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GEOPOLYMER MORTAR INCORPORATING HIGH **CALCIUM FLY ASH AND SILICA FUME**

V.C.PRABHA¹, V.REVATHI²

An attempt was made in the present work to study the compressive strength and microstructure of geopolymer containing high calcium fly ash (HCFA) and silica fume. Concentration of sodium hydroxide solution 8M, 10M, 12M & 14M, liquid to binder ratio 0.5 and sodium hydroxide to sodium silicate ratio 2.5 were selected for the mixes. Geopolymer mortar test results indicated that the mix with 40% silica fume by the weight of HCFA yielded higher compressive strength under ambient curing. The XRD pattern typically shows the major portion of amorphous phase of geopolymer. The existence of C-A-S-H gel, N-A-S-H gel and hydroxysodalite gel products were observed through SEM which developed dense microstructure and thus enhanced strength of HCFA and silica fume geopolymer.

Keywords: geopolymer mortar, compressive strength, ambient curing, High calcium fly ash, Silica fume

1. INTRODUCTION

The manufacture of cement causes emission of carbon-di-oxide into the atmosphere which results in environmental pollution. The increasing demands on the strength and durability characteristics of cement concrete prompts to add additives or admixtures or both for specific purposes. Geopolymer, an alternative binder produced from strong alumina silicate reactive materials and highly concentrated aqueous alkali hydroxide or silicate solution has potential to lower the significant carbon footprint of cement concrete [1-3].

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Materials like fly ash, GGBS, bottom ash, silica fume, metakaolin, rice husk ash etc. could be used as alumina silicate reactive materials [4-7]. Geopolymer made of using class F fly ash and GGBS are relatively common and acknowledged in many places [8, 9]. The most commonly used alkaline activators in geopolymer concrete are combinations of NaOH and Na₂SiO₃ solutions. It has been established that NaOH possesses greater capacity to liberate silicate and aluminate monomers [10]. Usually, this alkaline activator is prepared by mixing water, NaOH pellets and Na₂SiO₃ solution. Further different alkalis mixtures or other alkali metal systems are being used to obtain required strength and durability. It is important that the solution strictly must be in concentration form to avoid the crystallization of zeolite as the end product rather than an amorphous geopolymer [11, 12]. The compressive strength of geopolymer depends on the type of alumina silicate material and its particle fineness. The fine particles make higher discharge of silica and alumina in the alkali environment and leads to a higher strength geopolymer [13].

The compressive strength is higher for oven drying as compared to the specimens left in ambient curing [14, 15]. Ambient cured specimens gained their strength without elevated heating mainly because of the presence of high calcium content which has the ability to harden at ambient temperature. It is observed that the strength of the geopolymeric gel, interfacial bonding between the geopolymeric gel and aggregate determine the compressive strength [16]. Previous study also pointed out that crystalline C-S-H phase is formed at higher calcium concentrations, augmenting the strength. As a result of reactions with strong alkaline activators, the co-existence of geopolymeric gel and calcium silicate hydrate (C-S-H) gel resulted in HCFA geopolymer system (Xiaolu Guo et al). However, the strength enhancement of HCFA is not much higher compared to low calcium fly ash in geopolymeric reactions. The geoplymeric reaction of several alumina silicate materials does not participate in same amount of reaction for the same condition. For instance, two types of fly ash perform different reactions. The HCFA geopolymers attain higher strength at early age periods when it is steam cured [17-19]. The geopolymer prepared from silica fume and GGBS is reported to develop improved strength under ambient curing [20]. The inclusion of silica fume into low calcium fly ash geopolymer optimizes the microstructure and yielded higher strength [21-24]. Silica fume significantly reduced the porosity and permeability [25-27].

Besides, there is no study made on high calcium fly ash with the addition of silica fume in geopolymer. This present study aims to look into the compressive strength and micro structural study on addition of silica fume in HCFA geopolymer.



2. MATERIALS & EXPERIMENTAL PROGRAM

2.1. MATERIALS

Fly ash was collected from Neyveli Lignite Corporation in Cuddalore district of Tamilnadu. The HCFA was ground to become finer by ball mill for 8 hrs. The specific gravity of HCFA was found to be 2.25. The silica fume used in this study was purchased from Coimbatore. The specific gravity of silica fume used in the study was observed as 2.34. The river sand with specific gravity of 2.6 and fineness modulus of 2.1 was also used. Alkaline activators chosen were sodium silicate (Na₂SiO₃) and sodium hydroxide (NaOH). The sodium silicate solution and sodium hydroxide in flakes were procured from Mercury Sakthi enterprises company, Erode. To make the sodium hydroxide solution, flakes (97-98% purity) were dissolved in tap water. The chemical properties of fly ash and silica fume are presented in Table 2.1.

S.No	Chemical composition	Fly Ash	Silica Fume
1.	Silica (SiO ₂)	63.11%	93.67%
2.	Calcium Oxide (CaO)	17.13%	0.31%
3.	Aluminium Oxide (Al ₂ O ₃)	19.58%	0.83%
4.	Magnesium Oxide (MgO)	0.24%	0.84%
5.	Iron Oxide (Fe ₂ O3)	5.03%	1.30%
6.	Sodium Oxide (Na ₂ O)	0.29%	0.40%
7.	Potassium Oxide (K ₂ O)	0.84%	1.10%
8.	Loss ofIgnition (L.O.I)	1.55 %	2.10%

Table 2.1 Chemical Composition of Fly Ash & Silica Fume

2.2. MIX PROPORTION

The present study has High Calcium Fly Ash (HCFA) and silica fume as the source material for the geopolymer mortar. In this experiment, it is proposed to mix HCFA with silica fume contents 0%, 20%, 40%, 60% and 80% by total weight of the dry fly ash. Twenty numbers of mixes were studied. All geopolymer mortars were made with sand to fly ash ratio of 1:3. Liquid to binder ratio was 0.5. Sodium silicate to sodium hydroxide ratio was kept as 2.5. The molarity of NaOH varied from 8 M to 14M at an interval of 2M. The following mix identity is used for the geopolymer mortar of



various mix propositions. 'F' represents HCFA, 'S' identifies Silica Fume, and the suffix value of the numerical indicates the percentage of silica fume in the total weight of source material. The beginning numerical represents molar concentration. For example in '8FS₀' starting letter '8' indicates 8M concentration of NaOH. Various mixes combinations of HCFA geopolymer mortar used in the present study are given in Table 2.2.

Sl. No	Mix	High Calcium Fly Ash	Replacement % of Silica Fume	Silica Fume	Fine Aggregate	Sodium silicate	Sodium Hydroxide solution	
							mol/L	(kg/m^3)
1.	$8FS_0$	568.3	0%	-	1704.03	189.45	8	91.22
2.	8FS ₂₀	454.6	20%	113.7	1704.03	189.45	8	91.22
3.	8FS40	340.9	40%	227.3	1704.03	189.45	8	91.22
4.	8FS ₆₀	227.3	60%	340.8	1704.03	189.45	8	91.22
5.	8FS ₈₀	113.6	80%	454.5	1704.03	189.45	8	91.22
6.	10F S ₀	568.3	0%	-	1704.03	189.45	10	91.22
7.	10FS ₂₀	454.6	20%	113.7	1704.03	189.45	10	91.22
8.	10FS ₄₀	340.9	40%	227.3	1704.03	189.45	10	91.22
9.	10FS ₆₀	227.3	60%	340.8	1704.03	189.45	10	91.22
10.	$10FS_{80}$	113.6	80%	454.5	1704.03	189.45	10	91.22
11.	12F S ₀	568.3	0%	-	1704.03	189.45	12	91.22
12.	12FS ₂₀	454.6	20%	113.7	1704.03	189.45	12	91.22
13.	12FS ₄₀	340.9	40%	227.3	1704.03	189.45	12	91.22
14.	12FS ₆₀	227.3	60%	340.8	1704.03	189.45	12	91.22
15.	12FS ₈₀	113.6	80%	454.5	1704.03	189.45	12	91.22
16.	$14FS_0$	568.3	0%	-	1704.03	189.45	14	91.22
17.	$14FS_{20}$	454.6	20%	113.7	1704.03	189.45	14	91.22
18.	$14FS_{40}$	340.9	40%	227.3	1704.03	189.45	14	91.22
19.	14FS ₆₀	227.3	60%	340.8	1704.03	189.45	14	91.22
20.	14FS ₈₀	113.6	80%	454.5	1704.03	189.45	14	91.22

Table 2.2. Mix Proportions of High Calcium Geopolymer mortar with Silica Fume (Quantity of materials in kg/m³)

2.3 PREPARATION AND CURING

Fly ash with silica fume and river sand were mixed thoroughly and dry materials were added slowly in the activating solution prepared by dissolving sodium hydroxide pellets in water and mixing with sodium silicate solution. The mixing was continued until a homogeneous mix was obtained. Several mixes were prepared using fly ash, silica fume and river sand in each experimental series. The geopolymer mortar sample was cast in moulds which were compacted by vibrator machine to remove the entrapped air. After the specimens were cast properly, it was wrapped by plastic sheet



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and left out in ambient temperature for 24 hours and next day it was demoulded. The demoulded specimens were kept in ambient curing until the day of testing.

2.4 EXPERIMENTAL TESTING

The mortar specimen's compressive strength was measured by using 70.6 x 70.6 x 70.6 mm size cube specimens. The compressive strength was determined at the age of 3, 7, and 28 days. Also, the micro structural studies on geopolymer mortar samples which yielded significant compressive strength was examined using X-Ray Diffraction (XRD) and Scanning ElectronMicroscope-Energy Dispersive X-ray Analysis (SEM-EDXA).

3. RESULTS & DISCUSSION

3.1. COMPRESSIVE STRENGTH OF HCFA GEOPOLYMER MORTAR WITH SILICA FUME **AS AN ADDITIVE**

The compressive strength results of HCFA geopolymer mortar mixes with different percentage of silica fumeare presented in Table 3.1. From the experimental results, the compressive strength of 8FS geopolymer mortar ranged from 24.18MPa (8FS₀) to 38.62MPa (8FS₄₀) at the age of 28 days. The mix 8FS₄₀has attained higher strength than other mixes made with 8M NaOH concentration. The mix 8FS₄₀ achieved a maximum compressive strength of 38.62 MPa at 28 days. It is shown that 40% silica fume mix (8FS₄₀) imparted 38.7%, 52.8% and 59.7% higher the compressive strength than 8FS₀ at all the age of testing.

The trend observed in compressive strength results of 10FSgeopolymer mortar is almost similar to that of 8FSgeopolymer mortar mixes. However, the compressive strength of 10FSgeopolymer mortar is comparatively higher than the mixes made with 8FSgeopolymer mortar. The compressive strength of 10FSgeopolymer mortar was ranging from 29.87MPa (10FS₀) to 41.07MPa (10FS₄₀) at the age of 28 days. Also, it can be seen that 10FS₄₀produced maximum compressive strength of 41.07 MPa at age of 28 days. It can be noted that the compressive strength of 40% silica fume mix (10FS₄₀)has gained 37.6%, 32% and 37.1% higher than the reference mix $10FS_0$ at the age of 3, 7 and 28 days.

The compressive strength of 12FS geopolymer mortar mixes varies from 29.52MPa $(12FS_0)$ to 44.13MPa (12FS₄₀). It is evident that the range of compressive strength is comparatively higher than the mixes made with sodium hydroxide concentration of 8M and 10M. The mix 12FS₄₀ exhibited the highest compressive strength of 44.13 MPa at 28 days. It is clear that the compressive strength of 40% silica fume mix (10FS₄₀) has 41%, 16.5% and 21.5% improvement over reference mix 12FS₀at the age of 3, 7 and 28 days.

With respect to 14FSgeopolymer mortar mixes, the compressive strength is observed from 36.19 MPa ($14FS_0$) to 47.79 MPa ($14FS_{40}$). It is also evident that compressive strength of 14FSgeopolymer mortar is relatively higher than 8, 10, and 12M sodium hydroxide concentration mixes. The mix 14FS₄₀ achieved significantly higher compressive strength of 47.79 MPa at the age of 28 days. Further, 14FS₄₀ endorsed 40.7%, 27.7% and 20.5% more compressive strength at the age of 3, 7 and 28 days than the reference mix 14FS₀.

The increase in compressive strength of geopolymer incorporating HCFA and silica fume, on the one hand, may be related to denser microstructure systems. [28]Because of the packing effect of the fine silica fume, particles behave as micro-aggregate filler which disperses in geopolymer and fills the inner space inside the microstructure of geopolymer paste [29]. It is also evident that the higher NaOH concentration resulted higher compressive strength.

Molarity	Mix	Replacement	Compressive Strength (MPa)								
of NaOH	ID	% of Silica Fume		3 days		7 days			28 days		
nuon		i unic	Mean	STDEV	COV	Mean	STDEV	COV	Mean	STDEV	COV
	8FS ₀	0%	11.90	0.04	0.34	19.34	0.05	0.26	24.18	0.21	0.87
	8FS ₂₀	20%	15.63	0.62	3.97	26.14	0.84	3.21	33.78	0.54	1.60
8	8FS40	40%	16.50	0.04	0.24	29.56	0.05	0.17	38.62	0.31	0.80
	8FS ₆₀	60%	13.92	0.19	1.36	24.53	0.27	1.10	35.24	0.97	2.75
	8FS ₈₀	80%	11.05	0.17	1.54	18.17	0.41	2.26	27.32	0.77	2.82
	10F S ₀	0%	14.18	0.08	0.56	23.04	0.15	0.65	29.95	0.29	0.97
10	10FS ₂₀	20%	17.08	0.06	0.35	28.95	0.73	2.52	39.02	0.65	1.67
10	10FS ₄₀	40%	19.51	0.05	0.26	30.41	0.02	0.07	41.07	0.58	1.41
	10FS ₆₀	60%	16.93	0.65	3.84	25.87	0.37	1.43	37.59	0.82	2.18
	$10FS_{80}$	80%	13.52	0.1	0.74	21.93	0.01	0.05	29.87	0.71	2.38
10	12F S ₀	0%	15.35	0.31	2.02	27.8	0.36	1.29	36.31	0.62	1.71
	12FS ₂₀	20%	17.94	0.57	3.18	29.91	0.72	2.41	38.12	0.14	0.37
12	12FS ₄₀	40%	21.65	0.75	3.46	32.38	0.18	0.56	44.13	0.79	1.79
	12FS ₆₀	60%	16.93	0.16	0.95	26.75	0.58	2.17	37.72	0.22	0.58
	$12FS_{80}$	80%	14.18	0.09	0.63	23.86	0.54	2.26	29.52	0.51	1.73
14	14F S ₀	0%	16.56	0.24	1.45	27.87	0.77	2.76	39.65	0.84	2.12
	14FS ₂₀	20%	18.74	0.02	0.11	29.15	0.62	2.13	40.97	0.41	1.00
	$14FS_{40}$	40%	23.30	0.12	0.52	35.59	0.11	0.31	47.79	0.01	0.02
	$14FS_{60}$	60%	20.63	0.14	0.68	32.98	0.95	2.88	41.63	0.07	0.17
	$14FS_{80}$	80%	17.18	0.35	2.04	26.74	0.65	2.43	36.19	0.63	1.74

Table 3.1. Compressive Strength of HCFA Geopolymer Mortar with Silica Fume as an Additive

Note: STDEV= Standard Deviation, COV = Coefficient of Variance [%]

3.1.1 THE MATHEMATICAL REGRESSION MODEL

Based on experimental results, mathematical models for compressive strength and various percentage of silica fume is developed using polynomial regression equation.

COMPRESSIVE STRENGTH PREDICTION MODEL FOR VARIOUS SILICA FUME PERCENTAGES

The model consists of parameters Silica fume content percentage (denoted by s), Compressive strength of specimen in 8M concentration at 3, 7, and 28 days (denoted by 8fc₃, 8fc₇, and 8fc₂₈ respectively). Figure 3.1(a) shows the equations (gradients) of compressive strength for five mortar mixes (8FS0, $8FS_{20}$, $8FS_{40}$, $8FS_{60}$, $8FS_{80}$) at various silica fume replacements, and the corresponding coefficient of determination (R^2) or correlation coefficient (R). Correlation coefficient (R) for all the curves is closer to unity, indicating that the polynomial second order equation is found to fit well. The first factor considered was silica fume percentage. According to silica fume content in 8M of NaOH, the compressive strength results of 3, 7, and 28 days the regression equation was proposed in Eqn. (1.a),(1.b), and (1.c))

$8fc_3 = -29.69642857 s^2 + 22.04214286 s + 12.10628571; R^2 = 0.96$	(1.a)
$8fc_7 = -62.08928571 s^2 + 47.69642857 s + 19.37085714; R^2 = 0.97$	(1.b)
$8fc_{28} = -77.25 s^2 + 65.67 s + 24.1; R^2 = 0.99$	(1.c)

Figure 3.1(b) shows the equations of compressive strength for geopolymer mortar mixes (10FS₀, 10FS₂₀, 10FS₄₀, 10FS₆₀, 10FS₈₀) for various silica fume replacement percentage. At 10M concentration of NaOH, the compressive strength results for various percentage of silica fume was predicted by the following Eqn. (2.a), (2.b), and (2.c). Similarly for 12M and 14M concentration of NaOH the corresponding compressive strength results for 3,7,28 days was calculated by the regression Eqn.(3.a),(3.b), and (3.c) and Eqn.(4.a),(4.b), and (4.c) respectively. Fig 3.1(c) and Fig 3.1(d) represents the regression model predicted for the compressive strength for 12M and 14M concentration of NaOH mortar specimens.

$10fc_3 = -31.48214286s^2 + 24.45071429s + 14.01942857; R^2 = 0.95$	(2.a)
$10 f c_7 = -45.89285714 s^2 + 34.06428571 s + 23.42857143; \ R^2 \!=\! 0.93$	(2.b)
$10 fc_{28} = -69.83928571 s^2 + 55.07642857 s + 30.23085714; \ R^2 = 0.99$	(2.c)
$12 f c_3 \ = 519.53125 s^4 \ - \ 822.3958333 s^3 \ + \ 361.96875 s^2 \ - \ 30.70416667 s \ + \ 15.35; \ R^2 = 1$	(3.a)
$12 f c_7 = 502.6041667 s^4 - 779.375 s^3 + 331.3958333 s^2 - 28.575 s + 27.8; \ R^2 = 1$	(3.b)
$12 f c_{28} = 709.6354167 s^4 - 1197.8125 s^3 + 572.4895833 s^2 - 63.2125 s + 36.31; R^2 \! = \! 1$	(3.c)
$14 f c_3 = 418.229166 s^4 \text{ - } 702.0833333 s^3 + 333.8958333 s^2 \text{ - } 31.14166667 s + 16.56; \ R^2 = 1000 s^2 + 10000 s^2 + 1000 s^2 + 10$	(4.a)
$14 f c_7 \ = 511.1979167 s^4 \ - \ 909.4791667 s^3 \ + \ 467.0520833 s^2 \ - \ 54.720833 s \ + \ 27.87; \ R^2 = 1$	(4.b)
$14 f c_{28} = 838.0208333 s^4 \text{ - } 1390.625 s^3 + 668.4791667 s^2 \text{ - } 78.175 s + 39.65; \ R^2 = 1000 s^2 + 10000 s^2 + 10000 $	(4.c)



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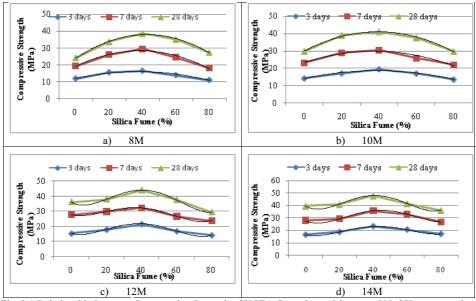


Fig. 3.1 Relationship between Compressive Strength of HCFA Geopolymer Mortar and NaOH concentration

COMPRESSIVE STRENGTH PREDICTION MODEL FOR VARIOUS SODIUM HYDROXIDE CONCENTRATIONS

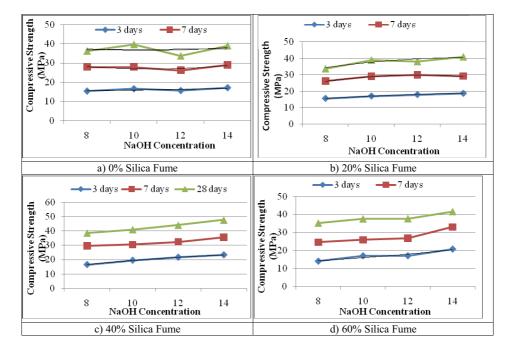
The mathematical model was also created by analysing the parameters molar concentration of NaOH (denoted by N) and compressive strength (fc) for various the percentage of silica fume. Where fc is the polynomial regression equation considering the effect of molar concentration of NaOH; the predicted results for 0% silica fume mortar mixes ($8FS_0$, $10FS_0$, $12FS_0$, $14FS_0$) are shown in Fig. 3.2(a). It was found that the value of correlation coefficient was near to 1, which means this mathematical model can predict the 3, 7, and 28 day compressive strength with very high accuracy due to the selection of appropriate equations. According to silica fume content, the compressive strength results for various molar concentration of NaOH is presented in Eq. (5.a), Eq. (5.b) and Eq. (5.c) for 3, 7, and 28 day compressive strength of geopolymer mortar for the specimens without silica fume content.

Fig 3.2(b) shows the compressive strength results for 20% silica fume content for mortar cubes, corresponding regression equation is denoted in Eq. (6.a), Eq. (6.b) and Eq. (6.c). Similarly for 40%, 60% and 80% silica fume contents mixes, the mathematical model is shown in Fig 3.2(c), Fig 3.2(d) and Fig 3.2(e). Corresponding regression equation for compressive strength is denoted in Eq. (7.a), Eq. (7.b) and Eq. (7.c), Eq. (8.a), Eq. (8.b) and Eq. (9.c).



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$fc_3 = -0.066875N^2 + 2.22875N - 1.5925; R^2 = 0.99$	(5.a)
$fc_7 = -0.226875N^2 + 6.50875N - 18.4975; R^2 = 0.97$	(5.b)
fc_{28} = -0.151875N ² + 5.97975N - 14.1185; R ² = 0-99	(5.c)
$fc_3 = -0.040625N^2 + 1.40325N + 7.0305; R^2 = 1$	(6.a)
$fc_7 = -0.223125N^2 + 5.40825N - 2.8395; R^2 = 1$	(6.b)
$fc_{28}{=}\;0.20604166666N^3 - 6.948749999N^2 + 77.42333332N - 246.38; R^2{=}\;1$	(6.c)
$fc_3 = -0.085N^2 + 2.997N - 2.017; R^2 = 1$	(7.a)
$fc_7 = 0.1475N^2 - 2.242N + 38.062; R^2 = 1$	(7.b)
$fc_{28} = 0.075625N^2 - 0.13525N + 34.8615; R^2 = 1$	(7.c)
$fc_3 = 0.13979166666N^3 - 4.569999999N^2 + 49.65583332N - 162.42; R^2 = 1$	(8.a)
$fc_7 = 0.12104166666N^3 - 3.688749999N^2 + 37.53333333N - 101.63; R^2 = 1$	(8.b)
$fc_{28} = 0.125N^3 - 4.027499999N^2 + 43.16999999N - 116.36; R^2 = 1$	(8.c)
$fc_3 = 0.08645833332N^3 - 2.819999999N^2 + 30.899166666N - 99.92999998; R^2 = 1$	(9.a)
$fc_7 = 0.057916666665N^3 - 1.96625N^2 + 23.14083333N - 70.769999999; R^2 = 1$	(9.b)
$fc_{28} = 0.20666666666N^3 - 6.562499999N^2 + 68.97333332N - 210.28; R^2 = 1$	(9.c)





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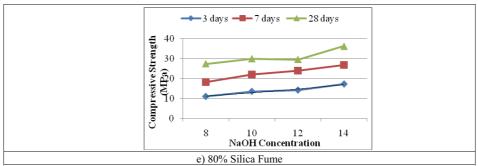


Fig. 3.2 Relationship between Compressive Strength of HCFA Geopolymer Mortar and Silica Fume

3.2. MICRO STRUCTURAL ANALYSIS

The higher compressive strength produced specimens were chosen to scan the microstructure and to understand the mechanism of geopolymeric reaction. Scanning Electron Microscope (SEM) with EDAX analysis and X-Ray Diffraction (XRD) were performed for geopolymer mortar powdered sample $14FS_0$ mix containing 100% HCFA, $14FS_{40}$ mix containing 40% silica fume and 60% HCFA collected at the age of 28 days. The samples were ground to fine powder for micro structural analysis.

3.2.1 XRD ANALYSIS

The XRD analysis results for the mortar specimens $14FS_0$ (100% HCFA), $14FS_{40}$ (40% silica fume, 60% HCFA) are shown in Fig. 3.3 and 3.4. In the present work, HCFA geopolymer mortar specimen ($14FS_0$) has the X- ray diffraction (XRD) as shown in Fig 3.3 with huge scattered peak ranging from 20-30° (20 max). It endorses the existence of quartz, calcite, and anatase in the matrix. Alkaline activators formed major amorphous phase in HCFA geopolymer.XRD pattern presented in Fig. 3.4 depicts the spectrum of geopolymer mortar prepared with 60% HCFA and 40% silica fume ($14FS_{40}$). The X- ray diffraction (XRD) spectra illustrates that it has huge scattered peak ranging from 20-30° (20 max). The major crystalline peak represents the compounds such as quartz, calcite, hematite, boetimite, and hornblende and anatase formation in the matrix. The additional compounds formations are higher in $14FS_{40}$ than reference $14FS_0$. The strength enhancement is evidenced due to the additional compounds formation. Further, the diffractogram indicates minor crystalline phase structure and major amorphous phase similar to $14FS_0$.



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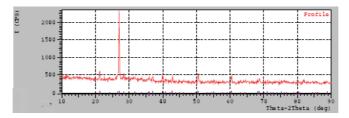


Fig.3.3 X-Ray Diffractogram of Fly ash Geopolymer Mortar 100% HCFA (14FS₀)

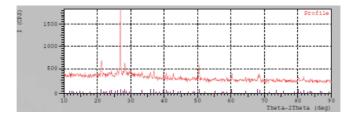


Fig.3.4 X-Ray Diffractogram of Geopolymer Mortar 60% HCFA, 40% silica fume (14FS₄₀)

3.2.2 SEM- EDAX ANALYSIS

The presence of C-A-S-H gel, N-A-S-H gel and hydroxysodalite gel products are identified in both $14FS_0$ and $14FS_{40}$ mix sample micrographs as shown in Fig 3.5 and 3.6. Further, the presence of voids is visible in $14FS_0$ mix sample. However, the incorporation of silica fume filled the voids and has shown much denser microstructure. The formation of additional gel products due to the incorporation of silica fume particles led to a compact microstructure and enhanced the strength[30]. Thus, it can be stated that the silica fume could increase the strength properties of geopolymer.

EDAX spectrum of $14FS_0$ and $14FS_{40}$ mix sample is given in Fig 3.5 and 3.6. The result confirms that elements such as silica, alumina, calcium, magnesium, sodium and iron are the dominating elements present in geopolymer mortar.



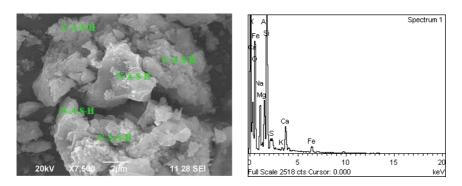


Fig.3.5. SEM-EDAX image of specimen 14FS₀, mortar cube 14M of NaOH, 100% HCFA

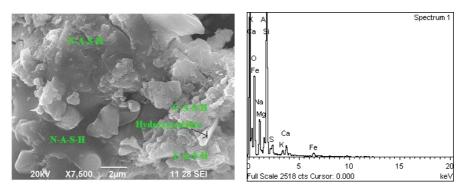


Fig.3.6. SEM-EDAX image of specimen 14FS₄₀, mortar cube 14M of NaOH 40% Silica fume 60% HCFA

4. CONCLUSION

In this study on HCFA based geopolymer mortar incorporating silica fume, the following conclusions are drawn.

Geopolymer mortar mixes produced noticeably higher compressive strength at all ages at ambient curing temperature when sodium hydroxide concentration was14M. When NaOH molar concentration was increased, the compressive strength of specimens was augmented by 18.4%, 28.5%, and 38.6% for 10M, 12M and 14M respectively. The addition of silica fume with HCFA increased the compressive strength of mortar specimens in all the four molar concentrations of NaOH (8M, 10M, 12M, and 14M) in the range of 20-58%. This is because of the filler effect of the silica fume and denseness of the alkaline liquid. However, silica fume content beyond 40% results in decreasing the compressive strength of mortar specimens. At 8M concentration of NaOH the maximum compressive strength of 38.62MPa was obtained for 40% replacement of silica fume



(8FS40). For every 2M interval the same 40% silica fume specimen attained its maximum compressive strength of 41.07 MPa, 44.13MPa and 47.79MPa for the mixes 10FS₄₀, 12FS₄₀ and 14FS₄₀ respectively. The mix containing 14FS₄₀ having 60% HCFA and 40% silica fume achieved higher compressive strength of 47.79MPa compared to all other mixes studied.

XRD Diffractogram analysis of geopolymer mortar indicates minor crystalline phase and major amorphous phase. More mineral compounds such as quartz, calcite, hematite, boetimite, hornblende and anatase formed in the matrix contributed higher strength in the 14FS₄₀. SEM micrograph indicates that HCFA with silica fume demonstrated dense and compact structure with more C-A-S-H gel, N-A-S-H gel and hydroxysodalite gel products and imparting significant gain in compressive strength.

References

- 1. Prakash R. Voraa, Urmil V. Dave,"Parametric Studies on Compressive Strength of Geopolymer Concrete", Procedia Engineering 51: 210 - 219, 2013.
- 2. Mohammed BS, Fang OC, "Mechanical and durability properties of concretes containing paper-mill residuals and fly ash", Construction and Building Materials 25:717-725, 2011.
- 3. Lateef N. Assi, Edward (Eddie) Deaver, Mohamed K. El Batanouny, Paul Ziehl, "Investigation of early compressive strength of fly ash-based geopolymer Concrete", Construction and Building Materials 112: 807-815, 2016.
- 4. Daniel L.Y. Kong, Jay G. Sanjayan, Kwesi Sagoe-Crentsil, "Comparative performance of geopolymers made with metakaolin and fly ash after exposure to elevated temperatures", Cement and Concrete Research37: 1583-1589, 2007.
- 5. Prakash R. Vora, Urmil V. Dave, "Parametric Studies on Compressive Strength of Geopolymer Concrete", Procedia Engineering 51: 210 - 219, 2013.
- 6. Rohit Zende, Mamatha. A, "Study on Fly Ash and GGBS Based Geopolymer Concrete under Ambient Curing", Journal of Emerging Technologies and Innovative Research 2 (7) (2015) 3082-3087.
- 7. Shadi Riahi, Ali Nazari, "The effects of nanoparticles on early age compressive strength of ash-based geopolymers", Ceramics International 38: 4467-4476, 2012.
- 8. Partha Sarathi Deb, Pradip Nath, Prabir Kumar Sarker, "The effects of ground granulated blast-furnace slag blending with fly ash and activator content on the workability and strength properties of geopolymer concrete temperature", cured at ambient Materials and Design (2014),doi: http://dx.doi.org/10.1016/j.matdes.2014.05.001.
- 9. Chien-Chung Chen, Ivan Diaz, Kathleen Menozzi, Luis Murillo, "An experimental study on slag/fly ash-based geopolymer concrete", Proceedings of ISER 11th International Conference, San Francisco, USA, 26-30, 2015.
- 10. Ashley Russell Kotwal, Yoo Jae Kim, Jiong Hu, Vedaraman Sriraman, "Characterization and Early Age Physical Properties of Ambient Cured Geopolymer Mortar Based on Class C Fly Ash", International Journal of Concrete Structures and Materials 9 (1): 35-43, 2015.
- 11. J. Temuujin, R.P.Williams, A. van Riessen, "Effect of mechanical activation of fly ash on the properties of geopolymer cured at ambient temperature", Journal of Materials Processing Technology 209: 5276-5280, 2009.
- 12. B. Vijaya Rangan, "Fly Ash-Based Geopolymer Concrete, Proceedings of the International Workshop on Geopolymer Cement and Concrete", Allied Publishers Private Limited, Mumbai, India, 68-106, 2010.
- 13. Xueying Li, Xinwei Ma, Shoujie Zhang, Enzu Zheng, "Mechanical Properties and Microstructure of Class C Fly Ash-Based Geopolymer Paste and Mortar", Materials 6: 1485-1495, 2013. doi:10.3390/ma6041485.
- 14. Guo, X.L. Shi, H.S. Dick, W.A, "Compressive strength and microstructural characteristics of class C fly ash geopolymer", Cement Concrete Composites 32: 142-147, 2010.
- 15. KolliRamujee, Potharaju.M, "Development of Low Calcium Flyash Based Geopolymer Concrete", IACSIT International Journal of Engineering and Technology 6(1): 1-4, 2014.
- 16. R.Manickavasagam, G.Mohan kumar, "Short term properties of High Calcium Flyashbased Geopolymer binder", IOSR Journal of Mechanical and Civil Engineering 14 (1) III: 13-20, Jan. - Feb. 2017.

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- V.Revathi, R.Saravanakumar, J.Thaarrini, "Effect of molar ratio of SiO₂/Na₂O, Na₂SiO₃/NaOH ratio and curing mode on the compressive strength of ground bottom ash geopolymer mortar", International Journal of Earth Sciences and Engineering 7 (4): 1511–1516, 2014.
- Eslam Gomaa, Simon Sargon, Cedric Kashosi, Mohamed ElGawady, "Fresh properties and compressive strength of high calcium alkali activated fly ash mortar", Journal of King Saud University – Engineering Sciences 29: 356–364, 2017.
- Xiaolu Guo, Huisheng Shi, Warren A. Dick, "Compressive strength and micro-structural characteristics of class C fly ash Geopolymer", Cement & Concrete Composites, 32: 142–147, 2010.
- Kong, D.L.Y, Sanjayan, J.G, "Effect of elevated temperatures on geopolymer paste, mortar and concrete", Cement Concrete Research40: 334–339, 2010.
- Manjunath S. Sontakki, Swapnil B. Cholekar, "Strength Performance Studies on Ambient Cured Silica fume based Geopolymer Concrete", International Research Journal of Engineering and Technology 2(7): e-ISSN: 2395-0056, p-ISSN: 2395-0072, 2015.
- D.R.M. Brew, K.J.D. MacKenzie, "Geopolymer synthesis using silica fume and sodium aluminate", Journal of Material Sciences, 42: 3990–3993, 2007.
- Khater HM, El-Sabbagh BA, Fanny M, Ezzat M, Lottfy M, "Effect of nano-silica on alkali activated water cooled slag geopolymer", ARPN Journal of Engineering and Applied Sciences 2 (2012) 170–176.
- 24. Riahi S, Nazari A, "The effects of nano particles on early age compressive strength of ash-based geopolymers", Ceramics International 38: 4467–76, 2012.
- 25. Ping Duan, Chunjie Yan, Wei Zhou, "Compressive strength and microstructure of fly ash based geopolymer blended with silica fume under thermal cycle", Cement and Concrete Composites 78 (2017) 108-119.
- Antonia, Lucky Chandraa, Djwantoro Hardjitoa, "The impact of using fly ash, silica fume and calcium carbonate on the workability and compressive strength of mortar", Procedia Engineering 125: 773 – 779, 2015.
- M. Rostami, K. Behfarnia, "The effect of silica fume on durability of alkali activated slag concrete", Construction and Building Materials 134: 262–268, 2017.
- Suresh Thokchom, Debabrata Dutta, Somnath Ghosh, "Effect of Incorporating Silica Fume in Fly Ash Geopolymers, International Journal of Civil and Environmental Engineering 5(12): 750-754, 2011.
- F.N. Okoye, J. Durgaprasad, N.B. Singh, "Effect of silica fume on the mechanical properties of fly ash basedgeopolymer concrete", Ceramics International, http://dx.doi.org/10.1016/j.ceramint.2015.10.084
- A.M. Rashad, S.R. Zeedan, "A preliminary study of blended pastes of cement and quartz powder under the effect of elevated temperature", Construction Building Materials 29: 672-681, 2012..

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