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Microstructure Studies on the Effect of Alkaline Activators to Flyash Ratio on Geopolymer Concrete

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Abstract: Fly ash-based geopolymers are new binding materials produced to replace the ordinary Portland cement used in concrete. In this research, the effect of alkaline activators to flyash ratio on the compressive, split tensile, flexural strength and microstructural study of fly ash-based geopolymers were studied. Alkaline activators to fly ash ratios of 0.30, 0.35, 0.40 and 0.45 and ratio of sodium silicate (Na2SiO3) to sodium hydroxide (NaOH) maintained constant throughout the study. The alkaline activator solution was prepared by mixing sodium silicate (Na2SiO3) with a 12M NaOH solution. The samples were casted and cured at a temperature 60° C for 24 hours in Dry oven and kept at room temperature till the testing (7days and 28 days). The test results indicated that the compressive, split tensile and flexural strength increased when the ratio of alkaline activator to fly ash was decreased.

The ratio of 0.30 produced the maximum compressive strength, which was 68.02 MPa at 28 days curing. SEM (Scanning Electron Microscope) test conducted on geopolymer concrete samples and observed that alkaline activators to fly ash ratio of 0.30 and 0.35, geopolymer samples has the continuous, homogenous matrices and less porous structure because of the high dissolution of the fly ash particles in the alkaline activator solution.

Keywords: Flyash, Alkaline Activator, Geopolymerisation, Compressive strength, Split tensile strength, Flexural strength, SEM analysis, Geopolymer

I. INTRODUCTION

The development of geopolymer concrete containing flyash is an attempt to answer the challenge to produce more environmentally friendly concrete. The use of by-product material, i.e. fly ash, as a base material for concrete binder to totally replace the use of portland cement through geopolymerisation process has been attracting a lot of attention globally. This attempt results in twofold benefits, i.e. to provide a solution with regard to the concern on the carbon dioxide emission from Portland cement production, and to provide way to effectively use fly ash [1].

The contribution of ordinary Portland cement (OPC) production worldwide to greenhouse gas emissions is estimated to be approximately 7% of the total greenhouse gas emissions to the earth's atmosphere [2]. Also, it has been reported that many concrete structures, especially those built in corrosive environments, start to deteriorate after 20 to 30 years, even though they have been designed for more than 50 years of service life [3].

Usage of concrete around the world is second only to water so the concrete industry has recognized issues and suggested an industrial waste Fly ash as a replacement to Portland cement for making concrete.

In concrete industry another forms of cementitious materials called geopolymer has been developed by Davidovits (1991) and proposed that binders could be produced by a polymeric reaction of alkaline liquids with the silicon and the aluminium in source materials of geological origin or by-product materials such as fly ash and rice husk ash that are rich in silicon and aluminium. He termed these binders as "geopolymers". Fly ash, one of the source materials for geopolymer binders, is available abundantly worldwide, but to date its utilization is limited. A comprehensive research program was commenced in 2001 on "Low-Calcium Fly Ash-Based Geopolymer Concrete". It was found that heat-cured low-calcium fly ash-based geopolymer concrete possesses high compressive strength, undergoes very little drying shrinkage and moderately low creep, and shows excellent resistance to sulphate and acid attack. Several researchers have reported that geopolymers do not suffer from alkali-aggregate reaction and possess excellent fire resistant.

II. OBJECTIVE

The main aim of the present work is to study the mechanical properties and microstructures of geopolymer concrete by keeping fixed molarity and constant ratio of sodium silicate to sodium hydroxide solution for different mix proportions, which shows better results.

III. MATERIALS

A. *Fly Ash*

The low-calcium dry fly ash used in the entire investigation brought from thermal power plant, Raichuru district, Karnataka. Specifications of Fly Ash given by the supplier shown in the Table 1 and Table 2.

Table 1: Chemical composition of fly ash

<table>
<tr><td>Chemical composition</td><td>Recommended by
RTPS (% mass)</td><td>Recommended by
IS 3812-1981 (% mass)</td></tr>
<tr><td>Silica</td><td>55 – 65</td><td rowspan="3">70</td></tr>
<tr><td>Iron Oxide</td><td>5 – 7</td></tr>
<tr><td>Aluminium Oxide</td><td>22 - 25</td></tr>
<tr><td>Calcium Oxide</td><td>5 – 7</td><td>-</td></tr>
<tr><td>Magnesium Oxide</td><td><1</td><td>5 max</td></tr>
<tr><td>Alkali Oxide</td><td><1</td><td>1.5 max</td></tr>
<tr><td>Loss of ignition</td><td>1 – 1.5</td><td>5 max</td></tr>
</table>

Table 2: Physical properties of fly ash

Properties	Results
Physical state	Powder form
Color	Grey
Specific Gravity	1.98
Fineness (45 micron)	28%

B. *Aggregates*

In this study coarse aggregates and river sand brought from local quarries nearby city. The surface dry conditioned coarse aggregates (SSD) of size 20mm, 14mm and 7mm taken for this experiment work.

Table 3: Physical properties of fine and coarse aggregates

Physical property	Fine aggregates	Coarse aggregates
Specific gravity	2.50	2.70
Bulk density	1.67(g/cm3)	1.65(g/cm3)
Fineness modulus	2.6	-
Silt content	0.6%	-

C. *Super Plasticiser*

In order to improve the workability of fresh concrete, high-range water-reducing naphthalene based super plasticiser i.e. Conplast SP-430 used for this experiment.

D. *Alkaline Liquid*

For an alkaline liquid, sodium silicate (Na_2SiO_3) solution and sodium hydroxide (NaOH) with 97% purity in pellet form used. In this eintire investigation, an alkaline activator to fly ash ratios of 0.45, 0.40, 0.35 and 0.30 kept constant for concrete mix of M30, M40, M50 and M60 respectivily with a Na_2SiO_3:NaOH mass ratio of 2.5 and Molarity 12M for 7 and 28 days curing.

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Fig.1 NaOH pellets


Fig. 2 Sodium Silicate Solution (Na_2SiO_3)


Fig. 3 Mixing of Na_2SiO_3 with NaOH

IV. EXPERIMENTAL WORK

A. *Molding, Curing and Testing*

All dry ingredients of geopolymer concrete mixed thoroughly in mechanical mixer and fresh, homogeneous, geopolymer paste collected into standard cube molds of size 150 x 150 x 150 mm, similarly cylinder molds of size 100 mm diameter, 200 mm height and beam molds of size 100 x 100 x 500 mm. The samples cured in dry oven for 24 hours with temperature of 60°C. After 24 hours samples kept at room temperature for 7 and 28 days. After age of curing, samples tested in universal testing machine. For Scanning electron microscopy (SEM) test, samples were cut into cubical shape of size 10 x 15 mm to analyze the microstructure of the geopolymer concrete samples.

B. *Results and Discussions*

Fig.4 shows the workability of geopolymer concrete for different mix and found that fresh GPC mixes highly viscous and cohesive with medium to high slump. The workability decreases with increase in the grade of the concrete, because of the decrease in the ratio of water to geopolymer solids. Maximum slump achieved for the mix of M30 with ratio of alkaline activators to flyash 0.45. As the grade of the concrete increases, the mix becomes stiffer, decreasing the workability, which results in strength reduction.

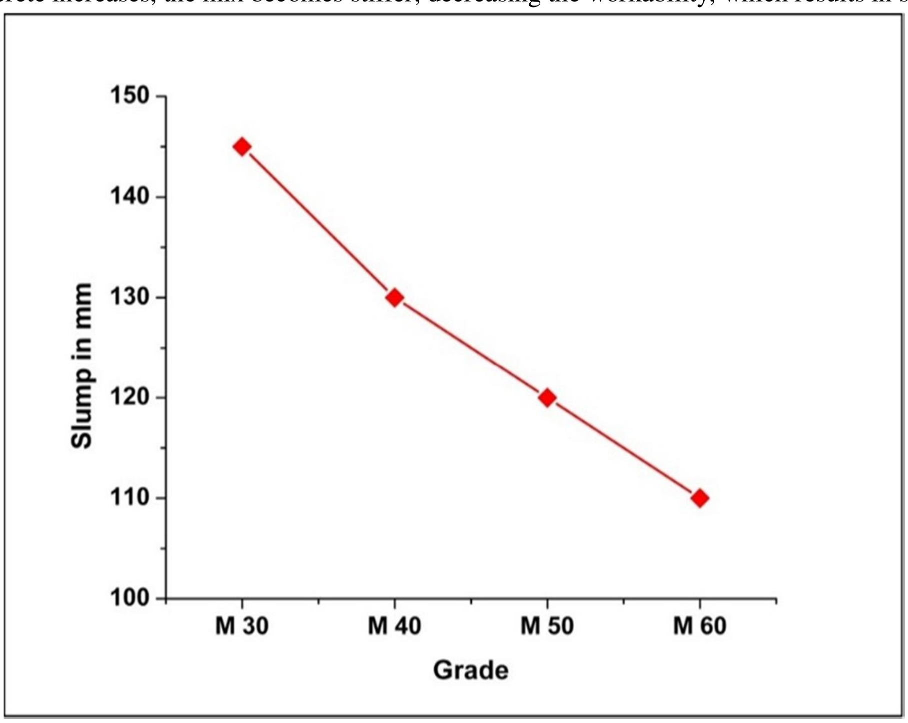


Fig. 4 Workability of geopolymer concrete

Compressive strength of the GPC increases with increase in the curing period. The maximum strength was obtained when the ratio of alkaline activator to flyash by mass was 0.30. There is no drastic increase in the strength as the curing period increases from 7 to 28 days. Lower the ratio of alkaline activator to flyash by mass results in higher compressive strength.

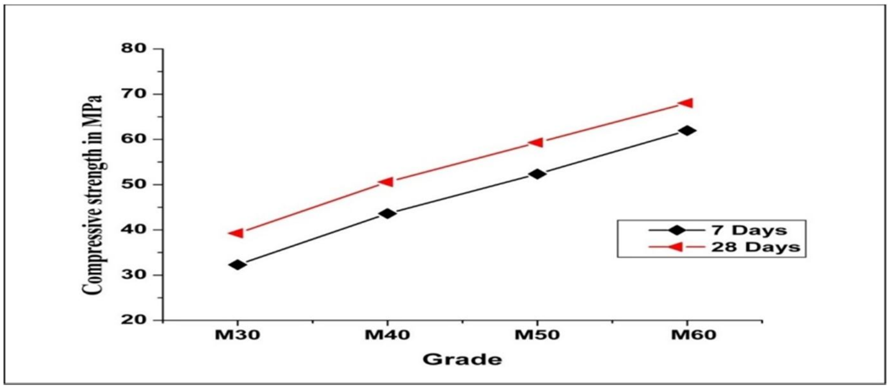


Fig. 5 Compressive Strength v/s Grade for Ratio of Na_2SiO_3: NaOH =2.5 and Molarity 12M

In split tensile test minimum strength observed for mix M30 with ratio of alkaline activator to flyash by mass is 0.45 with strength of 3.55 MPa at 7 days and 4.12 MPa at 28 days curing and maximum results was observed for mix M60 with ratio of alkaline activator to flyash by mass is 0.30, the corresponding strength for 7 and 28 days are 4.5 MPa and 5.50 MPa respectively. Lower the ratio of alkaline activator to flyash by mass results in higher split tensile strength. There is no drastic increase in the strength as the curing period increases after seven to twenty-eight days.

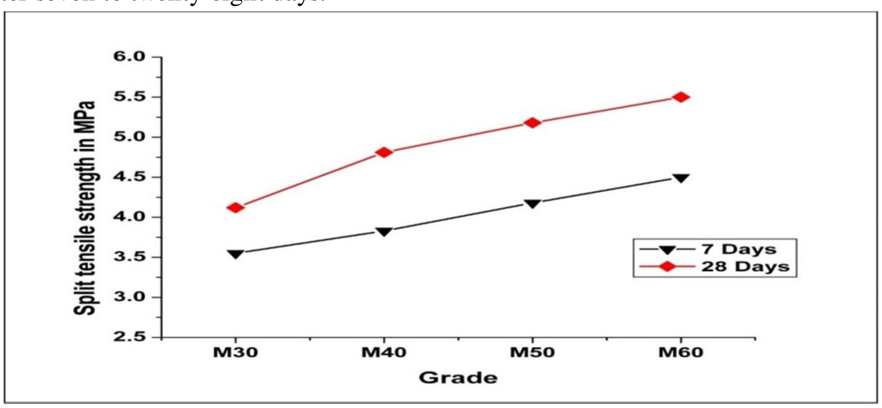


Fig. 6 Split Tensile Strength v/s Grade for Ratio of Na_2SiO_3: NaOH=2.5 and Molarity 12M

The flexural strength of concrete observed minimum for mix M30 with ratio of alkaline activator to flyash by mass 0.45 with strength of 9.33 MPa at 7 days and 9.81 MPa at 28 days curing and maximum strength for ratio of alkaline activator to flyash by mass of 0.30 with strength of 11.49 MPa at 7 days and 12.40 MPa at 28 days curing. There is no drastic increase in the strength as the curing period increases after 7 to 28 days. Lower the ratio of alkaline activator to flyash by mass results in higher flexural strength. As the alkaline activator to fly ash ratio decreases for any grade of GPC, flexural strength increases.

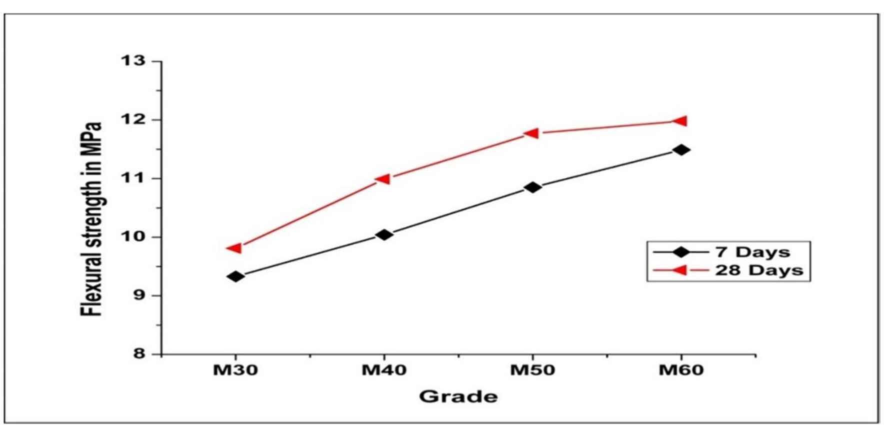
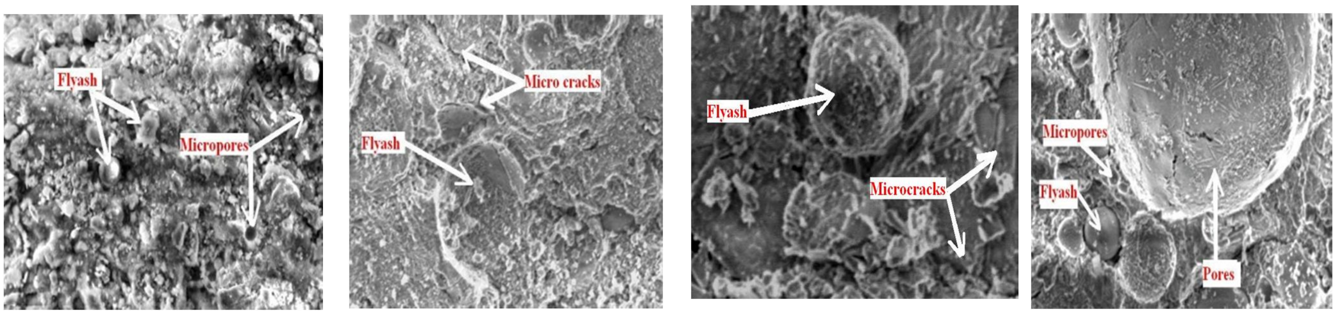


Fig. 7 Flexural Strength v/s Grade for Ratio of Na_2SiO_3: NaOH=2.5 and Molarity 12M

SEM test results of the geopolymer concrete mix of M30 and M40 synthesized with an alkaline activator to fly ash ratio of 0.45 and 0.40 showed the porous, heterogeneous matrix with a high content of unreacted fly ash microspheres, which gives low compressive strength. The concrete mix of M50 and M60 synthesized with alkaline activator to fly ash ratio of 0.35 and 0.30 had the formation of continuous, less porous, homogenous matrices that had microcracks showed maximum compressive strength 59.30 MPa and 68.02 MPa for 28 days curing. The lower dissolution rates associated with samples synthesized with alkaline activator to fly ash ratios of 0.35 and 0.30 resulted in high geopolymerization rates, more homogeneous matrices, and the higher compressive strength.



M30 M40 M50 M60

Fig. 8 Microstructure of Differernt Mixes of Flyash Based Geopolymer Concrete

V. CONCLUSIONS

Based on the obtained results of investigations the following conclusions can be drawn:

1) The flyash can be used as geopolymeric binder which can bind the fine and coarse aggregates to form geopolymer concrete so that it is considered as eco-friendly materials.
2) The slump of geopolymer concrete for given water content, decreases with an increase in fly ash content and increases with an increase in the ratio of alkaline activator to flyash by mass.
3) Without any sign of setting, fresh fly ash-based geopolymer concrete can be easily handled up to 110 to 130 minutes.
4) Increase in the grade of the concrete, the mix becomes stiffer resulting lower workability and reduction in strength.
5) As the ratio of water-to-geopolymer solids by mass increases, the strength of fly ash-based geopolymer concrete decreases.
6) Higher the mechanical properties of fly ash-based geopolymer concrete if less the ratio of alkaline activator to flyash by mass.
7) The failure modes of GPC samples are similar to that of ordinary Portland concrete samples.
8) Higher alkaline activator to fly ash ratio showed the porous, heterogeneous matrix with a high content of unreacted fly ash microspheres, which gives low compressive strength.
9) The lower dissolution rates associated with samples synthesized with alkaline activator to fly ash ratios resulted in high geopolymerization rates, less porous, more homogeneous matrices, and the higher compressive strength.

VI. SCOPE FOR THE FUTURE WORK

1) The present experimental work can be extended to Higher and ultra-high strength of Concrete.
2) Strength and durability properties of flyash based concrete by adding non-metallic and organic fibres can be studied.
3) This work can be extended by adding some other additives to the Fly Ash based concrete.
4) The study on use of flyash based geopolymer in pre-stressed concrete.
5) X-Ray diffraction and FTIR spectra analysis is need to be study on geopolymer concrete exposed to different sever environment.

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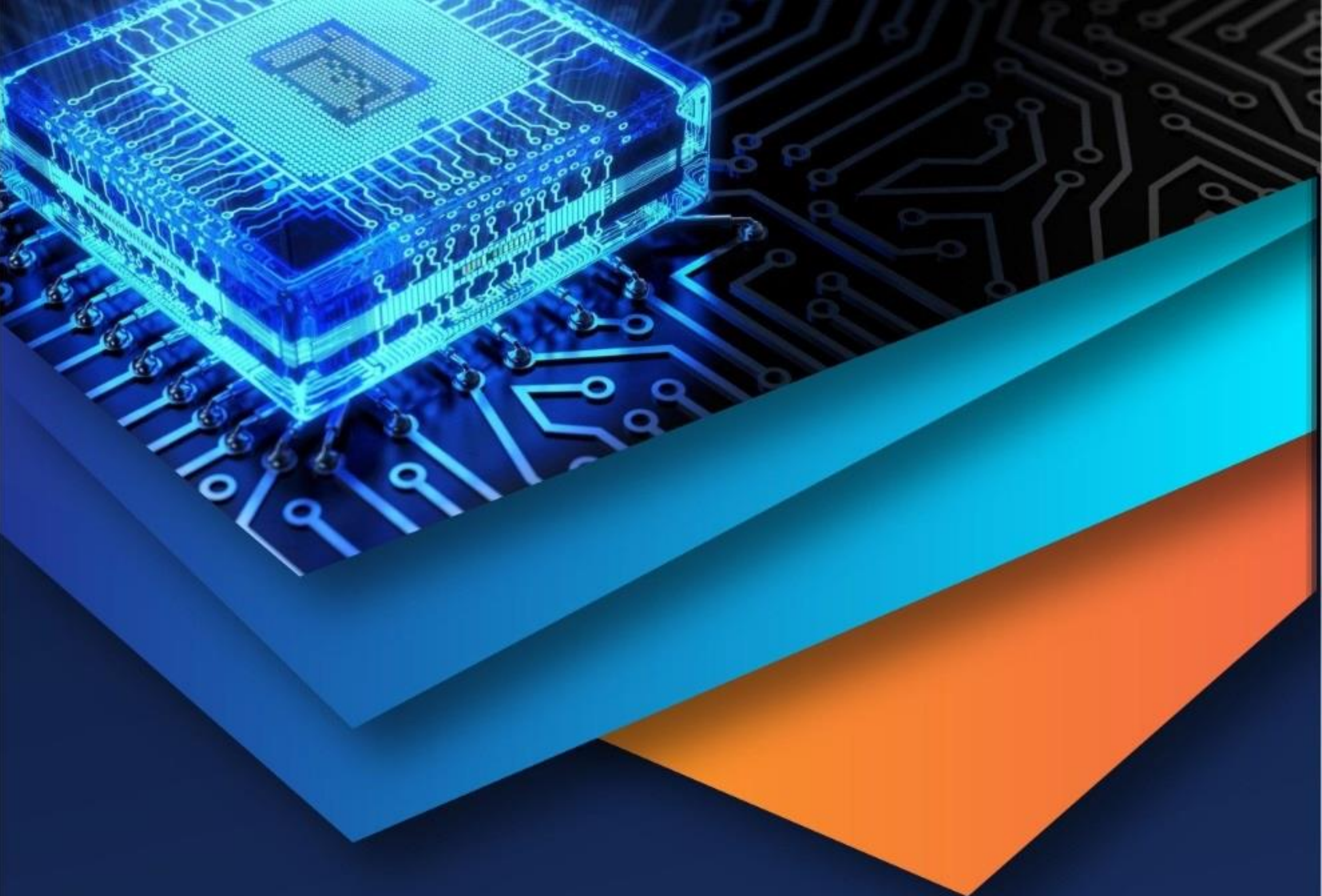

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