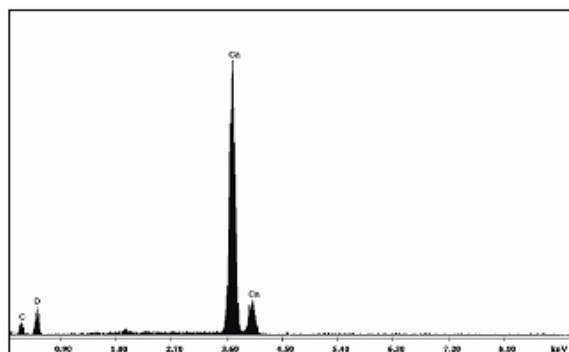


Fig 4: EDAX SPECTRA OF LIME STONE DUST

## 2.2 Preparation of specimens and testing

Sodium hydroxide pellets was mixed into water as required to maintain water to fly ash ratio in the activator solution as 0.33 . The Sodium hydroxide solution thus prepared was kept for one day before mixing with sodium silicate solution . The mixture of Sodium hydroxide solution and sodium silicate solution again was kept for few hours until its temperature came

down . The curing regime adopted was after Thakur and Ghosh [11] . At the very out set Lime stone dust (95.8%  $\text{CaCO}_3$ ) was mixed with fly ash as 10% , 15% by weight of fly ash . This Lime stone dust added fly ash was mixed with the activator solution for 5 minutes in a Hobart mixture for preparing Paste .

Paste were then transferred into 50mm x 50mm x 50mm cubical moulds and vibrated on a vibrating table for 2 minutes. Specimens were cured along with the moulds in an oven for a period of 48 hours at 650C and allowed to cool inside the oven before being removed to room temperature.

Specimens were removed and stored at room temperature at a dry place before testing. Some data of the present study are given in the Table 3.

The geopolymer specimens were immersed in 10% magnesium sulphate solution for 15 weeks after 3 days from casting. The specimens were tested for physical changes, weight changes and compressive strength changes at regular intervals.

Table 3. Details of Geopolymer Paste and Paste specimens

Sample ID	Na <sub>2</sub> O content in activator (%)	SiO <sub>2</sub> content in activator (%)	Lime stone dust (% by Wt of fly ash)	Type of specimen	Water / fly ash ratio	Curing temp. and duration
AGP1	8	8	0	Paste	0.33	650C and 48 hrs
GL1	8	8	10	Paste	0.33	650C and 48 hrs
GL2	8	8	15	Paste	0.33	650C and 48 hrs

## 3. RESULT AND DISCUSSION

### 3.1 Unexposed geopolymer specimens

#### 3.1.1 Apparent Porosity and Water absorption

Apparent porosity and water absorption of the geopolymer specimens have been determined to assess their pore characteristics. Fig. 5 and Fig. 6 presents the apparent porosity and water absorption for the specimens. Specimens have exhibited decreasing apparent porosity with additional Lime stone dust. GL2 specimen registered an improvement (21.69%) over that of AGP1 specimen (22.81%). Results of water absorption test support the porosity of the geopolymer specimens. Results of water absorption test support the

porosity of the geopolymer specimens. Improvements in water absorption are observed in case of geopolymer Pastes with additional Lime stone dust. The minimum water absorption for Paste specimen was found as low as 4.69% for GL2 specimen with 15% addition of Lime stone dust. An important influence on the strength of AA fly ash geopolymer is exerted by the present of Calcium compound Lime stone dust. Such admixtures exert an unambiguously favorable effect on the early strength development of geopolymer. Development of CSH phases over zeolite phases may be formed which infact reduces the pore sizes.[23]

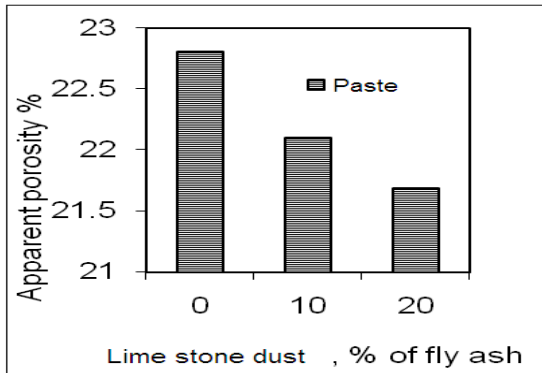


Fig 5: Apparent porosity of Geopolymer specimens.

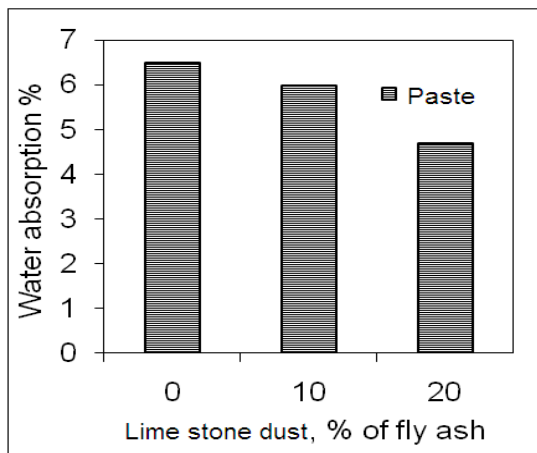


Fig 6: Water absorption of Geopolymer specimens.

### 3.1.2 Water sorptivity

Sorptivity is well thought-out to be an important property associated with durability of concrete specimens [24]. Water ingress into a non saturated cement concrete is due to sorption, driven by the capillary forces. The test for sorptivity is mainly measuring the capillary sorption of water with respect to time. Typical curves for cumulative absorption of water plotted against square root of time are presented in Fig 7, for the geopolymer specimens. For finding sorptivity, slope for the initial linear portion for each of these curves were determined which represents the sorptivity values. Fig 8 presents sorptivity results for the geopolymer Paste specimens. As observed from Fig 7, capillary sorption is initially rapid and becomes significantly low at later stages. The cumulative sorption is highest for AGP1 specimen which has maximum apparent porosity. In the same way, specimen having least apparent porosity GL2 indicates lowest cumulative sorption of water. Rate of suction is higher for Lime stone dust blended geopolymer. It may

be due to increment in suction rate through surface which is accelerated due to reduction in average pore sizes.

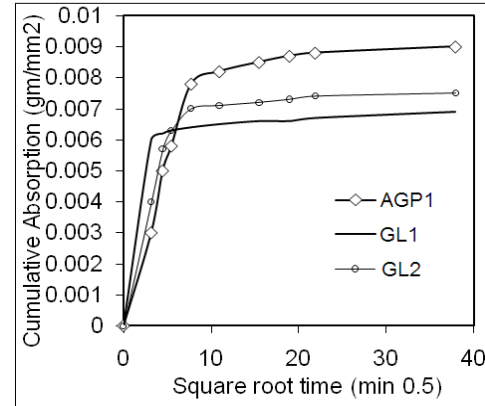


Fig 7: Trend of cumulative sorption of water for geopolymer specimens

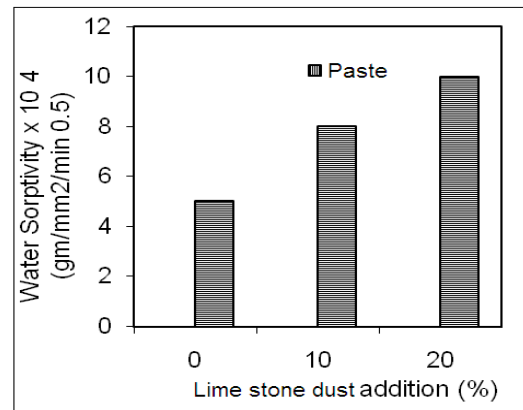


Fig 8: Water sorptivity of Geopolymer specimens

## 3.2 Durability in Magnesium sulphate

### 3.2.1 Visual observation

Changes in physical appearance of geopolymer specimens after exposure to magnesium sulphate solution were monitored at pre-selected intervals. Optical microscope, accuracy .02mm, Zoom capacity of 10 x has been used for the program.

Though the specimens were seen to remain structurally intact. The surface became a little softer as the duration of test progressed but could not be easily scratched with finger nails. Surface geopolymer Paste specimen without Lime stone dust received white deposits throughout the duration of exposure which were soft

and powdery during early stage of exposure but it became harder with time. It was observed that specimen containing Lime stone dust had the little to no white deposits. The white reaction product in question is probably magnesium aluminosilicate [25] . The SEM images has been shown in Fig 10a. In Fig 10b, the presence of Mg, Si, Al have been proved through the EDAX analysis. Such structures have been also reported by some authors [12, 26, 27]. Here the

product has been tested through SEM and EDAX .The decrease in the mean and median size causes significant reduction in the penetration of solution in body of geopolymer structure .Images of surfaces for paste specimens as seen under an optical microscope after 6 weeks in magnesium sulphate solution are shown in Fig. 9a, Fig 9b, Fig 9c. These images clearly reveal the presence of surface deposits basically for sample without Lime stone dust.

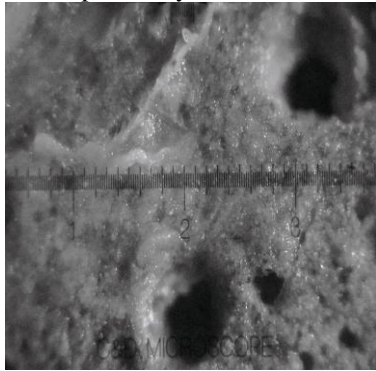


Fig:9a. AGP1



Fig:9b. GL1

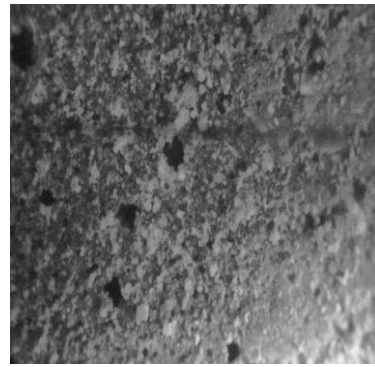


Fig: 9c. GL2

Fig. 9a. Geopolymer Paste specimens ,AGP1 after 6 weeks in 10% magnesium sulphate solution

Fig. 9b. Geopolymer Paste specimens ,GL1 after 6 weeks in 10% magnesium sulphate solution

Fig. 9c. Geopolymer Paste specimens ,GL2 after 6 weeks in 10% magnesium sulphate solution

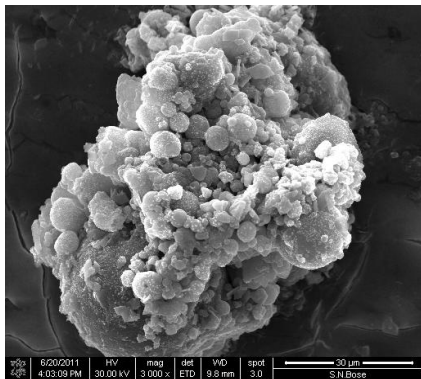


Fig: 10 a. SEM Image of white deposit under 3000X magnification

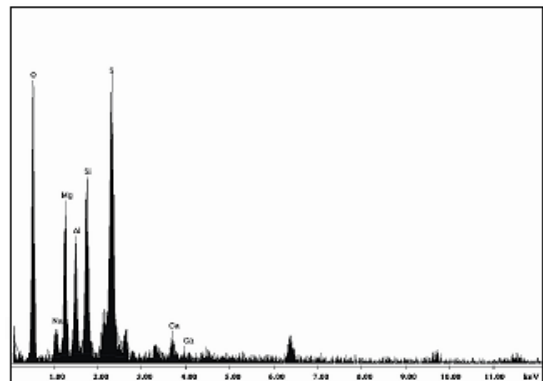


Fig : 10 b. EDAX of white deposits



### 3.2.2 Weight changes

After washing and removal of surface deposits the weights of exposed specimens were measured at regular intervals in saturated surface dry condition. Specimens are cleaned in running cold water for around five minutes and wiped dry with clean lint free cloth and then blown with clean dry air for around 5 minutes. Fig.11 presents the trends of weight changes for the alkali activated fly geopolymer specimens. Rapid increase in weight occurred for all Paste specimens up to 1 week of exposure. This is due to absorption of sulphate solution into the specimens. It is interesting to note that no gradual increase was observed for blended specimens after the early ascend. Weight gain continued gradually for 70 days in specimens without Lime stone dust. It signifies continuous infiltration of sulphate solution and the formation of reaction products due to interaction of geopolymer material with the exposure solution. For blended geopolymer weight of specimens were remain almost constant upto the end of test where the specimens without supplementary Lime stone dust began to decrease beyond 70 days of exposure. The drop in weight may be due to migration of alkalis from the specimens into the solution and also because of crash and dissolution of some reaction products. Ultimate weight for all the samples was higher than initial one. The trend for weight change at later stages of exposure beyond 15 weeks could lead to weight losses in the specimens without Lime stone dust. The trend of weight changes are noticed being influenced by the apparent porosity. Comparing Fig 5 with Fig 11 it could be accomplished in this way that the specimen with much apparent porosity permits more interruption of sulfate solution which causes reaction within the structure of the specimen. This newly constructed reaction product within the interior voids is responsible for weight increment. The defeat is because of the discharge of this reaction product afterward. Maximum weight gains are presented by specimens with maximum porosities.

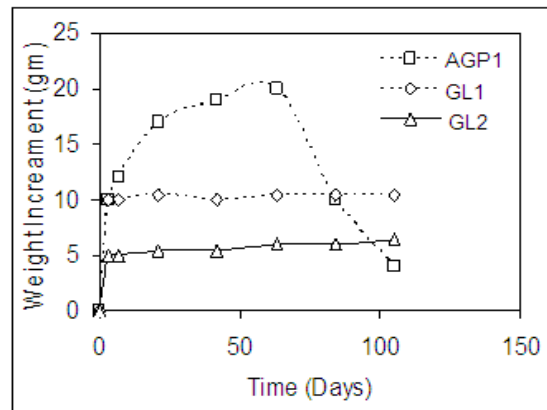


Fig. 11. Weight changes for geopolymer Paste specimens

### 3.3.3 Compressive strength changes

Fig. 12 present the variation of residual compressive strength over the entire period of exposure in 10% magnesium sulphate solution. Compressive strength of sulphate exposed specimen containing no additive reduced with time after getting a initial peak.. Bakharev [9] reported loss of strength due to migration of alkalis from the specimens and also due to diffusion of calcium and sulphur near the surface region. An almost constant strength feature was observed, for all the cases of blended specimens. AGP1, GL1 and GL2 specimens had residual strengths of 65.34%, 98.12% and 99.2% respectively at the end of 15 weeks exposure. It may be noted that residual compressive strength of geopolymer specimens are significantly influenced by their porosities. Though the trend shows drop in strength is obvious for all the cases but specimens with Lime stone dust will remain much intact under sulphate exposure.

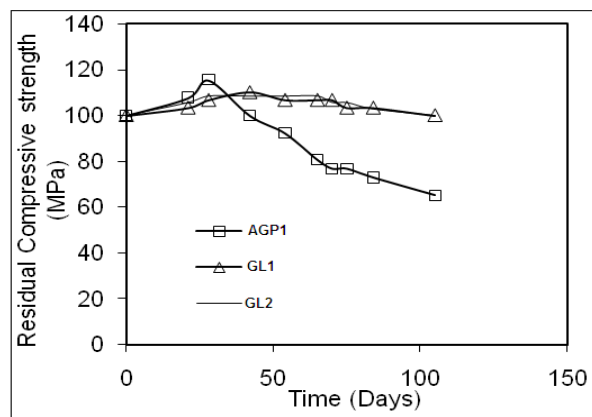


Fig. 12. Residual strength for geopolymer Paste

## 5. CONCLUSIONS

Based on the results of the present experimental investigation, following conclusions are drawn.

Addition of Lime stone dust causes decrement of apparent porosity and water absorption of fly ash based geopolymer Pastes.

Geopolymer Paste integrated with Lime stone dust resulted in higher sorptivity but cumulative sorption is highest for AGP1 specimen which is not blended.

Lime stone dust added geopolymer Pastes exhibits higher compressive strength.

Geopolymer Paste with additional Lime stone dust showed enhanced performance in exposure to magnesium sulphate solution.

Lime stone dust added geopolymer Pastes resulted in higher residual strength after submersion in 10% magnesium sulphate solution.

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