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Optimization of temperature imposed on activator before mixing

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ABSTRACT: *The research has been performed to assess the impact of temperatures on activator prior mixing. In this program, a set of activators having 8% of Na_2O by weight of fly ash were subjected to different temperatures ranging from 20°C to 95°C, for a period of 24 hours and 48 hours. The properties of geopolymer were investigated through workability and compressive strength. Scanning Electron Microscope (SEM) along with EDAX was done to predict the microstructural and mineralogical changes. Geopolymer paste prepared with the activator, which was subjected to a temperature ranging 35°C-50°C for duration of 48 hours, exhibited better mechanical properties.*

KEYWORDS: *Geopolymer, Sodium Silicate, temperature, SEM-EDAX, workability.*

I.INTRODUCTION

The geopolymerisation process also depends on many parameters including the chemical and mineralogical composition of the starting materials, curing temperature, water content, concentration of the alkaline compound, etc. (J. Temuujin. et. al. 2009). It is a geo-synthesis (reaction that chemically integrates minerals) that involves naturally occurring silico-aluminates (Hermann E. et. al. 1999). Geopolymers are cross-linked aluminium silicate networks with charge balancing alkaline cations with water retained in the internal pores (J. Temuujin. et. al. 2009). Geopolymers have been receiving increased attention over the last years because of their superior mechanical, chemical and thermal properties when compared to Portland based cements, and that also with significant lower CO_2 production.

Geopolymers are generally synthesized by activation of an alumino-silicate source like fly ash, blast furnace slag, silica fume with an alkaline hydroxide and silicate solution. The geopolymer gel binder consists of a predominantly X-ray amorphous alumino-silicate network, where the tetrahedral Al sites are charge-balanced by alkali metal cations (Catherine A et al. 2008). As a conventional practice, activating solutions of high soluble silicate concentrations are often used to produce geopolymers so that favorable setting and mechanical properties can be achieved (W.K.W. Lee et al. 2002). From the basic laws of chemistry, according to Le'Chatelier's principle (wikipedia.org, 2012), "If a chemical system at equilibrium experiences a change in concentration, temperature, volume, or partial pressure, then the equilibrium shifts to counteract the imposed change and a new equilibrium is established". It is a well-known fact that the dissolution of NaOH in water is an exothermic process, $NaOH(aq)=Na^+(aq)+OH^-(aq)+(heat)$. If the temperature increases then according to Le'Chatelier's principle, the backward reaction will be favoured. As a result of which, the dissolution rate of sodium hydroxide will be decelerated. Hence there must be an optimum temperature when the dissolution is higher. In this experimental study, we subject the activators to different temperatures and try to assess its impact on the mechanical properties of the geopolymer. The activator was subjected to different temperatures ranging from 20°C to 95°C, for a period of 24 hours. The research was concluded with the effect of that imposing temperature on the geopolymer properties.

II.MATERIALS AND METHOD

MATERIALS PROPERTIES : The chemical composition of low calcium Class F fly ash, collected from Kolaghat Thermal Power Plant near Kolkata, India, is given in TABLE 1. About 75% of particles were finer than 45 micron and Blaine's specific surface was 380m^2/kg. Laboratory grade sodium hydroxide in pellet form (98 percent purity) was used. Sodium silicate solution (Na_2O=8%, SiO_2=26.5% and 65.5% water) with silicate modulus~3.3 and a bulk density of 1410 kg/m was supplied by Loba Chemie Ltd, India. The alkaline activating solution was prepared by dissolving required quantity of sodium hydroxide pellets directly into water. The activator solution (Sodium hydroxide and water) was left at 20°C to 95°C temperature for 24 hours after that predetermined quantity of sodium silicate solution was

added 3 hours before being used to manufacture geopolymer specimens. This activator solution had Na_2O content equal to 8.0% of fly ash and SiO_2/Na_2O ratio equal to 1. Water to Fly ash ratio was of 0.33.

TABLE 1 Chemical analysis report of Fly ash

Chemical composition	Fly ash
SiO_2	56.01
Al_2O_3	29.8
Fe_2O_3	3.58
TiO_2	1.75
CaO	2.36
MgO	0.30
K_2O	0.73
Na_2O	0.61
SO_3	Nil
P_2O_5	0.44
Loss on ignition	0.40

Sample preparation

General : Fly ash was mixed with predetermined quantity of activator solution in a Hobart mixer, for 5 minutes. The mix was then transferred into 50mm x 50mm x 50 mm cubes. Table vibration was provided for 2 minutes to expel any entrapped air. Then, the cubes were cured in an oven for a period of 48 hours at 85^oC and allowed to cool inside the oven (Thakur R. et. al., 2007). After curing the specimens were removed and stored at room temperature at a dry place before testing. Table 2 depicts some important features of typical mixing details. After three days from curing, the geopolymer specimens were tested for its compressive strength and micro structural properties.

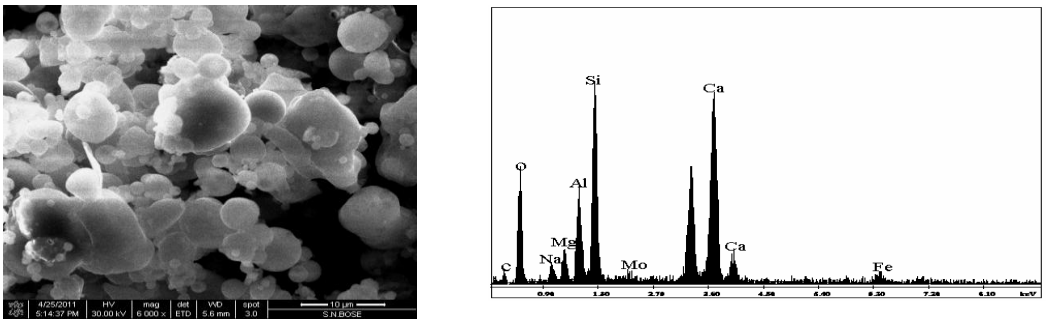


Fig. 1 SEM- EDAX of Fly ash particle

TABLE 2 Mixture composition of geopolymer activator

ID	Temperature imposed on Hydroxide solution prior mixing	Na_2O* content in activator	SiO_2/ Na_2O content in activator	Water to fly ash	Curing temp.	Curing Duration
PS20	20^0C	8	1	0.33	85^0C	48 Hrs.
PS35	35^0C	8	1	0.33	85^0C	48 Hrs.
PS50	50^0C	8	1	0.33	85^0C	48 Hrs.
PS65	65^0C	8	1	0.33	85^0C	48 Hrs.
PS80	80^0C	8	1	0.33	85^0C	48 Hrs.
PS95	95^0C	8	1	0.33	85^0C	48 Hrs.

*(%) of fly ash

Workability : To inspect the flow characteristics of the geopolymer paste, the workability has been predicted by a polar graph has been used in which there are 50 concentric circles and 40 spokes dividing the area into smaller parts to measure the areal change of the slump of the geopolymer paste. A cylindrical container and a circular glass slab (as shown in the Fig. 2-Fig. 5) has been used. The flow behavior of the geopolymer paste is quite different from the flow behavior of cement concrete. The increase or decrease in the workability indicates the change in polycondensation which effect on strength directly.

Typical procedure : In this experiment, the workability of a particular geopolymer sample paste has been determined by careful observation and study of the extent of spread of that particular geopolymer paste. A 7mm thick circular glass slab of 50cm diameter was used in this set up. A polar graph was used to study the extent of the spread. The polar graph consists of 50 concentric circles, equally spaced with the outermost circle having a diameter of 50cm.The circle was again divided into a number of sectors by 40 numbers of spokes (i.e. radial lines).This polar graph was placed below the glass slab. A brass cylinder, 6cm in diameter and 8cm in height, was used as a mould to hold the geopolymer paste. The mould was placed exactly at the center of the setup, ensuring that its center coincides with the center of the glass slab and that of the polar graph. After filling the mould with geopolymer paste it was raised slowly, ensuring that it was raised vertically, and thus allowing the paste to flow. After the flow had stopped, the readings corresponding to the outermost periphery of the flow was taken. The nth circle has n cm diameter. By the radii of all the concentric circles, the area of spread was calculated. In this study this phenomena was taken as the prime indication of the effect of imposing temperature on activator to the green geopolymer.

Area factor : factor denoted as AREA FACTOR, defined by the ratio of the final area of the slump to the area of the cylinder was calculated to assess the differential consistency. The increase of the area factor indicates the increase of geopolymer mobility.

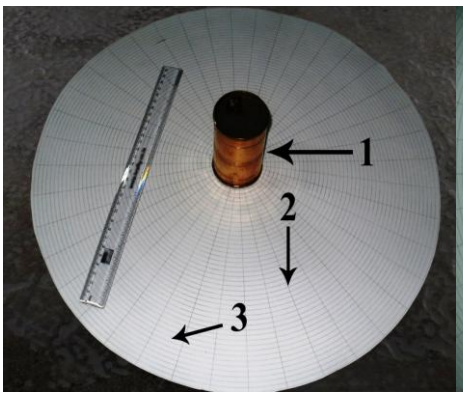


Fig. 2 The experimental setup

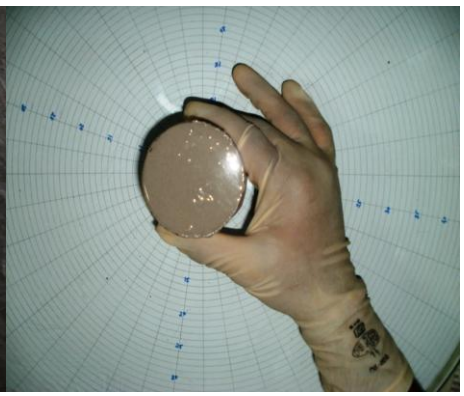


Fig. 3 Flow pattern of geopolymer paste

1.Cylindrical bras container. 2.Polar graph. 3.Circular glass slab.

Fig. 4 For sample PS20

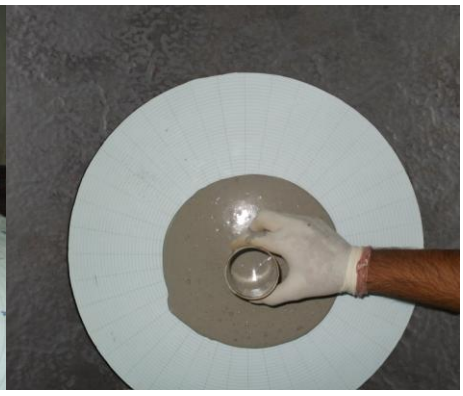


Fig. 5: For sample PS50

III.RESULTS AND DISCUSSION

Workability : With increase in OH- concentration, (Zuhua Z. et. al., 2009) the dissolution of fly ash is accelerated. Development of Si–O–Al–O skeleton is proceeded with the compensation of charge on Al atoms. These charges are compensated by Na+ ions (František škvára et. al. 2006). Hence it is evident that optimum polymerization is not possible without presence of adequate Na^+ and OH^- in the activator solution. Presence of Na^+ and OH^- in activator depends on dissolution of Sodium Hydroxide which is affected by temperature imposed on it. Maximum area factor is obtained for sample PS35 and PS50 which implies maximum dissolution and polymerization .In both the cases of PS20 and PS95 very low area factor were obtained.

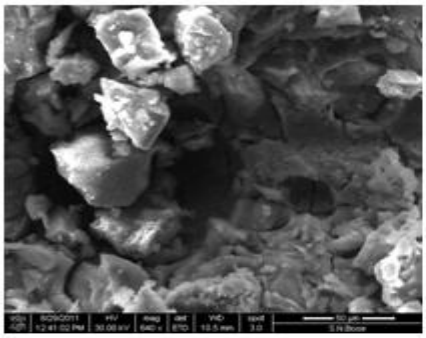
TABLE 3 Results of workability test of the geopolymer paste

SAMPLE ID	INITIAL DIAMETER(D1) (cm)	FINAL EQUIVALENT DIAMETER (D2) (cm)	INTIAL AREA (A1) cm^2	FINAL AREA AFTER FLOW(A2) (cm^2)	AREA FACTOR=A2/A1
PS15	6	6	28.26	28.26	1.0
PS30	6	20	28.26	314	11.11111
PS45	6	26	28.26	530.66	18.77778
PS60	6	18	28.26	254.34	9
PS75	6	9	28.26	63.585	2.25
PS90	6	6	28.26	28.26	1.0

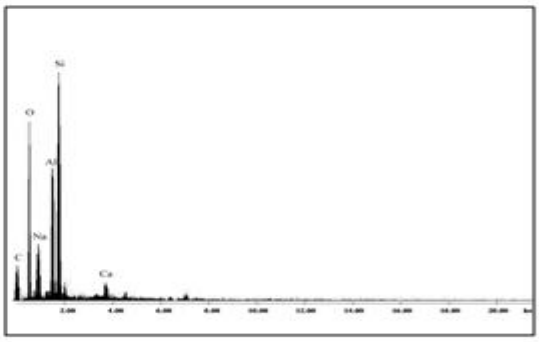
Microstructural investigations : At least 25 mm^2 of sampling area is needed to obtain a reliable result (J. Van Brakel, 1981). Fig. 6 characterize the ESEM micrographs for geopolymer paste specimens PS20, PS50, PS65, PS80 and PS95 along with their EDAX chart. It depicts an unreacted morphology for geopolymer sample PS20 and PS95.

The micrographs reveal mostly an amorphous phase in case of PS35 and PS50. In PS20 and PS90 unreacted crystalline component is clearly visible this may be Sodium Hydroxide.

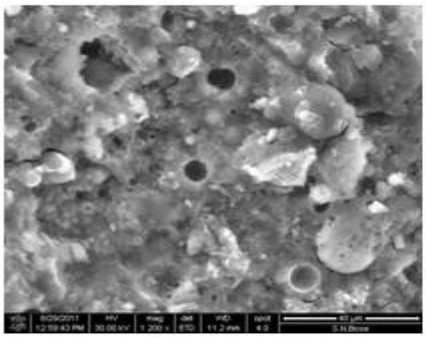
EDAX spectra of specimens show major elements such as Silicone (Si), Oxygen (O), Aluminum (Al), and Sodium (Na). The weight percentages according to EDAX quantification of PS20 and PS95 subjected at a specified position were O (38.8%), Na (12.12%) and O (27.62%), Na (20.5%) respectively. The weight percentage of those elements in case of PS50 shows O (44.32%), Si (19.32%), Al (12.32%), Na (8.13%) and Ca (0.24). For PS65 EDAX analysis capitulated the following O (35.7%), Si (17.27%), Al (9.02%), and Ca (3.91).



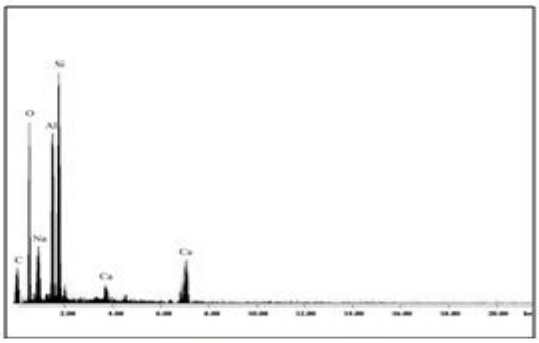
SEM Image of Sample PS20



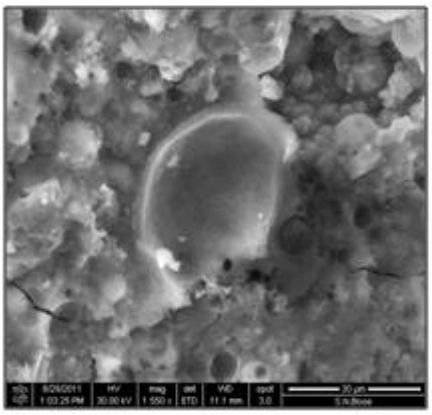
EDAX of Sample PS20



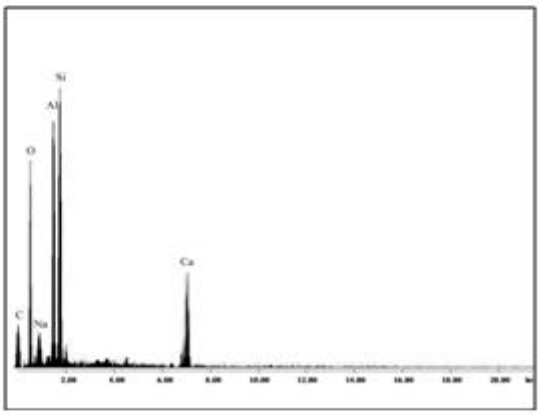
SEM Image of Sample PS50



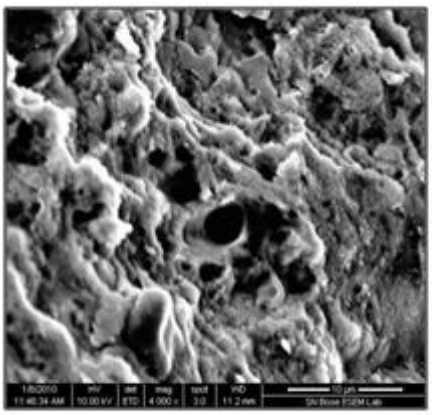
EDAX of Sample PS50



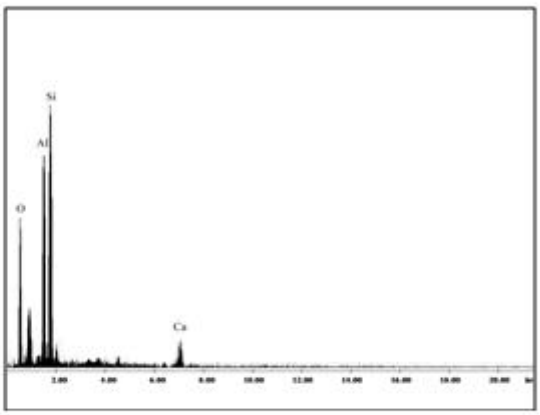
SEM Image of Sample PS65



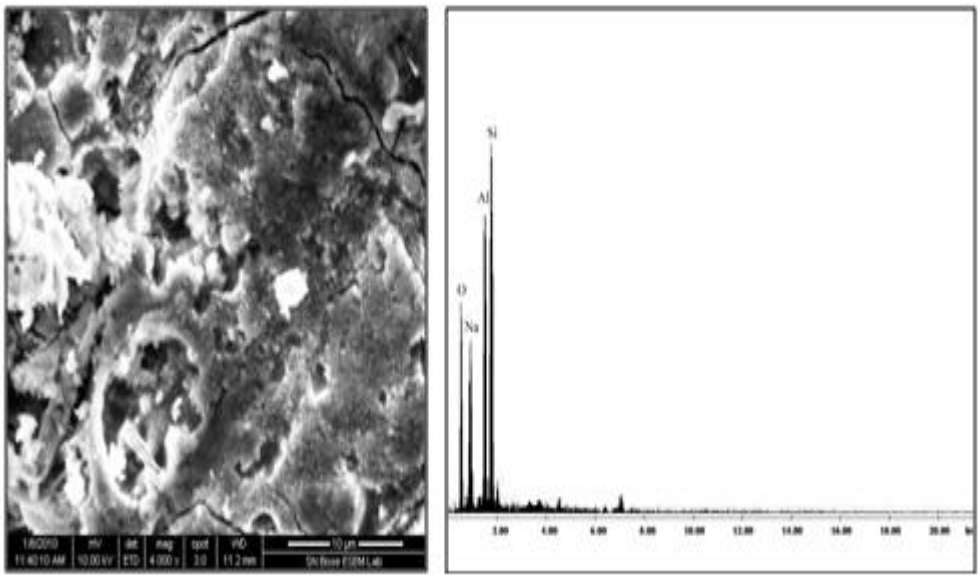
EDAX of Sample PS65



SEM Image of Sample PS80



EDAX of Sample PS80



SEM Image of Sample PS95 EDAX of Sample PS95

Fig. 6 SEM –EDAX analysis of different geopolymer specimens

Compressive strength : Successful result was defined only when there was a single break of the materials (Kriven, W. M. et. al. 2008). Fracture behavior of the geopolymer samples was often irregular as few areas were chip of before to ultimate facture. The compressive strength of the geopolymer paste was determined after 3 days respectively from manufacture. Ten specimens for each series were crushed in a digital compression testing machine and the average is reported. Compressive strength obtained for the specimens are presented in Fig. 7. Sample PS35 gave maximum compressive strength of 37 Mpa. Lowest compressive strength was achieved for sample PS20 as 19 MPa. For sample PS95 the compressive strength was measured as 15 Mpa.

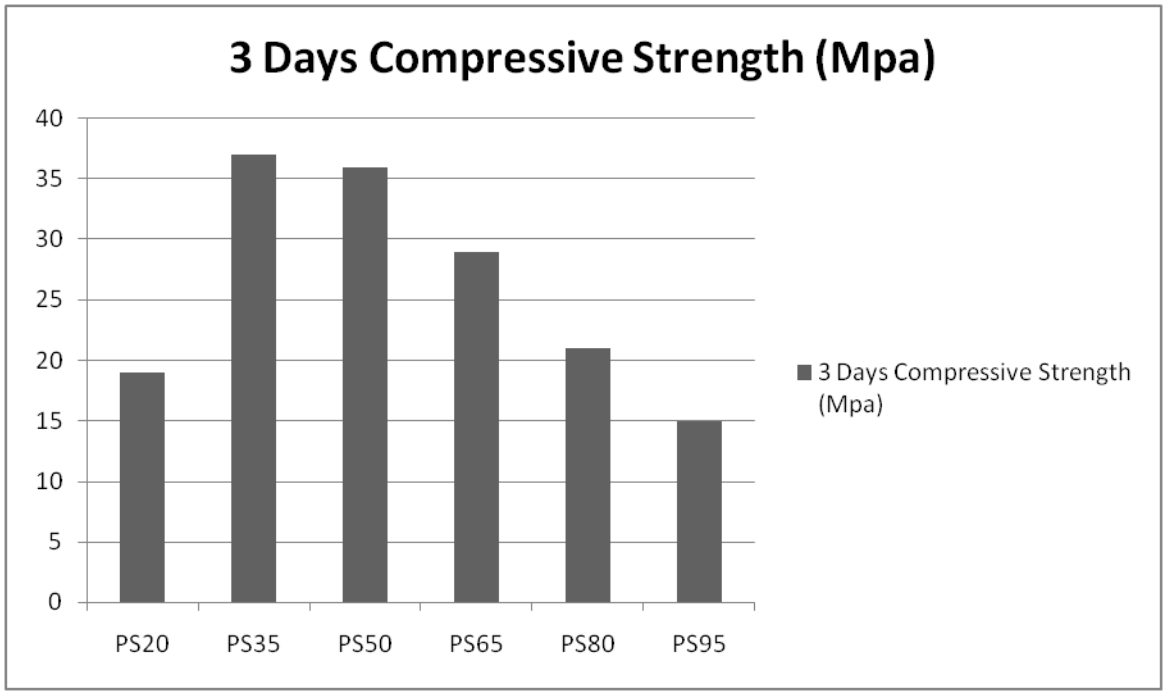


Fig. 7 Compressive strength of different Geopolymer sample

IV.CONCLUSION

[1] Imposing temperature within a range of 35^{o}C to 50^{o}C to the activator prior mixing is much favouarble to compressive strength.

[2] Compressive strength was very low for specimens PS20 and PS95 while imposing temperature on the activator were 200C and 950C respectively before mixing.

[3] Structural morphology shows better reaction for PS35and PS50 under Scanning Electron Microscopy.

[4] Consistency of geopolymer in green state is low for sample PS20 and PS95 implies less dissolution geopolymer species.

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