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Effectiveness of Alkali Activators on Nano Structured Flyashin Geopolymer Mortar

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Abstract

Background/Objectives: The cement industry is seeking ways to reduce the energy resource consumption and environmental load due to cement production. The usage of Supplementary Cementitious Material (SCMs) as a partial replacement of ordinary Portland cement can reduce its environmental load as well as the reduction in construction cost. **Methods:** In this work it is intended to analyze the mortarcube compressive strength by replacing the cement by 50% of Raw Fly ash and Sieved fly ash, with silica 10, 30% with 12 M Sodium Hydroxide and with and without the addition of lime 10, 30% in one series. Similarly, another set of studied with the ratio of 1:2.5 Sodium Hydroxide 12 M and Sodium Silicate asalkali activators to know the characteristics of the modified mortar. The mortar specimens curedin oven for 24 hrs at 60°c and then left for air curing for predetermined ages at 3, 7, 14, 28 days of curing. The micro structural test was conducted to know their morphological characteristics, particle size and their chemical compositions of cement, Raw fly ash and Sieved fly ash. **Findings:** Results from the work showed that modified mortar prepared using Sieved fly ashgave 9% increase in compressive strength than RFA. The higher percentage of replacements oflime is observed to increase the strength of Sieved Fly Ash (SFA) mortars. SEM results show that SFA sample taken at different resolution indicate smallest particle size to be 106.4 nm, and EDX results show that reducing the particle size has reduced the carbon content by 10% improved the properties of fly ash. **Application/Improvements:** Use of sieved fly ash, increases the strength in geopolymer properties is more advantageous in the construction industry for utilizing the fly ash in largequantity and thereby reducing the use of cement content also minimize the CO_2 emission during the production in the cement industry.

Keywords: Alkali Activated Mortar, Compressive Strength, Nano Structure Materials, Oven Curing, Sieved Fly Ash

1. Introduction

The ordinary Portland cement production process is energy intensive process and release a more quantity of CO_2 emissions to the environment[1]. The pervious researcher reported that 13500 million tonnes of CO_2 produced worldwide while production of cement[2]. The environment pollution have to minimize by inventing the alternate for cement in construction industry to minimization of cement usage[3]. Recently, a new cementitious materials like alkali activated geopolymer was developed to replace the part of Portland cement to minimise the harmful gas emissions to the environement[4]. The new generated material is usually fly ash based material and termed Supplementary Cementing Material (SCM) or alkali-activated fly ash cement materials[5,6]. The geopolymermortar is innovative materials prepared using alkali activated fly ash as the sole binder mixture. The innovative cementitious material has undergone substantial development over the last two decades due to their rich in mechanical performance and durability properties. The maximum size of fly ash particles range in size from 0.5 to 100 μm[7–10]. The many researcher have done extensive work in fly ash with geopolymer mortar which gives better properties in strength and resistance to the aggressive environment compare to the control mortar. The Geopolymer gel are formed and activate the fly ash mortar when alumino-silicates dissolve in a high molarity

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alkaline solution and precipitate in a hardened state[11–13]. In cement industry the significant quantity of fly ash is used in the cement production, since it is having high amount the pozzolanic property in the concrete contains due to high amount of SiO_2 and Al_2O_3 contents[14–16]. Many works carried out on the geopolymer is mainly based on Class F (low calcium) fly ash. But the work with rich in calcium fly ash in the geopolymer is limited. Hence, in the present workstudy is made on the mechanical strength behaviour of high calcium fly ash based geopolymer mortar with the addition of alkali activators of NaOH and NaOH + Na_2SiO_3. In addition, the microstructure analysis also carried out using Scanning Electron Microscopy (SEM), as well as Energy Dispersive X-Ray test (EDX).

2. Experimental Programme

2.1 Materials and Activator used

Material used in this work are Ordinary Portland Cement (OPC), Raw Fly Ash (RFA), Sieved Fly Ash (SFA), lime, silica and standard Ennore sand with alkali activators like Sodium Hydroxide (NaOH) and Sodium Silicate (Na_2SiO_3) solution. The Ordinary Portland Cement (OPC) grade 43 satisfying the requirements of ASTM C 150[17] was used. The source material which is the fly ash being used in this experiment is high calcium fly ash (Class C) named as a Raw fly ash which is collected from Neyveli lignite corporation, Neyveli, Tamil Nadu. The RFA is sieved in 45-micron sieve to obtain the finer fly ash is the Sieved fly ash. In the present study the lime powder and silica was purchased with 99.9% and 98% purity respectively. The standard Ennore sand consists of three grades in equal proportions of Grade-1 (2 mm–1 mm), Grade-2 (1 mm-0.5 mm) and Grade 3 (0.5 mm-0.09 mm) to prepare the geopolymer mortar[18]. The NaOH in the form of pellets and Na_2SiO_3 as a liquid gel form is used to make the alkali activator solution.

2.2 Mixes of Geo polymer Mortar

In the first series the three samples of OPC (M1), OPC and RFA in equal proportion (M2) and OPC and SFA in equal proportion (M3) are used. In the second set of series lime and silica were mixed in the M1, M2 and M3 with the Alkali activator. The details of materials proportion (OPC, RFA, SFA, lime and silica) with the alkali activators (12 M NaOH and 12 M NaOH+ Na_2SiO_3) and labels used are given in Table 1. Two alkali activator 12M NaOH and 12M NaOH + Na_2SiO_3 of 1:2.5 ratio is used to prepare the mortar samples. The alkali activator which is used, mixing of NaOH and Na_2SiO_3 Solution in the ratio of 1:2.5 prepared at room temperature and kept for 24 hrs before preparation of geopolymer mortar specimens. The binder to sand ratio 1:2.75 was used and the water to binder ratio is taken based on the Normal consistency according to the IS:4031[19] Part-4 used for all mix series to prepare the mortar specimens.

2.3 Specimen Preparation and Test Procedure

The cube of size 50mm is used to determine the compressive strength of mortar cube according to ASTM C 109[20]. The fly ash and sand are thoroughly mixed, after that the alkali solution has mixed on the weight basis. Alkali solution is mixed with the fly ash and sand until it gains the same colour throughout the material using Hobart mortar mixer. After that, the fresh mortar is placed in 50 mm cube steel mould in two layers and well compacted. The specimens prepared in the cube moulds are covered with Aluminium foil sheet[21] to prevent the loss of moisture. The prepared samples were kept in oven curing at temperatures of 60°c for the duration of 24 hrs. The moulds are taken out after 24 hrs from the elevated oven curing and kept at ambient temperature for air cur-

Table 1. Sample composition

Mix Series	Material proportions in %					Alkali Activator Used	
	OPC	RFA	SFA	LIME	SILICA	NaOH	NaOH 12M + Na_2SiO_3
M1	100	-	-	-	-	-	-
M2a	50	50	-	-	-	12M	-
M2b	40, 20	50	-	10,30	-	12M	-
M2c	40, 20	50	-	-	10,30	12M	-
M2d	50	50	-	-	-	-	1:2.5
M2e	40, 20	50	-	10,30	-	-	1:2.5
M2f	40, 20	50	-		10,30	-	1:2.5
M3a	50	-	50	-	-	12M	-
M3b	40, 20	-	50	10,30	-	12M	-
M3c	40, 20	-	50	-	10,30	12M	-
M3d	50	-	50	-	-	-	1:2.5
M3e	40, 20	-	50	10,30		-	1:2.5
M3f	40, 20	-	50	-	10,30	-	1:2.5

ing then tested at the duration of 3, 7, 14, 28 days to study the compressive strength of mortar specimens according to the IS:1727[22].

3. Results and Discussions

3.1 Physical Properties

From the physical analysis made on the samples, the Specific gravity and the Blaine's Surface area of OPC, RFA and SFA were found to be 3.1, 2.86 and 2.1 and 225, 422 and 727 % respectively. The specific surface area is increased for the SFA samples due to the particle size is small compare to the OPC and RFA samples. In Table 2 shown the results of basic properties like normal consistency, initial and final setting time of OPC, RFA and SFA samples. From the laboratory test results, it was inferred that SFA sample water demand is high consider to OPC and RFA sample due to its lesser particle size, which decreases its spreading ability[23,24]. Initial and final setting time measured at a controlled temperature of 27°c shows the early setting time in SFA than OPC and RFA; it may be due to its spherical in shape and smaller in particle size.

3.2 Chemical Composition

The chemical composition of OPC, RFA and SFA sample were determined and it is reported in Table 3. From the laboratory test results, it is inferred that the Al-Si ratio is more in SFA, which influences the time taken for setting and increases of bonding strength property of geopolymer mortar[25].

Table 2. Physical properties

Mix	Material Combination	Normal Consistency %	Initial Setting Time	Final Setting Time
M1	OPC	48	20	58
M2a	OPC+ RFA50%+NaOH 12M	52	25	65
M2d	OPC+ RFA50%+NaOH 12M+ Na_2SiO_3	53	23	58
M3a	OPC+ SFA50%+NaOH 12M	56	19	53
M3d	OPC+ SFA50%+NaOH 12M+ Na_2SiO_3	58	18	50

Table 3. Chemical composition of materials

Composition (%)	OPC	RFA	SFA
SiO_2	20.6	49.17	34.9
Al_2O_3	5.07	24.26	27.24
Fe_2O	2.9	6.04	7.02
CaO	63.9	11.41	18.97
MgO	1.53	5.02	1.7
SO_3	2.53	1.67	4.24
Na_2O	0.15	0.25	1.25
K_2O	0.73	0.05	1.7
LOI	1.58	2.02	1.08

3.3 Compressive Strength of Geopolymer Mortar

3.3.1 Compressive Strength of Control Mortar with GeopolymerMortar

To determine the compressive strength of OPC and geopolymermortar specimens were prepared by replacing 50% OPC by RFA and SFA with alkali activators at the varying curing periods of 3, 7, 14 and 28 days are shown in Figure 1. From the figure, it is inferred that the compressive strength of geopolymer specimens prepared using the alkali activator 12M NaOH with SFA shows the 14% higher than the geopolymer prepared with RFA at the testing age of 28 days. Similarly, the geopolymer specimen prepared using 12M NaOH and Na_2SiO_3 SFA shows the 9% higher compressive strength than RFA at the age of 28 days. However, the geopolymer mortar samples give lesser strength than the OPC mortar specimens. It may be due to the High volume replacement and also low pozzolanic reactivity of fly ash.

3.3.2 Effect of NaOHAlkali Activated Mortar

The strength of geopolymermortar cubes prepared with 50% replacement by RFA and SFA in OPC and alkali activator is studied by replacing the OPC with the lime content of 10 and 30 %. The variations of test results on curing periods are shown in Figure 2. The addition of 30% replacement of lime with the SFA mix gives 17% higher strength than the geopolymer mix with RFA. In general, the addition of lime of 10% and 30% on both RFA and SFA yields higher strength than the geopolymer mix[26]. Similar studies also made with silica instead of lime on the geopolymer mortars. At theage of 3, 7, 14 and 28 days of the compressive strength cubes were prepared with

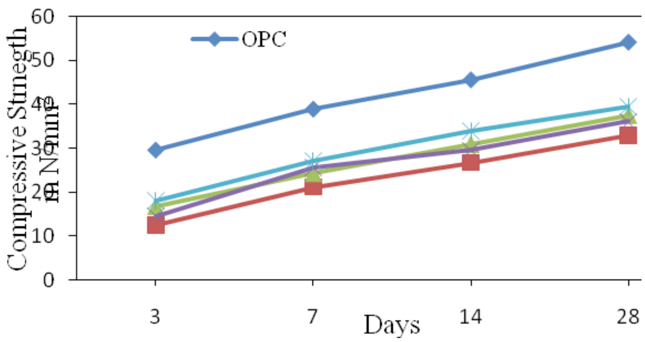

Figure 1. Variation of compressive strength of OPC and fly ash geo polymer mortar.

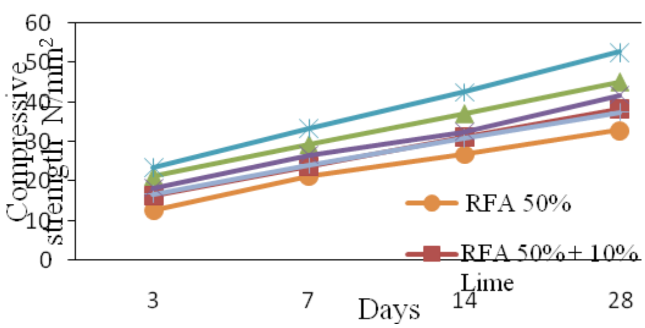

Figure 2. Compressive strength of RFA, SFA with addition of lime mortar with 12M NaOH.

different alkali activator specimen test results shown in Figure 3. The studies with the 10% and 30% of silica also show the higher strength with SFA than RFA in the order of 42% and 33% respectively.

3.3. 3Effect of 12M NaOH and Na2SiO3 Alkali Activated Mortar

In the other series of tests, the experiments were carried out to understand the effect of alkali activator on the geopolymer mix with the lime and silica addition of 10 and 30 % as given in Table 1. The studies with the alkali activator of 12M NaOH and Na_2SiO_3 with RFA and SFA are carried out similar to the mix with 12M NaOH. To study thecompressive strength of cubes prepared using silica and lime with alkali activator specimens test results are shown in Figures 4 and 5. From the results it was observed thatthe alkali activator 12M NaOH and Na_2SiO_3 show 5% higher compressive strength than the 12M NaOH on the geopolymer with SFA. Similarly, the addition of lime and silica content of 30% shows the higher strength of 11% and 6% respectively in SFA geopolymer mix than RFA mix. In

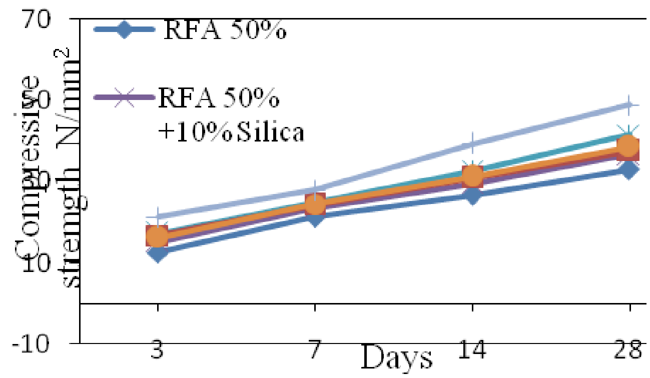

Figure 3. Compressive strength of RFA, SFA with addition of silica mortar with 12M NaOH.

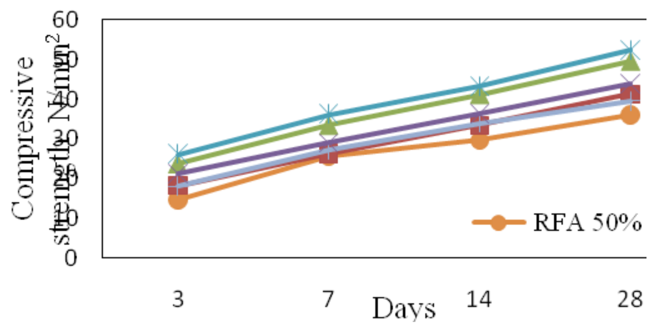

Figure 4. Compressive strength of RFA, SFA with addition of Lime with 12M NaOH and Na_2SiO_3 Alkali activated mortar.

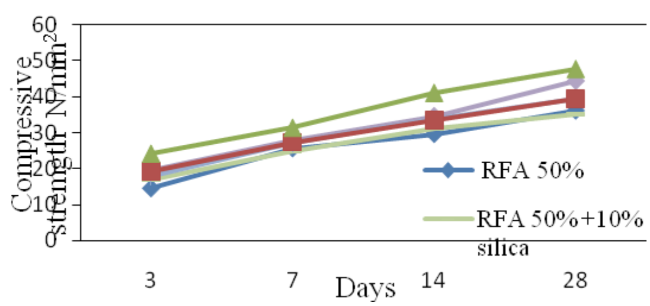

Figure 5. Compressive strength of RFA, SFA with addition of Silica with 12M NaOH and Na_2SiO_3 Alkali activated mortar.

general, the addition of alkali activator 12M NaOH and Na_2SiO_3 shows the higher strength than 12M NaOH.

3.4 Scanning Electron Microscope Test

Morphology features considering the shape and surface texture of the OPC mortar and alkali activated geopolymer specimen prepared with raw fly ash and sieved fly ash samples were analyzed using a scanning electron microscope. A SEM images of OPC, RFA and SFA geopolymer mortar specimen samples has been analysed at different resolutions at the testing duration of 28 days which are shown in Figure 6(a) and (b), 6(c) and (d) and 6(e) and (f) respectively. From the Figure 6(c) and (d) it is observed microstructures are shows mostly spherical in shape and glassy in the microstructure of RFA particles. The SFA geopolymer specimen image, Figure 6 (e) and (f) show that the particle surface is more uneven, irregular in shape[27] and also, there is a reduction in size.

3.5 EDX (Energy Dispersive X-Ray) Test

The Table 4 and Figures 7 (a), (b), (c) shows the EDX results of OPC, RFA and SFA samples which determine the chemical distribution of the material. From the results, it is inferred that silica and aluminium content is more in SFA compare to the RFA and OPC, which might be thereason for the strength variation

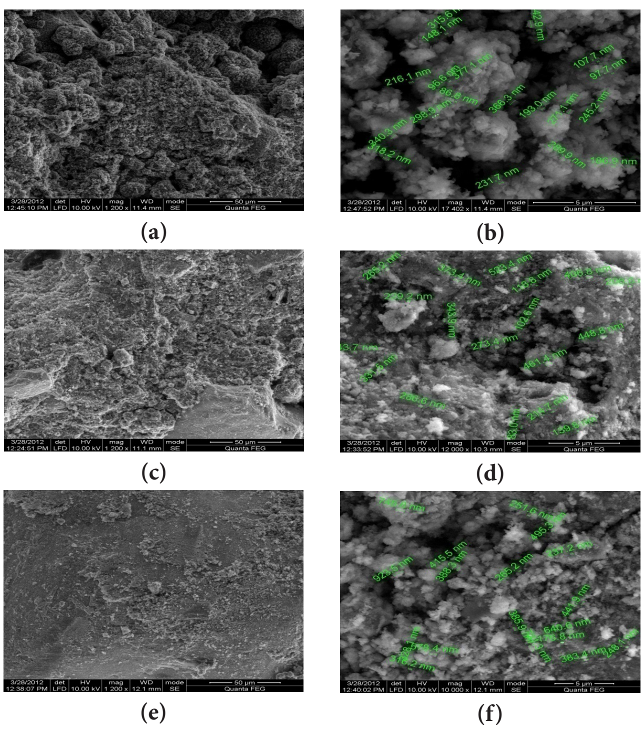

Figure 6. SEM images of OPC, RFA and SFA samples. **(a)** SEM images of OPC at magnification of 50 µm. **(b)** SEM images of OPC at magnification of 5 µm. **(c)** SEM images of RFA at magnification of 50 µm. **(d)** SEM images of RFA at magnification of 5 µm. **(e)** SEM images of SFA at magnification of 50 µm. **(f)** SEM images of SFA at magnification of 5µm.

Table 4. EDX results OPC, RFA and SFA samples

El.	OPC	RFA	SFA
O	46.73	46.56	46.79
C	0	25.05	24.13
Al	0	11.27	15
Si	7.58	7.36	10.84
Ca	45.69	5.03	1.25
Cu	0	0	0.74
Mg	0	0.99	0.69
Ti	0	0.94	0.56
Na	0	0.54	0
Fe	0	2.26	0

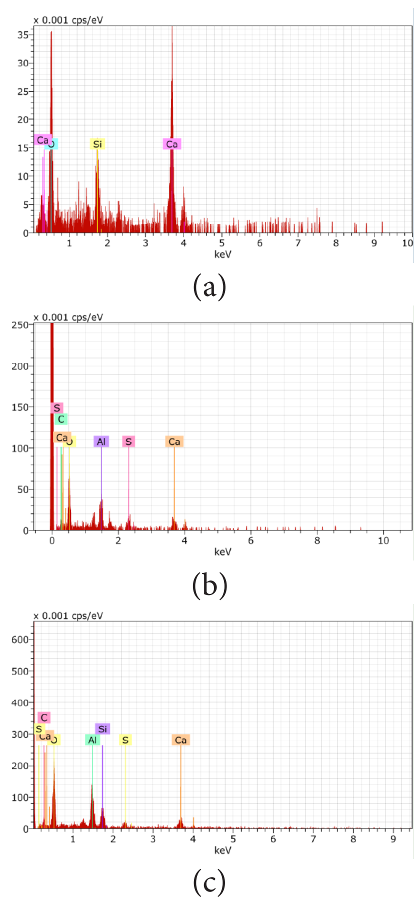

Figure 7. **(a)** EDS spectra of OPC. **(b)** EDS spectra of RFA. **(c)** EDS spectra of SFA

and increased durability in mortar specimens[28], which is evident from chemical composition and compressive strength results.

4. Conclusion

From the work carried out to study the mechanical properties of geopolymer mortars the following conclusion has drawn:

- It has found that the 50% replacement of OPC by RFA and SFA with alkali activators, the mortars with SFA shows higher strength than RFA, it can be due toparticle size reduction in and the particles are spherical in shape.
- The addition of 10, 30 % of Lime and silica with geopolymer mortar shows that 30% replacement of Lime gives the better strength of SFA than RFA in both Alkali activator solution which may due to the addition of CaO is more.
- The mineralogical phases and characteristics of microstructural elements of SFA mortars are differed, from the properties of OPC and RFA mortars in terms of the particle size and shape.
- From the laboratory test results of the study can conclude for the large scale usage of fly ash geopolymer mixtures for identifying the good binder materials in concerning the compressive strength and increase in setting time.

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