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Study on Characterization and Properties of Alkali Activated Fly Ash

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ABSTRACT

Concrete is a composite construction material, composed of cement and other cementitious materials such as fly ash and slag cement, aggregate (generally a coarse aggregate made of gravel or crushed rocks such as limestone, or granite, plus a fine aggregate such as sand), water and chemical admixtures. By increasing the curing temperatures in oven curing and steam curing the compressive strength has increased. This might be due to the rate of reaction of fly ash at higher temperatures. The oven curing gives higher compressive strength than normal curing and steam curing gives higher strength than oven curing. The decrease in the particle size increases the compressive strength. This might be due to the increase of smaller particles in fly ash would decrease the amount of pores.

KEY WORDS: alkali, analysis, characterization, concrete, fly ash.

1. INTRODUCTION

Concrete is a composite construction material, composed of cement and other cementitious materials such as fly ash and slag cement, aggregate (generally a coarse aggregate made of gravel or crushed rocks such as limestone, or granite, plus a fine aggregate such as sand), water and chemical admixtures. The cement industry is seeking ways to reduce the energy and resource consumption of concrete. Cement manufacturing releases CO_2 in the atmosphere both directly when calcium carbonate is heated, producing lime and carbon dioxide, and also indirectly through the use of energy if its production involves the emission of CO_2. The cement industry is the second largest CO_2 emitting industry behind power generation. It has been known that the use of supplementary cementing materials (SCMs) as a partial replacement of Portland cement in concrete can reduce its environmental load. The use of substantial volumes of SCMs reduces CO_2 emissions and saves energy and natural resources.

1.1. Supplementary cementitious material: Supplementary cementitious materials (SCMs) are generally by products from other processes or natural materials. They may or may not be further processed for use in concrete. Some of these materials are called pozzolans, which by themselves do not have any cementitious properties, but when used with Portland cement, react to form cementitious compounds. Other materials such as slag, do exhibit cementitious properties. For use in concrete, supplementary cementitious materials, sometimes referred to as mineral admixtures, need to meet requirements of established standards. They may be used individually or in combination in concrete. They may be added to the concrete mixture as blended cement or as a separately batched ingredient at the ready mixed concrete plant. Some of the supplementary cementitious materials used are fly ash, ground granulated blast furnace slag, condensed silica fume, limestone dust, cement kiln dust, and natural or manufactured pozzolans

1.2. Uses of SCMs: SCMs can be used for improved concrete performance in its fresh and hardened state. They are primarily used for improved workability, durability, and strength. These materials allow the concrete producer to design and modify the concrete mixture to suit the desired application. Concrete mixtures with high Portland cement contents are susceptible to cracking and increased heat generation. These effects can be controlled to a certain degree by using supplementary cementitious materials. Supplementary cementitious materials such as fly ash, slag and silica fume enable the concrete industry to use hundreds of millions of tons of byproduct materials that would otherwise be land filled as waste. Furthermore, their use reduces the consumption of Portland cement per unit volume of concrete. Portland cement has high energy consumption and emissions associated with its manufacture, which is conserved or reduced when the amount used in concrete is reduced.

1.3. Fly ash: Fly ash is one of the residues generated in the combustion of coal. Fly ash is generally captured from the chimneys of power generation facilities. These ash particles consist of silica, alumina, oxides of iron, calcium, and magnesium and toxic heavy metals like lead, arsenic, cobalt, and copper. This poses problems in the form of land use, health hazards, and environmental dangers. In addition to economic and ecological benefits, the use of fly ash in concrete improves its workability, reduces segregation, bleeding, heat evolution and permeability, inhibits alkali-aggregate reaction, and enhances sulfate resistance.

1.4. Fly ash classification: ASTM C618 categorizes fly ash as either Class C or Class F based on the origin of the coal used and the resulting fly ash chemical composition. Class F is a low-calcium fly ash and is pozzolanic, while Class C fly ash exhibits both pozzolanic and cementitious properties because of its high calcium content. The use of fly ash provides improved workability, increased long term compressive strength, reduced heat of hydration, decreased costs and increased resistance to alkali-silica reaction, and sulfate resistance (Class F only) when compared to unblended portland cement.

1.5. Class 'F' fly ash: The burning of harder, older anthracite and bituminous coal typically produces Class F fly ash. This fly ash is pozzolanic in nature, and contains less than 10% lime (CaO). Possessing pozzalonic properties, the glassy silica and alumina of Class F fly ash requires a cementing agent, such as Portland cement, quicklime, or hydrated lime, with the presence of water in order to react and produce cementitious compounds. Alternatively, the additions of a chemical activator such as sodium silicate to a Class F Fly ash can lead to the formation of a geopolymer.

1.6. Class 'C' fly ash: Fly ash produced from the burning of younger lignite or sub-bituminous coal, in addition to having pozzolanic properties, also has some self-cementing properties. In the presence of water, Class C fly ash will harden and gain strength over time. Class C fly ash generally contains more than 20% lime (CaO). Unlike Class F, self-cementing Class C fly ash does not require an activator. Alkali and sulfate (SO_4) contents are generally higher in Class C fly ash. Class C fly ash usually has cementitious properties in addition to pozzolanic properties due to free lime, whereas Class F is rarely cementitious when mixed with water alone.

1.7. Advantages of fly ash: When used as a portland cement replacement, fly ash offers the following advantages when compared to unmodified portland cement:

a) Increased early and late compressive strengths, b) Increased resistance to alkali silica reaction (ASR) when >15% is added, c) Less heat generation during hydration, d) Increased resistance to sulfate attack, e) Increased pore refinement, f) Decreased permeability, g) Decreased water demand, h) Increased workability, i) Decreased cost.

1.8. Cautions while using fly ash: When using Class C or F fly ash as a portland cement replacement, it is important to know several precautions.

a) The time of set may be slightly delayed, and the early compressive strengths (before 28 days) may be decreased slightly.

b) The fine aggregate fraction of the concrete will need to be modified because fly ash has a lower bulk specific gravity than portland cement, and therefore occupies a greater volume for an equal mass.

c) If using any organic admixtures such as air entrainment, the amount added must be modified since the carbon (LOI) in the fly ash adsorbs organic compounds.

d) If the fly ash has high calcium content, it should not be used in hydraulic applications.

e) When using this or any other alternative cementing material with Portland cement, it is necessary to create trial mixtures to ensure proper proportioning for the desired properties.

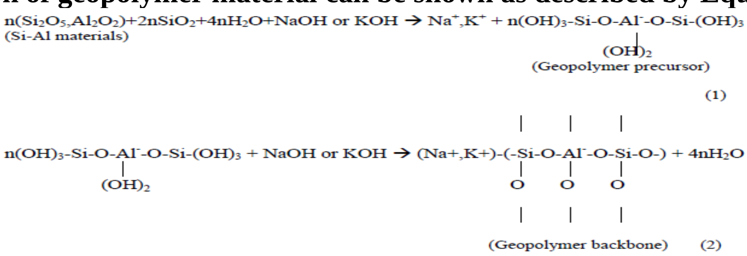
f) When using Class C fly ash as a portland cement replacement, it is important to know Class C fly ash must replace at least 25% of the portland cement to mitigate the effects of alkali silica reaction.

1.9. Chemical activation: The starting material and activating agent type and concentration are the most important parameters that influence the properties of the alkali-activated end product. The production of cement-free binders requires a starting material containing aluminum and silicon species that are soluble in highly alkaline solutions. The amount of vitreous silica and alumina present in the starting material plays a significant role in activation reactions and the properties of the reaction product.

Sodium hydroxide and sodium silicate are the more commonly used alkaline activating agents even though few studies have also been carried out with potassium hydroxide or sodium carbonate as the activating agent. The dissolution stage during alkali activation of alumino silicates is reported to be similar to that in the hydration of Portland cement at early ages. The dissolved species and the active surface groups in the starting material undergo polymerization to form the alkali-alumino silicate gel with the aluminum cationwhich hardens to form the binder matrix. $Mn[–(SiO_2)z–AlO_2]n–wH_2O$ is the general empirical formula proposed for the reaction product formed where M is the alkali ion, usually sodium or potassium, z is 1, 2, or 3, and n is the degree of polymerization.

2. EXPERIMENTAL

The schematic formation of geopolymer material can be shown as described by Equations (1) and (2)

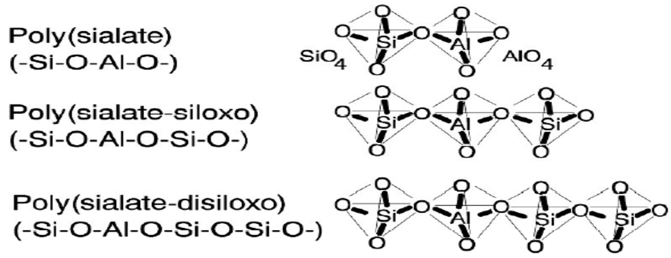


In the belowfigures, the exact mechanism of setting and hardening of the geopolymer material is not clear. However, most proposed mechanism consists the chemical reaction may comprise the following steps:

- Dissolution of Si and Al atoms from the source material through the action of hydroxide ions.
- Transportation or orientation or condensation of precursor ions into monomers.
- Setting or polycondensation/polymerisation of monomers into polymeric structures.

However, these three steps can overlap with each other and occur almost simultaneously, thus making it difficult to isolate and examine each of them separately. A geopolymer can take one of the three basic forms which are shown in figure.(Davidovits 1999).

- Poly (sialate), which has [-Si-O-Al-O-] as the repeating unit.
- Poly (sialate-siloxo), which has [-Si-O-Al-O-Si-O-] as the repeating unit.
- Poly (sialate-disiloxo), which has [-Si-O-Al-O-Si-O-Si-O-] as the repeating unit.



3. RESULTS AND DISCUSSION

3.1. Introduction: As per the experimental investigation 240 cube specimens are casted and tested. The properties of the materials used and the results attained by testing the specimens are presented and discussed in the following section.

3.2. Properties of fly ash: The fly ash used throughout the study is of class F grade and it is found out by the XRF test conducted on fly ash. Particle size distribution is found out for both normal and 45 micron sieved fly ash. Normal consistency for fly ash with different concentration of alkali is done.

3.3. Chemical composition: The chemical composition is done to find out the oxide content of fly ash. The chemical composition of normal fly ash and 45 micron sieved fly ash is given in the Table1. As per the ASTM 618, if the sum of the oxides of silicon, aluminium and ferrous content is greater than 70%, the sample is of Class F type. Hence, both the fly ash is of class F type. From the XRF results it is known that the sum of SiO_2, Al_2O_3 and Fe_2O_3 has decreased in 45 micron sieved fly ash. In which the change is notable in Al_2O_3 and lime content has increased up to 7%.

3.4. Physical properties: Physical properties such as specific gravity and surface area are tested and the results are tabulated in the Table 2.

Table.1.Chemical composition and index properties of fly ash

Tests	Normal Fly Ash	45 Micron Sieved Fly Ash
Chemical composition		
Silicon Dioxide (SiO_2), %	49.17	49.98
Aluminum Oxide (Al_2O_3), %	24.46	14.27
Iron Oxide (Fe_2O_3), %	6.04	6.70
Sum of SiO_2, Al_2O_3, Fe_2O_3, %	79.81	70.95
Calcium Oxide (CaO), %	11.41	18.22
Magnesium Oxide (MgO), %	5.02	1.24
Sulphur Trioxide (SO_3), %	1.67	4.41
Sodium Oxide (Na_2O), %	0.25	0.12
Potassium (K_2O), %	0.05	0.19
Chlorine (Cl), %	0.001	-
Index property		
Loss of Ignition, %	2.02	3.07

Table.2.Physical property of fly ash

Physical property	Normal fly ash
Specific Gravity	2.62
Blaine's Specific Surface Area (cm^2/gm)	4749

3.5. Particle size distribution: From the particle size distribution the range of materials in the particular diameter is found out from which the fineness can be calculated. The particle size distribution of normal fly ash and 45 micron sieved fly ash is represented in Fig 5.1.and Fig 5.2 respectively. From the particle size distribution the fineness of normal fly ash and 45 micron sieved fly ash is found out as 422.6m^2/kg and 750.3m^2/kg respectively. The mean diameter of the normal fly ash and 45 micron sieved fly ash is found as 21.35μm and 8.68μm respectively. The values of the particle size distribution are tabulated in Table 5.3 and Table 5.4.

Table.3.Particle size distribution of normal fly ash

Diameter of sieve(μm)	0.30	0.70	1.40	2.60	5.00	6.00	10.00	15.00
Cumulative %	1.33	4.36	7.98	14.63	24.33	27.64	38.57	48.90
Diameter of sieve(μm)	25.00	36.00	45.00	56.00	63.00	90.00	112.00	140.00
Cumulative %	64.39	78.61	87.21	94.00	96.63	99.90	100	100

Table.4.Particle size distribution of 45 micron sieved fly ash

Diameter of sieve(μm)	0.30	0.70	1.40	2.60	5.00	6.00	10.00	15.00
Cumulative %	2.57	9.55	18.88	34.71	55.33	61.09	74.78	81.23
Diameter of sieve(μm)	25.00	36.00	45.00	56.00	63.00	90.00	112.00	140.00
Cumulative %	89.29	96.57	99.12	99.93	100	100	100	100

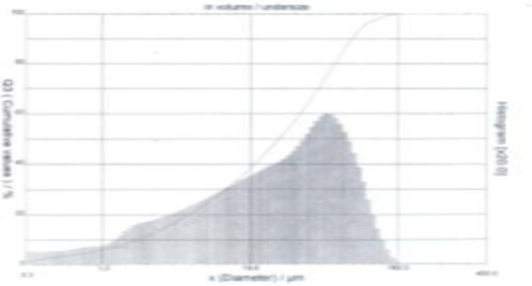


Fig.1.Particle size distribution of normal fly

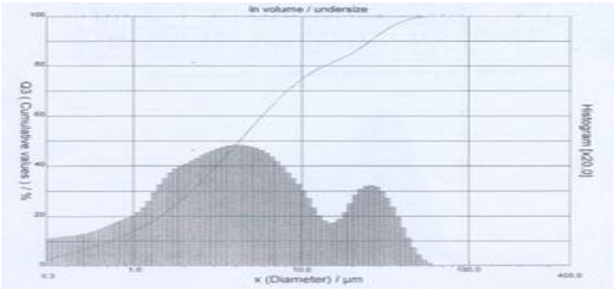


Fig.2.Particle size distribution of 45 micron sieved fly ash

From the particle size of both the fly ashes it is inferred that

- In 0.3μm 1.33 and 2.57 is cumulative% retained of normal and sieved fly ash respectively
- In 45 μm 87.21 and 99.12 is cumulative% retained of normal and sieved fly ash respectively
- In 63 μm 96.63 and 100 is cumulative% retained of normal and sieved fly ash respectively
- In 112 μm 100 and 100 is cumulative% retained of normal and sieved fly ash respectively.

3.6. Normal consistency: Normal consistency test is done on fly ash with different concentration of KOH solution and water with the help of vicat apparatus and their results are tabulated in Table 5 and Table 6. From the normal consistency conducted in the fly ash with water, it is known that the consistency value for fly ash is 52% of water by weight of fly ash and the consistency value for 4 molar solution is 60%, 6 molar solution is 59.25% and 8 molar solution is 57%. The consistency value decreases as the concentration of the solution increases.

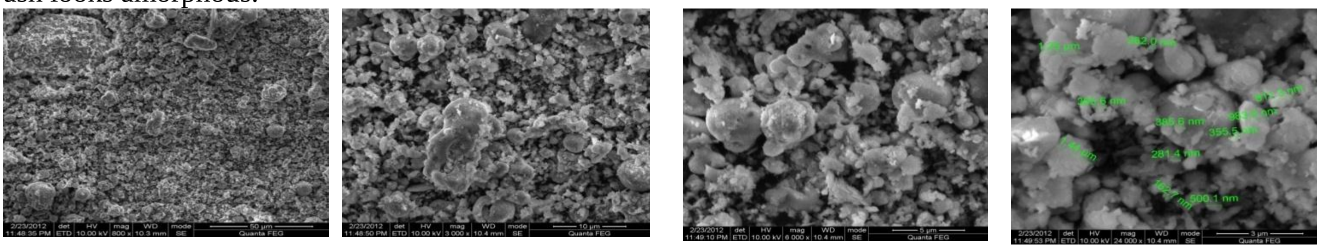
Table.5.Consistency test result of fly ash with varying concentration of KOH solution

Weight of Fly Ash (g)	Quantity of Soln. (%)	Weight of Soln. added (g)	Penetration of needle from bottom of mould (mm)		
			4 molar	6 molar	8 molar
400	42	168	38	35	38
400	44	176	38	34	38
400	46	184	37	34	37
400	48	192	34	32	36
400	50	200	32	30	34
400	52	208	31	29	33
400	54	216	30	26	30
400	56	224	29	24	26
400	57	228	28	22	6
400	58	232	26	18	-
400	59	236	25	11	-
400	59.25	237	21	7	-
400	59.5	238	15	-	-
400	59.75	239	10	-	-
400	60	240	5	-	-

Table.6.Consistency test result of fly ash with water

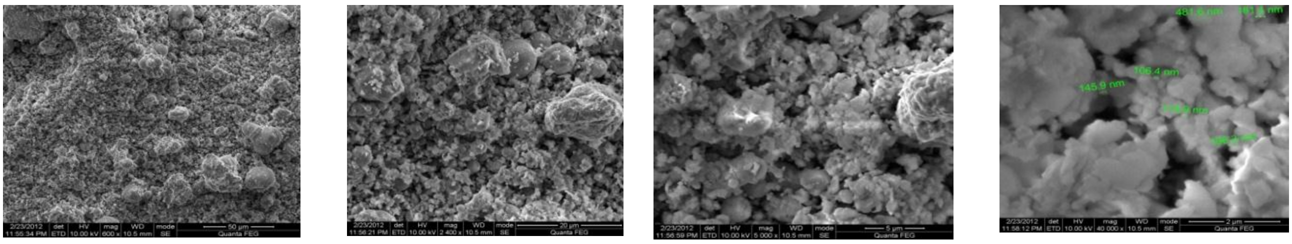
Weight of Fly Ash (g)	Quantity of water (%)	Weight of water added (g)	Penetration of needle from bottom of mould (mm)
400	40	160	38
400	42	168	34
400	44	176	32
400	46	184	26
400	48	192	21
400	50	200	11
400	52	208	4

3.7. Characterization study: Characterization study is done on both normal fly ash and fly ash passing through 45 micron sieve by using SEM analysis. Here the fly ash is magnified to different ranges to study their structure and size with the help of images. The SEM images of normal fly ash and fly ash passing through 45 micron sieve is shown in the Fig 3 and Fig 4 respectively. From the SEM images it has been inferred that the particle size of 45 micron sieved fly ash is smaller compared to the normal fly ash at different magnifications. Normal fly ash at magnification of 3µm has the smallest dimension of 281.4nm and that of 45 micron sieved fly ash has 106.4nm. In both the cases shape of the larger particles is more spherical than the smaller ones. The surface of both the fly ash looks amorphous.



a)Normal fly ash at 50µm b)Normal fly ash at 10µm c)Normal fly ash at 5µm d)Normal fly ash at 3µm

Fig.3.SEM images of normal fly ash at different magnification



a.45µ sieved fly ash at 50µm b.45µ sieved fly ash at 20µm c.45µ sieved fly ash at 5µm d.45µ sieved fly ash at 2µm

Fig.4.SEM images of fly ash passing through

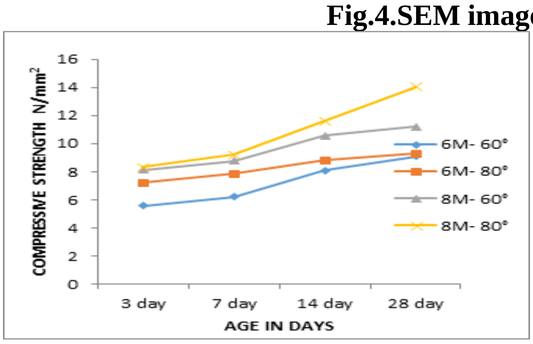


Fig.5.Compressive strength of steam cured specimen with normal fly ash

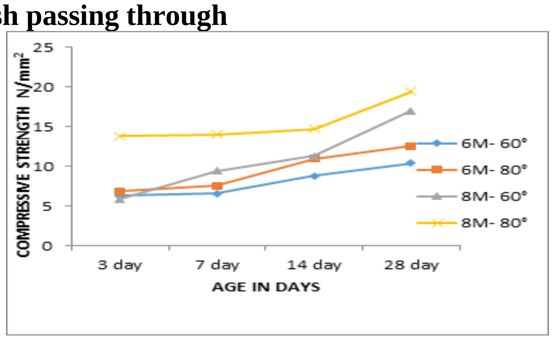


Fig.6.Compressive strength of steam cured specimen 45µ sieved fly ash

3.8. Compressive strength results of cubes: The compression strength results of the cubes casted are given in the following section. Compression strength results are studied and discussed based on the effect of molarity, temperature, curing method and particle size. The compressive strength of all the mixes at 3 day, 7 day, 14 day and 28 days with normal fly ash and 45 micron sieved fly ash is given in Table 7 and Table 8 respectively. From the compressive strength of steam cured specimen with normal fly ash and 45 micron sieved fly ash the specimen prepared with 8 molar solutions cured at 80ºC and 60ºCgives better result than the specimen with 6 molar solutions cured at 80ºC and 60ºC at 28 days.

3.9. Oven cured specimen: The compression test results of the cubes casted with normal fly ash and 45 micron sieved fly ash cured in oven at 60ºC and 80ºC with different concentration of KOH solution such as 6 molar and 8 molar are shown in Fig 7 and Fig 8 respectively.

Table.7.Compressive strength of cubes casted with Normal fly ash

Alkali Used	Molarity	Method of curing	Temperature in deg	Normal fly ash			
				3 day	7 day	14 day	28 day
KOH	6 molar	Oven	60	3.578	4.141	4.185	5.31
			80	3.841	4.13	4.432	5.841
		Steam	60	5.598	6.217	8.076	9.0715
			80	7.203	7.849	8.799	9.271
		Normal	27	3.11	3.524	3.965	4.213
	8 molar	Oven	60	5.321	6.913	8.871	10.032
			80	6.739	8.183	10.5335	13.075
		Steam	60	8.129	8.7825	10.548	11.215
			80	8.343	9.214	11.614	14.021
		Normal	27	5.201	5.562	7.345	9.321

Table.8.Compressive strength of cubes casted with 45 micron sieved fly ash

Alkali Used	Molarity	Method of curing	Temperature in deg	Normal fly ash			
				3 day	7 day	14 day	28 day
KOH	6 molar	Oven	60	5.986	7.5655	7.915	9.214
			80	4.57	10.108	10.8705	11.424
		Steam	60	6.2935	6.594	8.814	10.365
			80	6.826	7.5345	10.942	12.563
		Normal	27	4.657	5.6065	5.692	6.391
	8 molar	Oven	60	5.9535	8.057	9.586	13.009
			80	12.6705	13.726	14.152	15.42
		Steam	60	5.896	9.445	11.298	17.053
			80	13.783	13.9905	14.742	19.463
		Normal	27	7.058	7.145	8.75	9.953

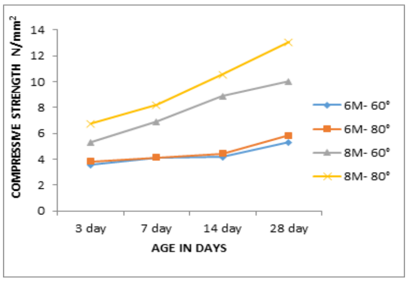


Fig.7.Compressive strength of oven cured specimen with normal fly ash

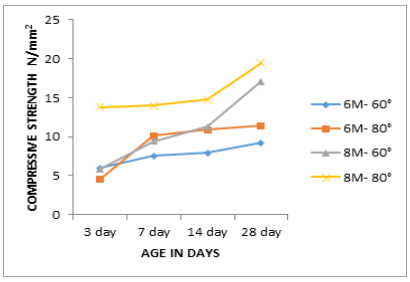


Fig.8.Compressive strength of oven cured specimen with 45µ sieved fly ash

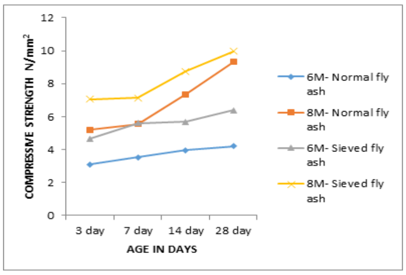


Fig.9.Compressive strength of normally cured specimen

From the compressive strength of oven cured specimen the cubes casted with 8 molar at 80ºC gives better result than 6M, 8M at 60ºC and 6M at 80ºC. The rate of increase of compressive strength is higher in 14 days to 28 days than from 7 days to 14 days.

3.10. Normally cured specimen: The compression test results of the cubes normally cured casted with normal fly ash and 45 micron sieved fly ash are shown in Fig 9.From the results given above it is observed that compressive strength increases as the concentration of the alkali increases. It is same in all the cases such as mortar cubes prepared with normal fly ash and fly ash passing through 45 micron sieve cured in different conditions such as steam curing, oven curing and normal curing. This might be due to the rate of dissolution of Si and Al in fly ash at higher concentration.

3.11. Effect of temperature on compressive strength: To find the effect of temperature on compressive strength, in steam curing and oven curing the cubes are cured at two different temperatures. The temperatures are 60ºC and 80ºC. The Fig 10 shows the compressive strength of cubes casted with normal fly ash and 45 micron sieved fly ash cured at steam curing and oven curing at different temperatures. From the compressive strength result based on the temperature it is observed that the compressive strength increases as the temperature increases. This might be due to the rate of reaction of fly ash at higher temperature. This is same in both the fly ash used and in different concentration of alkali.

3.12. Effect of curing method on compressive strength: To study the effect of curing method on the fly ash mortar cubes three different curing methods are adopted they are steam curing, oven cuing and normal curing. The following Fig 5.12 to 5.15 show the compressive strength of cubes casted with both the fly ashes and three different curing methods with 6 molar concentration of KOH at different periods such as 3rd day, 7th day, 14th day and 28th day. Figures 15, 16, 17 shows the compressive strength of cubes casted with both the fly ashes and three different curing methods with 8 molar concentration of KOH at different periods.

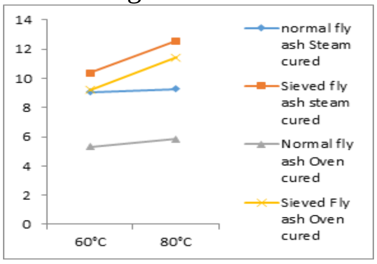


Fig 10 Compressive strength of cubes casted with 6M solution steam and oven cured at 60° and 80°C

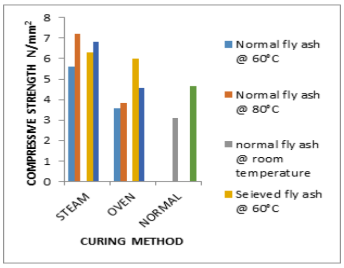


Fig 11 Compressive strength of cubes with NFA, SFA and 6M solution at 3rd day

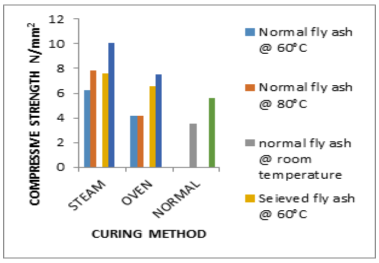


Fig 12 Compressive strength of cubes with NFA, SFA and 6M solution at 7th day

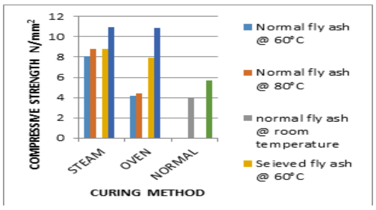


Fig 13 Compressive strength of cubes with NFA, SFA and 6M solution at 14th day

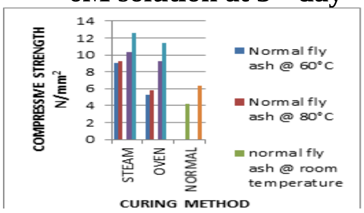


Fig 14 Compressive strength of cubes with NFA, SFA and 6M solution at 28th day

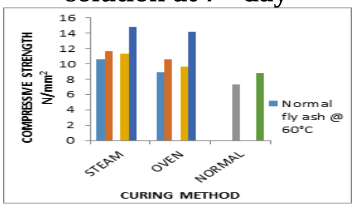


Fig 15 Compressive strength of cubes with NFA, SFA and 8M solution at 7th day

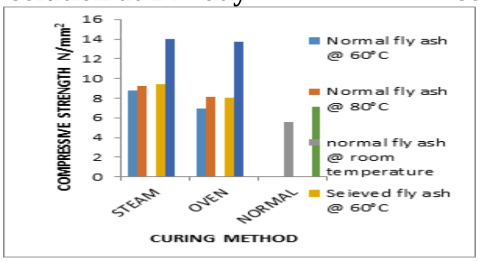


Fig 16 Compressive strength of cubes with NFA, SFA and 8M solution at 14th day

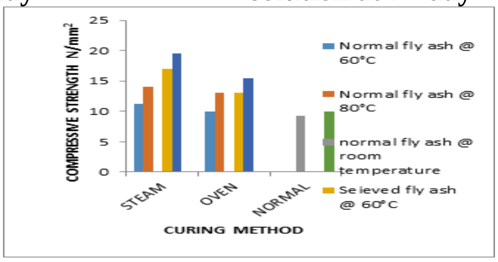


Fig 17 Compressive strength of cubes with NFA, SFA and 8M solution at 28th day

From the results of compressive strength based on curing methods at different periods the following observations are made. Steam curing gives higher strength compared to oven curing and in turn oven curing gives higher compressive strength than normal curing for all the mixes with different molarities at different periods. The mixes containing 8M the difference of compressive strength is high for cubes cured at 80°C than cubes cured at 60°C, but in the case of mixes with 6M the difference is not much higher. The compressive strength is higher for 45 micron sieved fly ash compared to normal fly ash at all the curing methods adopted.

3.13. Effect of particle size on compressive strength: To observe the effect of particle size on compressive strength in the study normal fly ash and fly ash passing through 45 micron sieved is used. From the above given results and the figures shown it is evident that the particle size have an inverse effect on compressive strength. As the particle size increases the compressive strength decreases in all the cases discussed such as at different concentration of alkali, different curing conditions and different curing temperatures. This might be due to the presence of smaller particles, which will fill the voids.

4. CONCLUSION

From the compressive strength tests conducted on mortar cubes prepared with fly ash and alkali it is observed that

- The increase in the concentration of the alkali increases the compressive strength. This may be due to the rate of dissolution of Si and Al in the fly ash at higher molarity of KOH.
- By increasing the curing temperatures in oven curing and steam curing the compressive strength has increased. This might be due to the rate of reaction of fly ash at higher temperatures.
- The oven curing gives higher compressive strength than normal curing and steam curing gives higher strength than oven curing.
- The decrease in the particle size increases the compressive strength. This might be due to the increase of smaller particles in fly ash would decrease the amount of pores.
- From the characterization study on normal fly ash and 45 micron sieved fly ash
- It has been inferred that the particle size of 45 micron sieved fly ash is smaller compared to the normal fly ash at different magnifications.
- In both the cases shape of the larger particles is more spherical than the smaller ones.

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