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Properties of Geopolymer Paste and Mortar

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ABSTRACT: Geopolymers are the new generation green material that has a great potential of replacing the conventional cementitious materials. The fresh and hardened properties of sodium hydroxide activated binary blends of slag and fly ash based geopolymer paste and mortars are reported in this paper. Experimental outcomes on fresh and hardened properties such as normal consistency, flow value, setting time, drying shrinkage, soundness, and compressive strengths of the geopolymer binders are presented. Additionally, the chemical products, bonding and microstructural changes occurring during the setting and hardening course are examined. The experimental outcomes showed that the physical and mechanical properties of the binders are very much akin to that of conventional cement and the same is significantly influenced by the chemical composition of the source materials, concentration of the activator and the processing environment. The consistency and setting times of geopolymers are found to be within the ranges that are prescribed for ordinary Portland cement. Highest compressive strength of around 44 MPa is obtained for slag based geopolymer mortar that is activated using 8 M sodium hydroxide solution. Fly ash and slag geopolymers exhibited excellent stability against expansion and shrinkage. Raw materials are optimized by design of experiment and the fitted model shows a good relation with the experimental data.

KEYWORDS: Geopolymer, Fly ash, Slag, Physical and mechanical properties, Optimization technique.

I. INTRODUCTION

The geopolymerization reaction which occurs depends on the nature of the source materials, activator type and the processing conditions. Aluminium and silicon oxygen tetrahedrons form a three-dimensional polymeric structure where alkalization acts as a charge balancing role for aluminium. Geopolymers exhibit high compressive strength, low shrinkage, fast/ slow setting time with excellent acid and fire resistance. The manufacturing process requires less energy and emits about 80 to 90% less greenhouse gas. Considering the economic and environmental aspects, the manufacturing process is beneficial. The fresh and hardened properties of cementitious materials are important to use them in construction dealings. The comprehensive properties of cement like construction consistency and workability are imperative as ease for with greater homogeneity. The setting properties of the cementitious binder are also equally critical as those controls the transportation time, placement, finishing and other such operations. Also, the mechanical properties such as compressive strength are an important parameter. The composition of raw material, placing and curing condition greatly influences these properties.

Large amount of CO₂ is emitted during the conventional cement production processes, which are energy intensive. Geopolymers are an alternative to the traditional cementitious binders which are manufactured by activating the industrial by-products rich in aluminosilicates which include slag, fly ash, red mud, etc. with alkaline solution. Geopolymerization reaction occurs when a suitable alkali activator solution is used to activate aluminosilicate source materials. Huge amounts of by-products are generated from industrial processes which affect the environment adversely. Production process of conventional cement is energy demanding and it also emits enormous amounts of greenhouse gases. Geopolymers are the new generation green material that has a great potential of replacing

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the conventional cementitious materials. The fresh and hardened properties of sodium hydroxide activated binary blends of slag and fly ash based geopolymer paste and mortars are reported in this paper.

The mineralogical, chemical and microstructural analysis was also carried out to substantiate the physical and mechanical behavior of fly ash–slag geopolymer pastes and mortars. Section II describes about available literatures on geopolymer cement are summarized and discussed. Granulated blast furnace slag (GBS) and class-F fly ash (FA) are used for the preparation of the geopolymer (GP) as the mineral precursor that has materials in Section III. Section IV presents experimental results showing results of images tested. Finally, Section V presents conclusion.

II. LITERATURE REVIEW

Puertas et al. (2000) concluded that the activator concentration and the slag content showed significant impact on strength development, whereas the effect of curing temperature is minimal while assessing the mechanical behavior of sodium hydroxide activated fly ash/slag paste. Temuujin et al. (2009a, b) assessed the effect of mechanical activation of fly ash on compressive strength of geopolymer. The milled fly ash resulted in the change in particle size, morphology and an increased reactivity. Songpiriyakij et al. (2010) made fly ash geopolymer using rice husk and bark ash. The reactivity of source material and the quality of matrix formed contributed to the enhanced strength. Nath and Sarker (2014) reported the setting, workability and strength properties of fly ash geopolymer. Topark-Ngarm et al. (2014) opined that the presence of high calcium content in fly ash resulted in producing high strength geopolymer concrete with setting time of 28–58 min.

Cihangir et al. (2015) studied the effect of activator type, concentration and slag composition on strength and stability of alkali-activated slag. In short-term curing, sodium hydroxide resulted in higher unconfined compressive strength while sodium silicate resulted in higher unconfined compressive strength during long-term curing. Junaid et al. (2015) considered various parameters for the development of fly ash geopolymer and stated that the alkali concentration, composition of sodium silicate, silicate to hydroxide ratio, temperature and curing time were the influencing parameters. Rao and Rao (2015) studied the setting time and compressive strength of fly ash and slag based geopolymers activated with alkali solution (combination of sodium hydroxide and sodium silicate). Increased alkali concentration resulted an increased setting time while slag addition decreased the setting time. In this paper, fresh and hardened properties of geopolymer paste and mortar prepared from the fly ash–slag blends activated with sodium hydroxide solution under ambient condition were emphasized.

III. MATERIALS AND METHODOLOGY

Granulated blast furnace slag (GBS) and class-F fly ash (FA) are used for the preparation of the geopolymer (GP) as the mineral precursor. Standard sand conforming IS: 650 (1991) is used for preparing geopolymer mortar specimen. Slag and the fly ash are collected from slag granulation and captive thermal power plants of the Rourkela Steel Plant (RSP), Odisha, India respectively. These materials are further oven dried for 24 h at a temperature of 105 °C to 110 °C prior to use. Pulverizing GBS in a ball mill obtained Ground granulated blast furnace slag (GGBS). The Bruner–Emmett–Teller (BET) specific surface values for FA and GGBS are found to be 28.07 and 22.73 m²/g respectively. These raw materials are dried under sun and thoroughly mixed to achieve homogeneity of the sample. Sodium hydroxide (Noah) flakes with 98% purity are used as an alkali activator, procured from Loba Chemie Pvt. Ltd., Mumbai, India. Different concentrations of Noah solutions are prepared a day before its use by dissolving Noah flakes in distilled water accordingly. X-ray fluorescence (XRF) spectroscopy was used to analyse the oxide composition of raw materials and are summarized in Table 1. Silicon dioxide, aluminum oxide, calcium oxide, and magnesium oxide are among the major constituents found. However, fly ash consist oxides of silicon, aluminum and iron.

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Table 1-Oxide composition of raw materials

Compounds	FA	GGBS	Compounds	FA	GGBS
MgO	0.542	9.700	Fe ₂ O ₃	5.450	0.60
Al ₂ O ₃	28.497	18.00	Na ₂ O	0.600	0.088
SiO ₂	55.706	29.31	MnO	0.430	0.14
K ₂ O	1.264	1.04	TiO ₂	0.611	1.04
P ₂ O ₅	0.605	–	SO ₃	0.903	1.40
CaO	2.143	37.2	Loss on ignition	2.549	0.81

IV. EXPERIMENTAL RESULTS

a) Normal Consistency

Normal consistency values of various GGBS-FA mixes are found to vary from 26 to 39% (Table 2). These conform the range recommended for OPC. An increase of GGBS fraction in the mix, results in a reduction of the normal consistency value. The specific surface of fly ash and GGBS are 28.07 and 22.73 m²/g respectively and both these materials are non-plastic in nature. Owing to greater specific surface area of fly ash, a mix containing higher fraction of fly ash, requires more amount of alkaline solution to lubricate the particle surface and thus it exhibits greater consistency value. The consistency values of the mixes are increased with an increase of the concentration of the alkali solution. The viscosity of the NaOH solutions depends on its concentration. A highly viscous solution makes the geopolymer paste plastic in nature, which in turn during testing limits the sinking of plunger attached to the Vicat apparatus into the paste. Therefore, the consistency value increases. This effect is more clearly visible in fly ash rich mixtures compared to slag rich mixes. Thus, it can be concluded that the normal consistency values of binary blended FA-GGBS geopolymer mixes are the function of chemical composition, fineness of source materials and the concentration of the alkaline activator solution.

Table 2- Normal consistency values (%) of FA-GGBS geopolymers

FA-GGBS mixes	NaOH concentration (M)				
	2	4	8	12	16
S0	30	30	31	33	39
S20	29	29	30	32	38
S40	28	29	30	31	36
S60	27	28	29	31	34
S80	26	28	29	30	32

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FA-GGBS mixes	NaOH concentration (M)				
	2	4	8	12	16
S100	26	28	29	30	31

b) Setting time

The initial setting (IS) and final setting (FS) of FA-GGBS mixes are presented in minutes in fig 1. The IS times of the mixes are found to vary from 25 to 300 min whereas the FS times vary between 40 and 490 min except the mix S0. The S0 (100% fly ash) sample displayed an IS time of 12 h and the FS time exceeds 24 h. This is owing to sluggish chemical reaction of fly ash with time under an ambient room temperature.

Hydration Products, Chemical Bonds and Microstructure: The aluminosilicate phase of slag and fly ash undergoes polymerization process when activated by the alkali solution. GGBS is a rich source of aluminosilicates and also calcium compounds. It undergoes hydration reaction besides geopolymerization process. The reaction compounds formed by the activation of fly ash and slag with 8 M NaOH solution at different periods are examined primarily by XRD analysis. The XRD patterns of the raw materials are different as the slag is purely a glassy material whereas fly ash encompasses peaks of quartz, hematite, mullite and corundum fig 2. The main characteristic bands of both raw materials and geopolymers after setting are shown in fig 3. The SEM micrographs with EDX map of samples at their IS and FS times fig 4 indicate the formation of some new structures.

c) Soundness

The occurrence of free lime and magnesia makes the geopolymer binder unsound as they may cause expansion in the hardened binder. The soundness values of geopolymer specimens synthesized by activating fly ash and slag with different concentration of NaOH solutions are assessed. The FA geopolymer exhibited zero expansion value whereas; an increased expansion of about 1 mm is observed in case of GGBS geopolymer. Commonly, unsoundness of ordinary Portland cement is caused owing to the existence of greater proportion of free lime, magnesia and calcium sulphate. XRF analysis of slag indicates that it contains about 10% of magnesium oxide.

d) Drying Shrinkage

The drying shrinkage behaviour of slag and fly ash mortar specimens, prepared using NaOH solutions of varied concentrations, are determined by performing shrinkage tests after curing periods of 28 days. The experimental findings are presented in Table 3. It is noticed that slag specimens exhibited greater shrinkage values than specimens prepared from fly ash. This is attributed to the decrease in hydral volume during the hydration reaction of slag at later stage of curing. Additionally, the concentrations of alkali solution are found to influence the shrinkage values of specimens. A higher concentration solution reduces the shrinkage value. This is owing to the loss of higher amount of capillary water that is available in the samples prepared at low molarity of NaOH solution.

Table 3- Drying shrinkage and flow diameter of FA and GGBS geopolymer

NaOH concentration (M)	Drying shrinkage (micro-strain)		Flow diameter (mm)	
	S0	S100	S0	S100
2	500	1050	150	137
4	440	900	144	124

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NaOH concentration (M)	Drying shrinkage (micro-strain)		Flow diameter (mm)	
	S0	S100	S0	S100
8	400	800	130	116
12	360	770	124	104
16	320	750	112	101

e) Constituency-flow

The workability of the FA and GGBS based geopolymer mortars; that are activated by NaOH solutions of various concentrations; are studied by measuring the flow value of the binder. The flow table test is adopted as water comes out from the viscous geopolymer mortar during geopolymerization process shown in fig 5. The flow value of the geopolymer mortars are determined by measuring the flow diameter of the mortar. Fly ash geopolymer mortar displayed higher flow value than slag based geopolymer irrespective of the concentration of the activating solution. This is related to arrangement of the particles. More spherical particles of fly ash encourage the free flow of the mortar while the angular slag particles hinder the flow due to higher interlocking between particles.

f) Optimization

The optimization of GGBS content (A) and NaOH concentration (B) giving the maximum compressive is done by RSM. The experimental values of response for the input parameters are assembled and built-in with the non-linear cubic polynomial model as it showed a good coefficient of determination with a lower standard error. The scattering of predicted responses with the observed values of compressive strength for 28 days cured specimens.

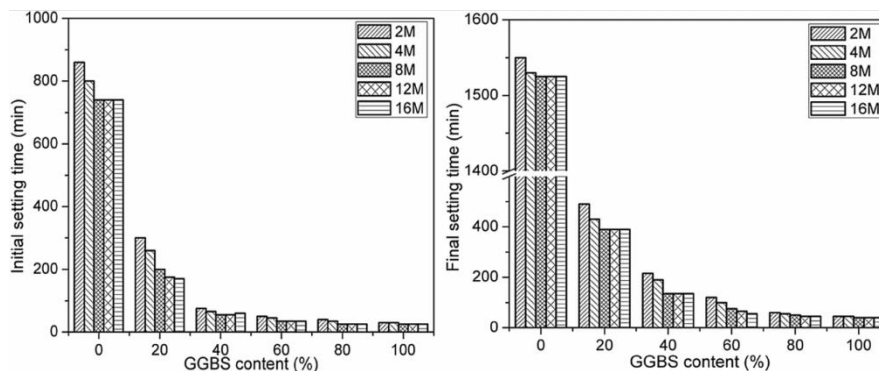


fig1: The initial setting (IS) and final setting (FS) of FA-GGBS mixes

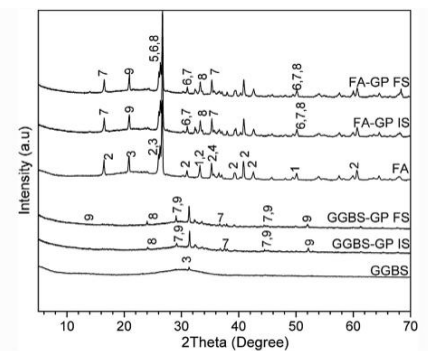


fig 2 : The XRD patterns of the raw materials

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Vol. 9, Issue 3, March 2020

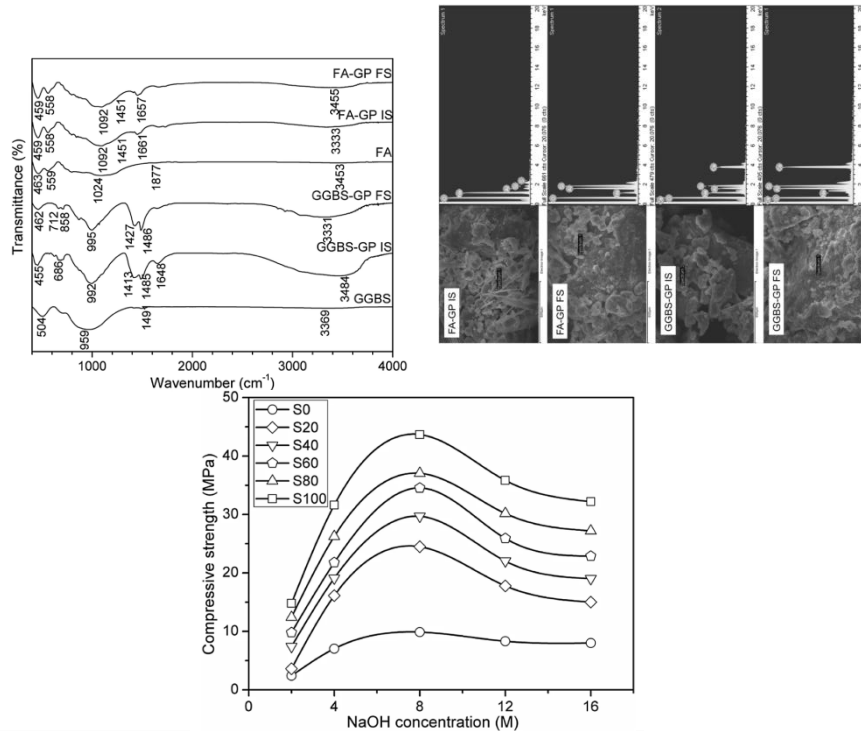


fig3: characteristic bands of both fig 4: formation of some new structures fig 5:flow value of the geopolymer mortars Raw materials and geopolymers

V. CONCLUSION

The experimental outcomes showed that the physical and mechanical properties of the binders are very much akin to that of conventional cement and the same is significantly influenced by the chemical composition of the source materials, concentration of the activator and the processing environment. The consistency and setting times of geopolymers are found to be within the ranges that are prescribed for ordinary Portland cement. Raw materials are optimized by design of experiment and the fitted model shows a good relation with the experimental data. Fly ash when replaced by a small amount of slag is studied under an ambient condition, where a mixture of sodium silicate and sodium hydroxide is used to activate the material. The workability of geopolymer is not representative of cement due to the high viscosity of the alkaline solution. Increase in the slag content resulted in the reduced workability and setting time and an increased compressive strength. The source material proportion and the activator concentration were the parameters considered in this study. Experimental outcomes on fresh and hardened properties such as normal consistency, flow value, setting time, drying shrinkage, soundness, and compressive strengths of the geopolymer binders are presented. Additionally, the chemical products, bonding and microstructural changes occurring during the setting and hardening course are examined.

Consistency, flow, setting, shrinkage, soundness, and compressive strength values of slag-fly ash blended geopolymers are investigated. The chemical composition and microstructure changes associated to physical and mechanical properties are correlated. Based on experimental outcomes, the concentration of alkali and slag content are the influential parameters governing the physico-mechanical property of FA-GGBS blended binary geopolymer. The consistency, setting, soundness and strength properties are greatly influenced by these parameters. Finally, it is

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Vol. 9, Issue 3, March 2020

concluded that the use of industrial waste products such as slag and fly ash could be utilized for production of a sustainable, eco-friendly cementing material comparable to conventional cementitious materials.

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