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Effect of %Na_2O and %Sio_2 on Apperent Porosity And Sorptivity of Flyash Based Geopolymer

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ABSTRACT: *- The objectives of the present research is to appreciate the effect of %Na_2O and %SiO_2 on apparent porosity and sorptivity of fly ash based geopolymer mortar . No simple direct relationship between compressive strength and sorptivity could be established. However, generally there is decrease in water sorptivity and water absorption with increase in strength Locally available low calcium fly ash from a thermal power plant located near Kolkata, India, was used . Fly ash used are lignite coal based and falls under class F category. This study helped arrive at certain level of understanding which will be useful to the researchers and manufacturers.*

Keywords: *– Compressive strength, Geopolymer, Porosity, Microstructure, Flyash*

I. INTRODUCTION

Geopolymers is an inorganic polymeric materials formed by activating silica-aluminum rich minerals with alkaline or alkaline-silicate solution at ambient or higher temperature level. Potential applications includes: fire resistant materials, thermal insulating material, low energy tiles , waste containment, paver blocks etc.

Geopolymerisation is a very complex multiphase exothermic process, involving a series of dissolution-reorientation-solidification reaction analogous to zeolite synthesis. High alkaline solutions are used to induce the silicon and aluminium atoms in the source material to dissolve, forming three dimensional polymeric structure consisting of -Si-O-Al-O- bonds, represented as follows

$$M_n [-(SiO_2)_z–AlO_2]_n . wH_2O$$

Where: M = the alkaline element or cation such as potassium, sodium or calcium; the symbol – indicates the presence of a bond, n is the degree of polycondensation or polymerisation; z is 1, 2, 3, or higher. The exact reaction mechanism which explains the setting and hardening of geopolymers is not yet quite understood, although it is thought to be dependent on the aluminosilicate base material as well as on the composition of alkaline activator.

Optimization of such a complex system requires systematic study of a number of synthesizing parameters as well as of their interactions. Secondly, fly ash from different sources show different level of reactivity under specific geopolymer synthesis conditions and consequently affects the final properties. Hence , for manufacturing high performance geopolymer binder from fly ash, it is necessary to understand the effects of a various synthesis parameters and their relationship . The Geopolymer mix composition is normally controlled by adjusting alkali and silicate content of activating solution. The SiO_2/Al_2O_3 molar ratio is an extremely important parameter which has major influence on setting time and workability which in turn affects physical and mechanical properties as well as on its microstructure.

The properties of fly ash based geopolymer mortar depends on chemical composition and quantity of fly ash as well as activator solution. It may be noted here that, %Na_2O (by weight of fly ash) and %SiO_2 of the mix significantly affect apparent porosity and sorptivity of geopolymer mortar. Moreover, these studies are important for locally available fly ash for wide applications in the industry.

II. EXPERIMENTAL INVESTIGATION

The experimental investigation has been conducted at Jadavpur University, Kolkata, India. The humidity and ambient temperature in the laboratory are ranging from 75% to 90% and 25°C to 40°C respectively. The experimental methodology was divided in two main parts: (1) Preparation of geopolymer mortar (2) Testing to obtain apparent porosity and sorptivity geopolymer mortar. The laboratory tests were conducted as per relevant Indian standard /ASTM codes. Outline of the tests conducted are provided below.

2.1 Bulk density and Apparent porosity

The bulk density and apparent porosity for geopolymer specimen was determined according to Archimedes principle with water as immersion medium. The procedure adopted was as follows: dry all specimens in a ventilated oven at 65°C for 48 hours. Record the weight of dried specimens as W_d. Immerse the specimens in water at room temperature (28°C) for minimum 48 hours. Weight the specimens while suspended by a thin wire and completely submerged in water and record W_i. Remove specimens from water and allow water to drain off by placing them on a wire mesh, removing visible surface water with a damp cloth; weigh and record saturated weight as $W_{s.}$ The bulk density and apparent porosity was then calculated as follow:

Dry density (Kg/m^3) = $[W_d / (W_s-W_i)] \times 1000$
Apparent Porosity (%) = $[(W_s-W_d) / (W_s-W_i)] \times 100$

2.2 Water Absorption

The volume of pore space in specimen matrix, as distinct from the ease with which a fluid can penetrate it, is measured as absorption. Water absorption is usually measured by drying a specimen to a constant mass, immersing it in water, and measuring the increase in mass as a percentage of dry mass. In the present research, water absorption of specimens was determined as per ASTM C-642. The 28 days aged specimens were dried for 48 hours at 65°C & then immersed in water for 24 hours. The test specimens soaked in water were removed from the immersion container, wiped clean and weighted immediately in saturated-surface-dry (SSD) condition to find increase in mass.

2.3 Water Sorptivity

Permeability test measure the response of concrete to pressure, which is rarely the driving force of fluid entering concrete, there is a need of another type of test. Such a test measures the rate of absorption of water by capillary suction of unsaturated concrete placed in contact with water; no head of water exists. The sorptivity test according to Neville A.M., determines the rate of capillary rise absorption by mortar/ concrete cube. The specimens rest on small supports in a manner such that only the lowest 2 to 5mm of the cube is submerged. The increase in the mass of the prism with time is recorded. The specimens should be dried prior to the experiment. It has been shown that there exists a relation of form.

$$I= S\sqrt{t} \tag{1}$$

Where,
I=increase in mass per unit area (gm/mm^2) since beginning of the test per unit of cross-sectional area in contact with water; as increase in mass is due to ingress of water. It is expressed in mm.
t=time, measured in minutes, at which the mass is determined.
S=Sorptivity in $mm/min^{0.5}$

2.4 Raw Materials

Typical locally available low calcium Class F fly ash from a thermal power station near Kolkata was used throughout the research. The alkaline activator solutions were prepared by mixing sodium hydroxide in pellets form in sodium silicate solution and distilled water.

2.4.1 FLYASH

The chemical compositions of the fly ash, as determined by X-Ray Fluorescence (XRF) analysis, are given in Table 2.1.

Table 2.1: Chemical composition of the Fly ash (mass %)

Oxide	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**TiO_2**	**CaO**	**MgO**	**K_2O**	**Na_2O**	**LOI**
Mass (%)	**55.15**	**30.85**	**3.15**	**1.85**	**2.45**	**0.35**	**0.8**	**0.65**	**0.45**

The total percentage of (SiO_2+ Al_2O_3+ Fe_2O_3) is greater than 70%. The calcium oxide content is less than 10%. Hence, as per ASTM C 6128-03, it can be classified as class F fly ash (or siliceous pulverised fuel ash conforming to IS 3812(Part-I)-2003 specifications). The colour of the fly ash was dark gray. About 90% of the particles were smaller than $45\mu m$, and the Blain specific surface area of fly ash was $395 m^2/kg$. The Scanning Electron Microscopy (SEM) image of fly ash show that the fly ash particle are generally spherical in shape of varying size. The mineral composition of fly ash was obtained by XRD analysis and it was found that the major crystalline constituents of fly ash included quartz (SiO_2), mullite (Al_2O_3), and magnetite (Fe_3O_4). The fly ash is also constituted of an X-ray amorphous phases indicated by the broad hump registered between 2θ =20° and 30°

2.4.2 ACTIVATOR SOLUTION

The alkaline activator was a combination of sodium silicate and sodium hydroxide solutions. The sodium hydroxide solids were laboratory grade in pellets form, with a specific gravity of 2.15, 97% purity. To avoid effects of unknown contaminants, distilled water was used for preparing the activator solutions.

The sodium hydroxide (NaOH) solution was prepared by dissolving NaOH pellets in water. The mass of NaOH solids in a solution was varied depending on the required concentration of the solution. The chemical composition of the sodium silicate solution was Na_2O=14.7%, SiO_2=29.4%, and water 55.9% by mass. The other characteristics of the sodium silicate solution were specific gravity=1.48 g/cc. The activator solution was prepared at least one day prior to its use .

2.5 MANUFACTURING PROCESS

Following steps were followed during manufacturing of Geopolymer

- Mixing of sodium silicate solution, sodium hydroxide pellets and water according to predefined proportion to make alkaline activator, at least one day prior to its use .
- Hobart mixer with rotating blades, was used for preparing geopolymer mix
- Mixing of fly ash and alkaline activator in the Hobart mixer for about five to six minutes to get a homogeneous paste.
- Apparent Porosity and Sorptivity tests were conducted.

2.6 SYTHESIZING PARAMETERS

The proportion of geopolymer mix was varied by changing quantity and proportion of sodium silicate and sodium hydroxide in activating solution. The effect of alkali content %Na_2O and silicate content %SiO_2 on apparent porosity and sorptivity of geopolymer mortar was studied.

2.7 MIX COMPOSITION

The effect of water content expressed as water to geopolymer binder (geopolymer binder is sum total of mass of fly ash + mass of solids in activating solution). Water to Geopolymer binder ratio was varied from 0.325 to 0.365 by changing quantity of water. The Geopolymer paste were prepared by varying % Na_2O from 4.25% to 10.25% and % SiO_2 from 4.5% to 17% . The water to fly ash ratio of 0.325 was kept constant. Again, water to fly ash ratio was varied from 0.325 to 0.365, keeping % Na_2O and % SiO_2 constant. Sand to fly ash ratio was strictly maintained at 1:1.

III. DISCUSSION ON TEST RESULTS

3.1 Bulk density and Apparent porosity

The specimen bulk density and apparent porosity of Geopolymer mortar specimens is summarized in Table 3.1 and 3.2.

Table-3.1: Effect of %Na_2O on Bulk density and Apparent porosity

% Na_2O	% SiO_2	Water/ Fly ash Ratio	Bulk Density (Kg/m3)	Apparent Porosity (%)
4.25	8	0.325	1820	20.1
6.25	8	0.325	1900	16.3
8.25	8	0.325	1980	9.1
10.25	8	0.325	2010	8.2

The alkali content (%Na_2O) of the mix was varied from 4.25% to 10.25% and %SiO_2 and water to fly ash ratio was kept constant to 8% and 0.325 respectively. The maximum bulk density of 2010 Kg/m^3 and minimum porosity of 8.2%.

It has been observed that bulk density increased and apparent porosity decreased almost linearly with increasing alkali content from 4.25% to 8.25 %. However, further increasing alkali content to 10.25% , the effect was not significant. The possible reason for lower bulk density and higher apparent porosity at lower alkali content and vice versa is incomplete Geopolymerisation of fly ash. At lower alkali content of 4.25% of Na_2O, the dissolution of fly ash get reduced and less Geopolymer gel is formed creating more porosity among unreacted fly ash particles in the Geopolymer matrix where as at higher alkali content of 8.25% most of the reactive phases in fly ash get converted to gel making the matrix more compact and dense resulting in higher bulk density and low porosity.

Table-3.2 : Effect of %SiO_2 on Bulk density and Apparent porosity

% Na_2O	% SiO_2	Water/ Fly ash Ratio	Bulk Density (Kg/m^3)	Apparent Porosity (%)
8	0.00	0.325	1880	16.28
8	4.25	0.325	1951	11.66
8	8.75	0.325	2012	8.92
8	13	0.325	2037	8.72
8	17.00	0.325	2060	8.22

%Na_2O and water to fly ash ratio was kept constant to 8 % and 0.325 respectively. The maximum bulk density of 2060 Kg/m^3 and minimum porosity of 8.22%. It has been observed that bulk density increased almost linearly with increasing silicate content and no substantial improvement was observed for silicate ratio beyond nearly 1.0.

3.2 Water absorption

Table 3.3 and 3.4 presents water absorption of the fly ash based Geopolymers mortar for change in alkali content and silica content. The water-to-fly ash ratio and sand to binder ratio of the mix was kept constant to 0.325 and 1.0 respectively.

Table 3.3 Effect of %Na_2O on Water absorption

% Na_2O (a)	%SiO_2	Water/ Fly ash ratio	% Water absorption
4.25	8	0.325	11.7
6.25	8	0.325	10.1
8.25	8	0.325	7.3
10.25	8	0.325	6.9

It has been observed from the Table-3.3, that the Geopolymer mortar specimen having alkali content of 4.25%, showed maximum water absorption of 11.7%. Whereas, specimen having alkali content of 10.25% revealed minimum water absorption of 6.9%. It is also observed that water absorption decreased with increasing %Na_2O in the Geopolymer mix. At lower alkali content of 4.25% and 6.25% the water absorption was more indicating higher void content in the Geopolymer specimen. Moreover, the Geopolymerisation reaction is not complete at lower alkali content and large number of unreacted fly ash particles also present in the Geopolymer matrix. The fly ash particles content nano pores and adsorb additional water. At higher alkali content of 8.25% and 10.25% the water absorption is less due to lower void spaces. At higher alkali content more quantity of fly ash particles dissolved forming higher quantity of gel which reduces void space in the matrix resulted in decreasing water absorption.

Table 3.4 Effect of % SiO_2 on Water absorption

% Na_2O	%SiO_2	Water/ Fly ash ratio	% water absorption
8	0.00	0.325	11.32
8	4.25	0.325	9.02
8	8.50	0.325	8.11
8	12.75	0.325	7.41
8	17.00	0.325	7.23

Table-3.4, presents the water absorption of the fly ash based Geopolymers mortar specimens for different silica content. Water to fly ash ratio and alkali content (%Na_2O) of Geopolymers mix was kept constant to 0.325 and 8% respectively.

The trend indicated that water absorption increased almost linearly with silicate content in the Geopolymer mix. The water absorption was more indicating higher void content due to incomplete process of Geopolymerisation. It has been observed during earlier study that addition of soluble silicate makes the Geopolymer structure more homogeneous and compact. Higher silica content involved formation of higher quantity of aluminosilicate gel and provides very good interparticle bonding. The silicate occupies the void spaces between the fly ash particles resulted in lower water absorption.

3.3 Water Sorptivity

The strength is not an appropriate parameter for assessing durability. The characterisation of pore structure can be appreciated to some extent by measuring rate of water absorption by capillary suction of unsaturated specimen placed in contact with water under no water head , known as sorptivity. The measurement of sorptivity has primary importance in durability assessment of Geopolymers. The experimental results on water sorptivity are shown in Table 3.5 and Table 3.6 respectively.

Table 3.5 Effect of Na_2O% on Water sorptivity

% Na_2O	%SiO_2	Sorptivity $s= i/\sqrt{t}$
4.25	8	7.34E-04
6.25	8	6.84E-04
8.25	8	4.12E-04
10.25	8	3.96E-04

Table 3.6 Effect of %SiO_2 on Water Sorptivity

% Na_2O	%SiO_2	Sorptivity $s= i/\sqrt{t}$
8.5	0.00	5.80E-04
8.5	4.25	5.0E-04
8.5	8.50	4.1E-04
8.5	12.75	3.84E-04
8.5	17.00	3.62E-04

It has been observed from above results that, the rate of water absorption through capillary absorption is almost linear in the initial period of four hours. After four hours the sample gets almost saturated and there was no further water absorption. Cumulative water absorption decreased with increasing %Na_2O. It decreased with increasing SiO_2/Na_2O ratio. It is observed that the water sorptivity is more at lower alkali content . However , there is no substantial reduction when % of Na_2O increased from 8.25% to 10.25%, which indicate that the pore structure is almost similar even though the strength values are quite different. The water sorptivity decreased almost linearly with increasing silicate ratio. Results indicated that addition of soluble silicate has positive effect on water sorptivity. The pore structure of the Geopolymer composite becomes more compact and homogeneous with increasing silicate content resulted in decreasing water sorptivity. From the discussion it can be concluded that water sorptivity of Geopolymer mortar specimen is significantly affected by alkali content and silicate ratio of Geopolymer mix. Addition of soluble silica has positive effect on sorptivity as pore structure becomes more dense and compact at higher dosage of soluble silicate.

IV. CONCLUSION

The study revealed that the apparent porosity and sorptivity as well as microstructure depended basically on alkali content and silica content. Strong alkali solutions are needed to dissolve fly ash during the process of geopolymerisation. There is no direct relationship between compressive strength and sorptivity. However, generally there is decrease in water sorptivity and water absorption with increase in compressive strength and bulk density.

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