



Impact in Mineralogical Composition of Geopolymerization Actuation using Fly Ash as the Sole Binder

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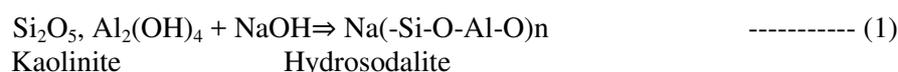
Abstract

The use of large amounts of cement quantity to increase in carbon dioxide emissions in the world and hence an alternative binding material is required for a sustainable development. The utilization of alumina silicate materials to mostly replace cement content or creation of geopolymer concrete is a noteworthy improvement towards the gainful utilization of modern waste items. Alumino silicates, for example, fly ash, blast furnace slag and metakaolin can be actuated utilizing soluble arrangements of hydroxide and silicates of Sodium or potassium to deliver cement free binders. In this experimental study, Geopolymer mortar specimens were prepared with fly ash and activated with NaOH 6M and 8M solution. Effect of particle size of raw flyash (RFA), particles sized less than 45 μ sieved fly ash (SFA) concentration of NaOH (6M, 8M) and varying curing methods and temperatures are curing at ambient temperature, oven curing at 60°C, 80°C, Steam Curing at 60°C, 80°C were the parameters considered. The RFA mortar specimens casted with 8M NaOH solution utilizing normal Curing indicates 12% more compressive strength compared to mortar samples activated with 6M NaOH solution. The SFA mortar specimens shows 5% increase in compressive strength under the same conditions implying the effect of particle size on the compressive strength development. Specimens prepared with 45μ sieved fly ash developed higher strength compared to specimens prepared with raw fly ash. In the present study there is a particular instance of a conceivable modern use of these new materials in the segment of precast industry.

1. Introduction

Geopolymer is an alkaline based binder materials formed in secondary cementitious materials, for example, fly ash and rice husk ash. In polymerization process, when a basic arrangement is utilized to respond with the silica and the alumina from the raw material [1,2]. The polymerisation process work under alkaline condition on Si-Al minerals under takes a quick substance initiation, which gave the polymeric chain in the method for three dimensional and ring structure comprising of Si-O-Al-O bonds [3-5].

In geopolymerization process the alumino silicate kaolinite reacts with NaOH at 100-150°C and polycondenses into hydrated sodalite (a tecto-alumino-silicate), or hydro-sodalite. The reaction mechanism is shown below in equation (1):



Geopolymerisation (Figure 1) process involves reaction under alkaline solution condition on Si-Al minerals, that shows in a polymeric chain with three dimensional and ring structure consisting of Si-O-Al-O bonds as shown below [6-9]:



Where, M- The alkaline element such as potassium, sodium or calcium; the symbol shows presence of a bond and n- polymerisation degree; z- 1, 2, 3, or more.

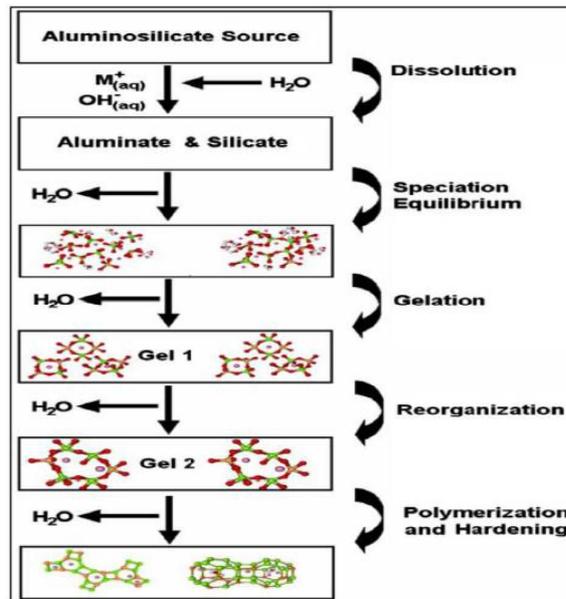
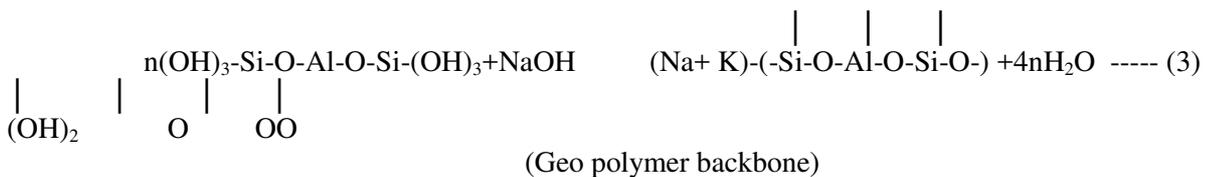
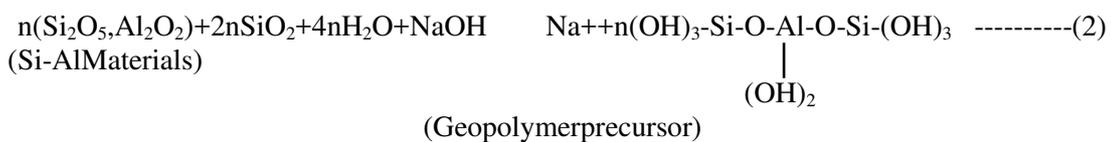


Figure 1: Geo Polymerization Process

The schematic formation of geopolymer mortar specimen shown is in equations (2) and (3).



From the equation (2) observed that, the water is released when the chemical reaction starts which results the formation of geopolymers. This water is removed from geopolymer matrix process during the curing periods, decrease the porosity in the geopolymer matrix. This process will ensure that the performance of geopolymers increases and also, the chemical reaction plays important role which enhance the workability to the mortar during handling process [10, 11]. Reactivity of natural pozzolan might be increased by prolonged grinding, thermal activation, chemical activation, alkali activation. Reduction in particle size directly affects the reactivity of fly ash, which is evident from the testing of mortar specimens [12-14]. The method of curing adopted plays an active role in the strength development of geopolymers specimens prepared utilizing fly ash. A higher curing temperature does not guarantee higher 28 day compressive strength [15]. The oven and steam curing time can be restricted to 24 hours since rate of strength development is high in this period.

In the present study, compressive strength of geopolymer mortar specimens prepared with high concentration of alkali activator has been determined. The effect of sieved fly ash over the raw fly ash geopolymer mortar and effect of temperature and curing methods is considered.

2 Experimental Work

2.1 Materials

The fly ash is collected from Neyveli, Tamil Nadu was utilized as a part of present work, which is Class C fly ash as indicated by the ASTM C 618 [16]. The collected class C fly ash is termed as raw Fly Ash (RFA). The RFA material passing through a 45µ sieve is termed as Sieved Fly Ash (SFA). Sodium hydroxide solution is the sole alkaline activator. Aluminosilicate materials are more soluble in sodium based solutions and it is cheaper than Potassium-based solutions. The sodium hydroxide (NaOH) solution was prepared by diluting the pellets in distilled water. Ennosandof Grade I, Grade II and Grade III conforming to IS 650:1991 [17] was collected from Tamil Nadu Minerals Limited mixed in equal proportion is used for the preparation of mortar cubes.

2.2 Preparation of Geopolymer Specimens

The mortar specimens were prepared completely RFA and SFA without cement. The NaOH alkali activated solution were prepared with 6 Molarity and 8 Molarity concentrations. The binder to ratio used to prepare the mortar cubes is 1:2.75. The mix ratio is adopted as per the ASTM C 270. The water to binder ratio were taken based on the normal consistency value according to IS 4031: part-4 [18]. The three different curing methods are Normal, Steam and Oven curing with 60°C and 80°C temperature were used for curing the specimens. The mortar specimens were prepared with the sand and fly ash samples are mixed until the mixture is thoroughly blended for 5 minutes. The required amount of the alkali is added based on normal consistency value and mixing is done thoroughly through Hobart mortar mixer according to the IS4031. The 50 mm size of steel moulds is filled with mortar in two layers with proper compaction. Three mortar cube specimens were prepared for each mix combination and curing method to determine the compressive strength of mortar cubes.

2.3 Curing and Testing of Mortar specimens

The RFA and SFA sample basic test properties are Blaine's fineness test, physical properties and chemical properties test was performed according the IS1727:1984. The mortar cube specimens were cured with three unique types curing like Normal, Steam and oven curing. The cubes for oven and steam curing were wrapped by aluminum thwart sheet before putting in the oven for 24 hours following 2 hours of after casting kept in oven at 60°C and 80°C. Demoulding of cubes is done following two hours of the predetermined curing time frame. For steam curing, the cubes were put for 24 hours in the steam curing machine at 60°C and 80°C. Demoulding is done following four hours of the required curing time frame. For ordinary (curing at encompassing temperature) the mortar cubes are demoulded following 24 hours length from the season of casting. Amid curing of geopolymer mortars examples at elevated temperatures, test specimens are to be wrapped and afterward sealed. This precautionary measure has been taken keeping in mind the end goal to keep intemperate loss of dampness from the specimens during curing. The compressive strength for the cubes arranged with RFA and SFA mortar cubes were conducted at age of 3, 7, 14, 28 days subsequent to casting. The mortar cubes were tried in determined age understanding with ASTM C109 [19].

2.4 Microstructural studies

The microstructural behavior characteristics of Raw fly ash and Sieved fly ash were examined utilizing a X-ray diffraction characterization strategy to locate the crystalline size of the quartz stages by utilizing a quickening voltage of 30 kV and a current of 20 mA. The specimens examined at a speed of 2 deg.min⁻¹ in the 2θ territory from 10 to 70°. The microstructures of raw fly ash and sieved fly ash samples utilized as a part of this study are investigated utilizing an examining scanning electron microscope(SEM) to find out the basic organization, morphology characters and surface [11-13].

3 Results and Discussions

3.1 Basic Property Test

The fundamental property test are Specific gravity, normal consistency, particle size analysis of RFA and SFA materials were tried. The physical and chemical composition of RFA and SFA material test outcomes appeared in Table 1.

Table 1: Physical and Chemical Composition of materials

Composition	RFA	SFA
Silicon Dioxide (SiO ₂), %	35.17	34.91
Aluminum Oxide (Al ₂ O ₃), %	27.6	27.8
Iron Oxide (Fe ₂ O ₃), %	6.84	7.10
Sum of SiO ₂ , Al ₂ O ₃ , Fe ₂ O ₃ , %	69.61	69.81
Calcium Oxide (CaO), %	19.41	18.87
Magnesium Oxide (MgO), %	1.72	1.71
Sulphur Trioxide (SO ₃), %	4.67	4.37
Potassium (K ₂ O), %	0.25	0.2
Loss on Ignition, %	2.72	3.68
Specific Gravity	2.62	2.55
Blaine's Specific Surface Area (cm ² /gm)	2657	4749
Mean diameter particle size in μ	21.35	8.62

The Blaine's fineness value was 2657 cm²/gm for RFA and 4749 cm²/gm. For SFA Blaine's values were increases while comparing with RFA samples due to the fineness of SFA samples. The normal consistency tests were done on the RFA and SFA with 6 and 8M of NaOH solution. It is seen that the consistency of RFA with the use of NaOH solution of 6 and 8M is 51.25% and 48.25% individually. The consistency of SFA with the expansion of NaOH solution of 6 and 8M is 47.5% and 45% individually. From the consistency test values it is watched that as the concentration of the alkali was increased, the consistency decreased. These results prove the expanded dissolvability of the alumino silicate material in fly ash at higher groupings of the alkaline solution [15]. The RFA and SFA sample chemical composition test results are compared and it is presented in Table 1. From the results, it is observed that, the chemical composition of SFA values are almost similar to that of RFA. However the specific gravity and the particle mean size is decreased in SFA. Considering the specific surface of SFA, it is increased from 2657cm²/gm to 4749 cm²/gm.

3.2 Effect of Change in Molarity of Activator Solution

The RFA tests with NaOH 6M and 8M solution used to prepare mortar cubes to decide the compressive strength at the testing age of 3, 7, 14 and 28th day. The test outcomes are presented in (Figure 2 and 3) respectively. 8M oven Cured specimens at 60°C and 80°C gives 49% and 32% higher strength in comparison to 6M oven cured mortar samples. The 8M steam Cured specimens at 60°C and 80°C gives 6% and 12% higher compressive strength in comparison to 6M steam cured mortar samples. The 8M normal cured specimens show 12% increase in compressive strength in comparison to 6M samples cured normally.

The test results of SFA mortar cubes are shown in (Figure 4 and 5). It is seen that the 8M oven curing with 60°C and 80°C gives 15% and 21% higher compressive strength compared to 6M oven cured mortar samples. The 8M steam Cured samples in 60°C and 80°C gives 3% and 6% higher compressive strength compared to 6M steam curing mortar samples. The 8M normal Cured samples give 5% higher compressive strength compared to 6M samples [21,11]. In early stage the Oven curing and steam curing gives more compressive strength in comparison with the normal curing mortar specimens, it may be due to the curing of sample in elevated temperature. Due to the increase in temperature there is a possibility of changes the polymerization process and it offers the better binding properties of between the particles in mortar specimen.

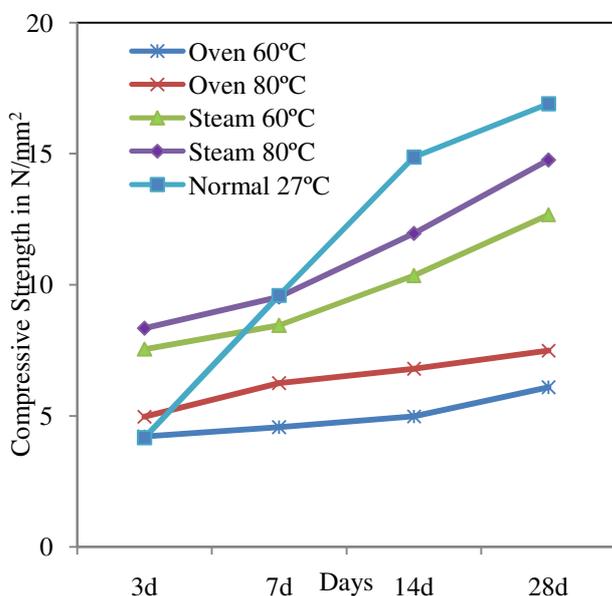


Figure 2: Compressive strength of RFA mortar specimens with 6M Alkali solution

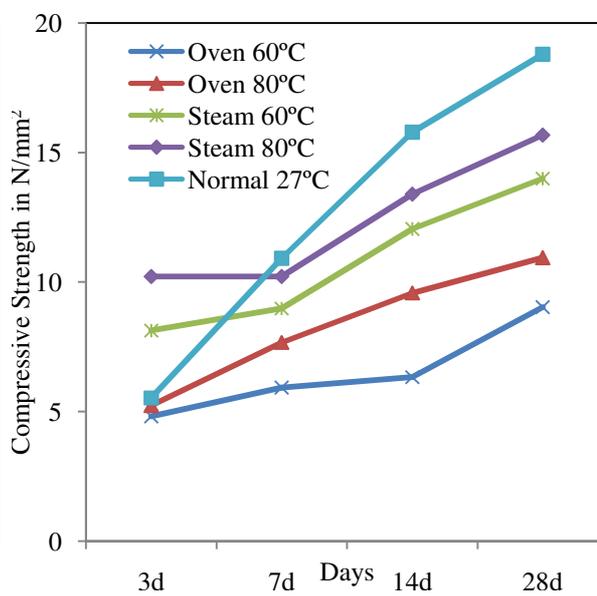


Figure 3: Compressive strength of RFA mortar specimens with 8M Alkali solution

3.3 Effect of particle size on compressive strength

The (Figure 6 and 7) represent the effect of particle size reduction on 28 day compressive strength of mortar cubes prepared with RFA and SFA in 6M and 8M. It is seen that the SFA mortar cubes prepared in 6M solution and cured in oven at 60°C and 80°C mortar gives 30 and 37% higher compressive strength compared to RFA specimens. The SFA mortar cubes prepared in 8M solution and cured in oven at 60°C and 80°C show 23% and 6% higher compressive strength compared to RFA specimens.

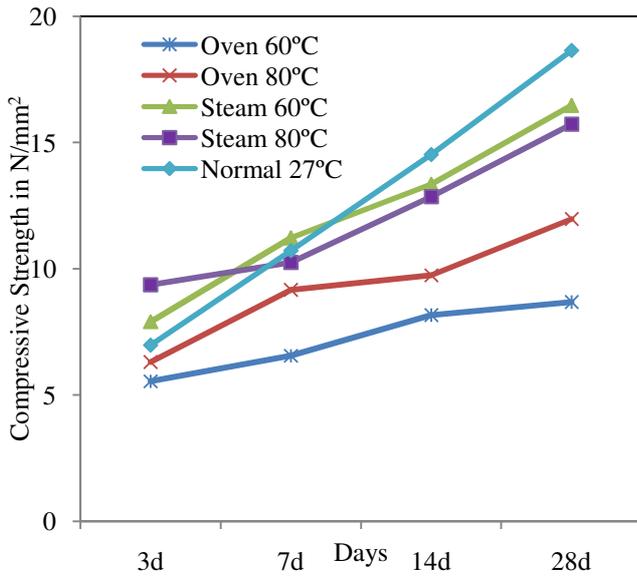


Figure 4: Compressive strength of SFA mortar specimens with 6M Alkali solution

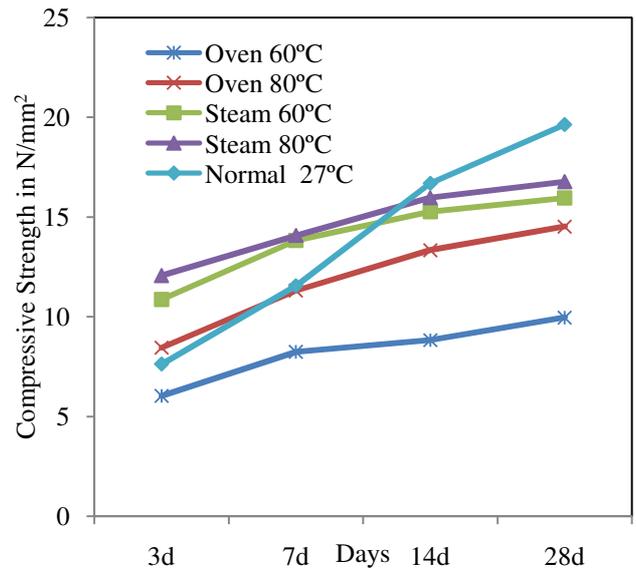


Figure 5: Compressive strength of SFA mortar specimens with 8M Alkali solution

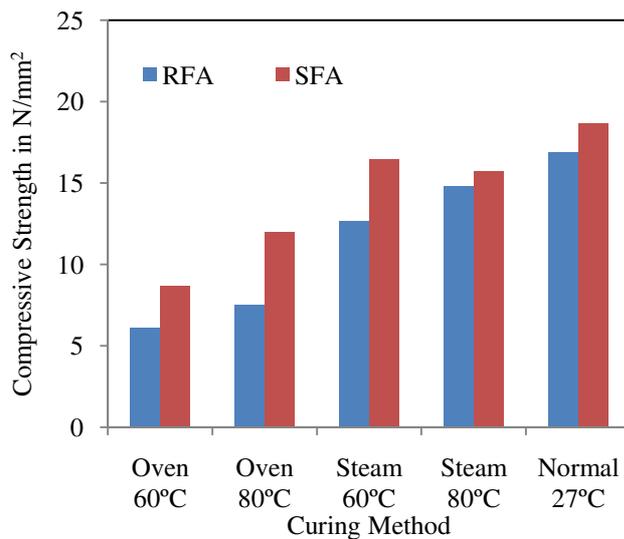


Figure 6: Compressive strength of 6M with Different curing Methods at 28 days

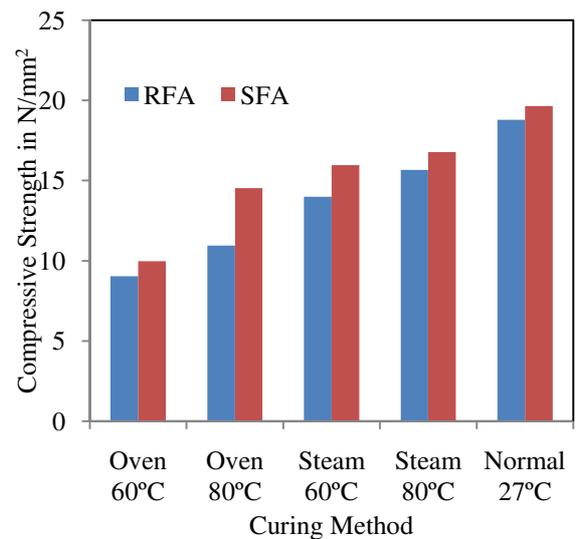


Figure 7: Compressive strength of 8M with Different curing Methods at 28 days

Similarly, the SFA mortar cubes prepared in 6M solution and cured at 60°C and 80°C in steam gives 9% and 25% higher compressive strength compared to RFA specimens. The SFA mortar cubes prepared in 8M steam cured at 60°C and 80°C in steam show 12% and 7% higher compressive strengths compared to RFA specimens.

3.4 Effect of Method of Curing and Fineness of Fly Ash on Cube Compressive Strength

The steam cured specimens gave a higher average compressive strength for RFA than FFA for the same molar ratio. The 28 day compressive strength test results is presented in (Figure 8). From the test results, it is observed that the compressive strength is increased in normal curing compare to the steam curing and oven curing. By comparing oven curing, the steam curing shows higher strength. The steam curing specimens strength is increased due to the polymerization process is done minimum water evaporation is occur. But in oven curing due to high temperature water evaporation is more. So the strength gain is more for steam cured specimens prepared with 8M concentration [22]. However the samples cured at ambient temperatures showed a higher 28day compressive strength than the other methods of curing.

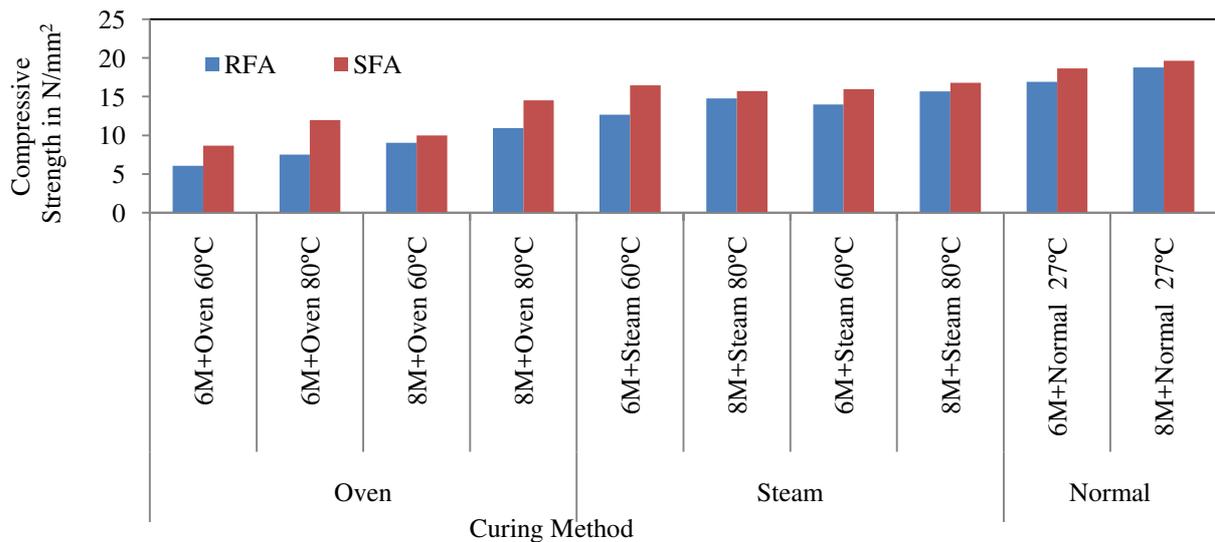


Figure 8: Compressive strength of mortar specimens with different curing methods and particle size reduction

3.5 SEM images of RFA and SFA samples

From the mix series, SEM images at 50µm magnification were analyzed the specimens at the age of 28 days, which showed highest compressive strength were taken as optimum mix proportions for SEM analysis. Images of RFA mortar specimens prepared with 8M solution at normal curing and SFA with 8M solution with normal curing at 28 days is presented in (Figure 9 and 10).

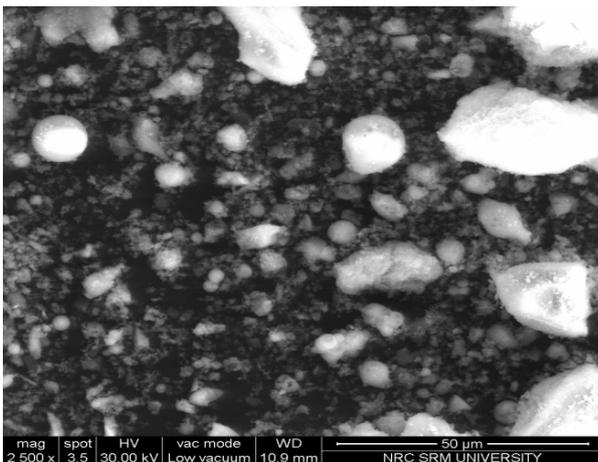


Figure 9: SEM image of RFA samples with 8M solution

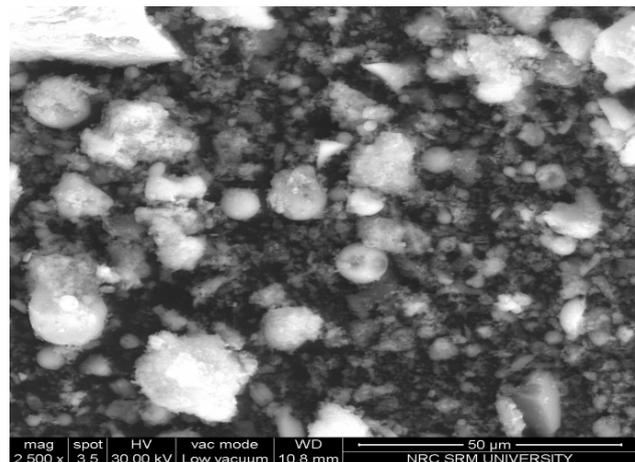


Figure 10: SEM image of SFA samples with 8M solution

The SEM pictures show that for the RFA test the smallest particle size measured was 162.7nm. For the fly ash test sieved utilizing 45µ sieve the smallest particle size noticed is 106.4 nm. [23, 24]. The effect of presence of smaller sized particles observed in 45micron sieved fly ash is evident in the increased compressive strength.

3.6 X-Ray Diffraction Analysis

X-ray diffraction is used to find the crystallite size of the phases of RFA and SFA samples, the results of which is shown in (Figure 11). PANalytical 'X' Pert PRO diffractometer using Cu-K α radiation ($\lambda = 1.54056 \text{ \AA}$) at an accelerating voltage of 40 kV and a current of 30 mA is used. XRD measurement showing phases present (peak position), phase concentrations (peak intensity), amorphous content (background hump) and crystallite size (peak width) is done. The samples were scanned at a step size of 0.017 ($^{\circ}$ Theta) in the 2θ range from 5° to 100° . The XRD patterns work on the principle of Bragg's law: $n\lambda = 2d \sin \Theta$. Comparisons of XRD powder pattern of the samples with RFA and SFA after 28 days of hydration shows that this similar peak present in both samples. This is, indeed, evident from the increased amorphous hump present below the crystalline peaks at both samples [25].

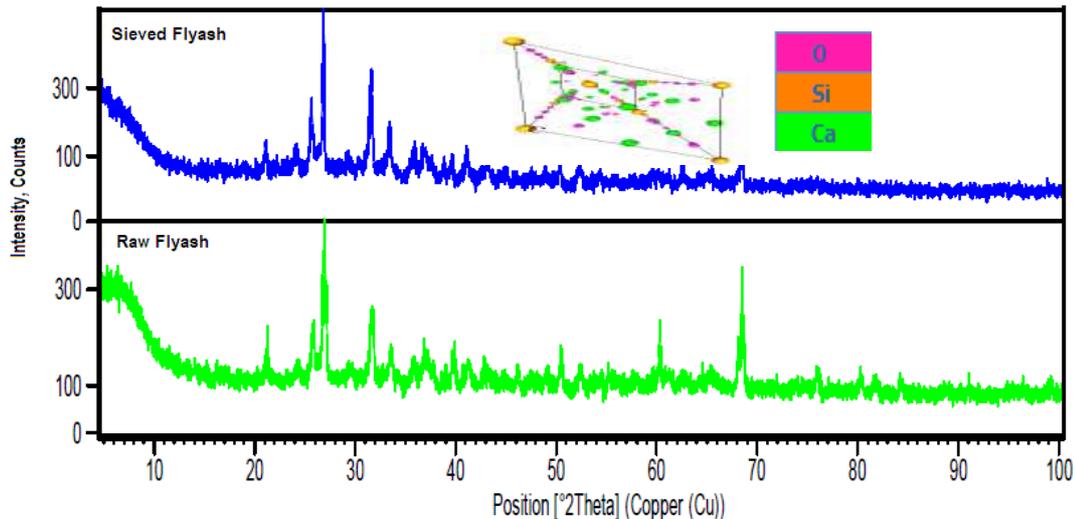


Figure 11: X-Ray Diffractogram of Raw Fly Ash and Sieved Fly Ash

Conclusions

From the detailed tests conducted by varying particle size, curing method, temperature, and concentration of alkaline solutions the following conclusions were drawn.

- Decrease in particle size affects the compressive strength development. Specimens prepared with SFA gained higher compressive strength than RFA specimens.
- The water demand is more for RFA sample compare to the SFA samples in both 6M and 8M solution.
- Specimens prepared with 8M molar concentration of alkaline base gives high compressive strength values under various curing conditions compare with 6M solution.
- Steam Curing brought about higher compressive strength improvement compared to oven curing and normal curing for samples prepared for both raw fly ash and sieved fly ash particles.
- Curing at elevated resulted in higher initial strength at compared to normal curing.
- By comparing raw fly ash and sieved fly ash on both oven and steam curing at 80°C brought about higher compressive strength than 60°C temperature curing
- Further increase in activator concentrations and use of finer particles can result in higher strengths. Geopolymer mortar / concrete can be a viable option for manufacture of precast products.

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