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Development of Flyash-Based Geopolymer Mix using Parametric Analysis

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Abstract**: *In the present scenario, geopolymer concrete creates an attention in the construction industry due to its ecofriendly nature. The enhanced property of geopolymer makes it as a promising alternative material for the development of infrastructure. Even though geopolymer has improved properties when compared to normal cement concrete, its mix design is not yet been a standardized procedure to be followed for precast works. Several factors such as liquid to fly ash ratio, silicate to hydroxide ratio, concentration of NaOH (Molarity), dosage of super plasticizer, curing temperature, rest period prior to curing and curing time affects the strength while designing the mix. This paper details a parametric analysis to develop geopolymer mix by considering the above seven factors. Test results proved that geopolymer mix prepared with liquid to fly ash ratio of 0.4, silicate to hydroxide ratio of 2.5 and 10 M of NaOH cured in hot air oven at 80 ºC for 6 hours with 1% super plasticizer showed enhanced compressive strength of 27.20 MPa.***

Index Terms**: *super plasticizer, curing temperature, rest period.***

I. INTRODUCTION

Climate change, resource productivity and industrial ecology are the three major sustainability issues which should be considered to meet the infrastructural needs. The global energy use is divided into several sectors such as building services, industry, transport, building construction and others in which the energy consumed for building services was found to be at a highest rate of 45%. Energy consumption may lead to hazardous environment and hence it is very important to reduce the energy used by the buildings in order to provide safe surroundings. Currently our environment is under a great pressure, as global cement industries are the one that contribute to greenhouse gas emission which is likely to increase the CO_2 emission into the atmosphere by about 50% in 2020 from the present [1]. Among all greenhouse gases, about 65% of global warming is caused due to CO_2 and, SO_3 and NO_x further cause greenhouse effect and acid rain [2]. Geopolymer is an alternative technique which has a great potential to replace ordinary Portland cement in concrete. Geopolymerization is the process of formation of three dimensional tetrahedral structures by the dissolution of solid aluminosilicate under a high alkalinity aqueous solution through polymerization mechanism. Under high alkaline condition, the dissolution of alumina and silica elements of aluminosilicate material occurs which releases AlO_4 and SiO_3 ions. Then as a simultaneous process polycondensation, gelation and further condensation forms amorphous gel which has a great power to act as a binder [3].Raw materials which are rich in silica and alumina, such as, fly ash, metakaolin, red mud, and ground granulated blast furnace slag are used to produce geopolymer. When the raw material is mixed with sodium or potassium-based alkaline liquids it reduces the embodied CO_2 up to 80% [4,5]. In 1979, Fly ash-based geopolymer concrete was first introduced by Davidovits in which industrial waste fly ash totally replaces cement in concrete [6]. Flyash, a waste industrial by-product that poses a serious problem of disposal. Its usage in geopolymer leads to the development of sustainable concrete and its annual production is about 75-80% among the total ash production [7,8]. High quality geopolymer concrete of about 2.5m^3 is produced using 1 tonne of low calcium fly ash as there is a risk of quick set while using high calcium fly ash [9,10]. Alkaline activators play an important role in the process of dissolution of Si and Al oxides [11]. Hence for economical purpose, sodium-based silicate and hydroxide solution is used along with fly ash in order to achieve adequate strength through geopolymerization process. There are some factors that influence the strength parameter of geopolymer concrete such as fly ash reactivity, liquid to fly ash ratio, silicate to hydroxide ratio, dosage of super plasticizer, nature and concentration of activator solution, curing temperature, curing time, curing type, rest period and handling time. This paper focus on the experimental study to optimize various parameters to obtain higher compressive strength of fly ash-based geopolymer mortar. Here the most important seven factors such as liquid to fly ash ratio, silicate to hydroxide ratio, concentration of NaOH (Molarity), dosage of super plasticizer, curing temperature, rest period prior to curing and curing time are considered for the prediction of compressive strength.

II. EXPERIMENT

A. Materials Used

In geopolymer mix, flyash, an ash generated while burning coal in thermal power station is used instead of cement in concrete. There are two types of flyash namely Class F and Class C according to ASTM C 618 [12]. In this research work, Class F-type flyash is used as the main source material. The chemical composition of the flyash used here is presented in Table 1 and the SEM image of flyash is shown in Figure 1. Normal river sand passing through 2.36 mm sieve is used as the filler material. A combination of sodium hydroxide prepared at a required Molarity is mixed along with sodium silicate is used as alkaline solution instead of water in concrete.

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These solutions are prepared one day before in order to activate the process of polymerization as it reduces bleeding and segregation.

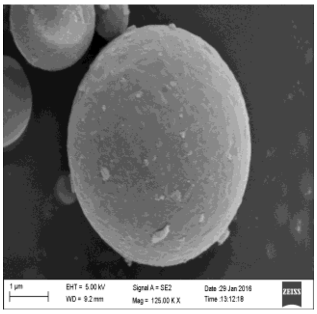

Figure 1: SEM image of Flyash [13]

Table 1: Oxide Compositions of Class F Flyash using XRF

Chemical	Component (wt %)
SiO_2	52.15
Al_2O_3	27.71
Fe_2O_3	5.09
CaO	0.51
MgO	1.01
TiO_2	3.94
K_2O	1.46
Na_2O	0.27
CuO	0.24
LOI	6.80
Total	99.18

B. Preparation of Test Specimens

As a first process, flyash and sand are mixed for 4 minutes in the pan mixer continuously. After thorough mixing of the dry materials, alkaline liquids along with the required amount of superplasticizer are added to continue the process of wet mixing for about 3 minutes. The total amount of fresh geopolymer mortar mix is divided into three equal parts and is filled in 70.6 mm x 70.6 mm x 70.6 mm mortar cube. Proper compaction is given using tamping rod and vibrating table in order to improve its strength. The fresh mix in the mortar cube is covered using plastic sheets to prevent the water evaporation from the mix. For curing the specimen, the mix along with the steel mould is placed inside the hot air oven for a particular time period. Then the specimens are allowed to cool inside the oven and tested for its strength. It is found that several factors affect the strength of the geopolymer. As liquid to fly ash ratio and silicate to hydroxide ratio are the two main factors that affects the mix proportion severely, it has been considered as variation factor in the first stage of the trial. From the Table 2, it has been found that about 20 trial mixes were tried to find the correct proportion for geopolymer.

The average of three specimens are considered to be the compressive strength of geopolymer mortar according to IS 456-2000[14]. In the second stage, further 9 trial mixes were carried out as shown in Table 3, by varying other factors such as Molarity of NaOH, dosage of super plasticizer, curing temperature and rest period which may automatically increases the strength of the mix. As a third stage, 8 trials were carried out only by varying the curing period for the purpose of energy conservation which is shown in Table 4.

The SEM micrograph of the final geopolymer mortar specimen is taken using SEM SU1510 model at a working distance of 6.5 mm is shown in Figure 2. The image implies that there are only some micro cracks and spherical shaped fly ash particle get partially dissolved to create small sized pores in the matrix. Some fly ash particles are present as unreacted in hollow cavities due to the space left behind by dissolved fly ash particle.

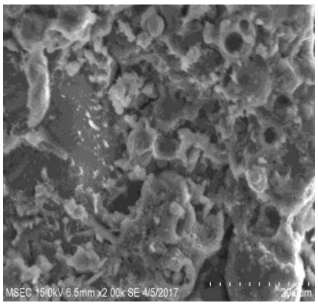

Figure 2: SEM image of fly ash-based geopolymer mortar

III. RESULTS AND DISCUSSION

A. Liquid to fly ash ratio and Silicate to hydroxide ratio

Alkaline liquid is the combination of sodium hydroxide and sodium silicate. There is no use in the addition of alkaline liquid separately into the mix, as NaOH activates sodium silicate solution to get involve in polymerization process.

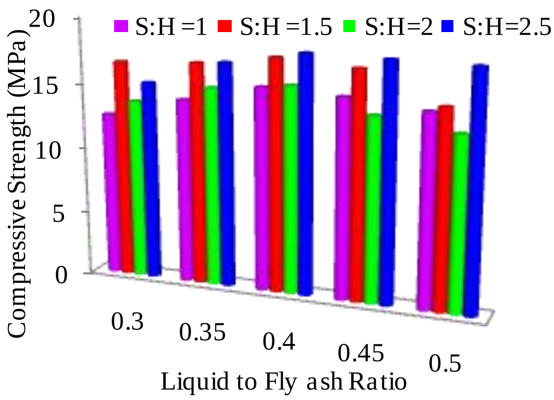

Figure 3: Variation of compressive strength with liquid to fly ash ratio and silicate to hydroxide ratio

Table 2: Trial Mix Proportion of Geopolymer Mortar by varying liquid to fly ash ratio and silicate to hydroxide ratio

S.No	Mix ID	Fly ash (gm)	NaOH		Na_2SiO_3 (gm)	Sand (gm)	Liquid to Flyash Ratio	Super Plasticizer (%)	Silicate to Hydroxide ratio	Curing Temperature (ºC)	Rest period (Hours)	Curing Time (Hours)
			Mass (gm)	Molarity (M)								
1.	LS1	256.44	38.47	8	38.47	300.00	0.30	1	1	60	24	24
2.	LS2	246.47	44.36	8	44.36	298.00	0.35	1	1	60	24	24
3.	LS3	238.13	47.63	8	47.63	300.00	0.40	1	1	60	24	24
4.	LS4	231.18	50.86	8	50.86	300.00	0.45	1	1	60	24	24
5.	LS5	222.25	55.56	8	55.56	300.00	0.50	1	1	60	24	24
6.	LS6	256.44	30.77	8	46.16	300.00	0.30	1	1.5	60	24	24
7.	LS7	247.43	34.64	8	51.96	299.00	0.35	1	1.5	60	24	24
8.	LS8	238.13	38.10	8	57.15	300.00	0.40	1	1.5	60	24	24
9.	LS9	230.33	41.46	8	62.19	299.00	0.45	1	1.5	60	24	24
10.	LS10	222.25	44.45	8	66.68	300.00	0.50	1	1.5	60	24	24
11.	LS11	256.44	25.64	8	51.29	300.00	0.30	1	2	60	24	24
12.	LS12	247.43	29.69	8	56.91	299.00	0.35	1	2	60	24	24
13.	LS13	238.13	30.96	8	64.30	300.00	0.40	1	2	60	24	24
14.	LS14	230.33	34.55	8	69.10	299.00	0.45	1	2	60	24	24
15.	LS15	222.25	37.78	8	73.34	300.00	0.50	1	2	60	24	24
16.	LS16	256.86	22.09	8	53.94	300.00	0.30	1	2.5	60	24	24
17.	LS17	247.43	24.74	8	61.86	299.00	0.35	1	2.5	60	24	24
18.	LS18	238.13	27.15	8	68.11	300.00	0.40	1	2.5	60	24	24
19.	LS19	230.33	29.71	8	73.71	299.00	0.45	1	2.5	60	24	24
20.	LS20	222.00	31.75	8	79.92	300.00	0.50	1	2.5	60	24	24

Table 3: Trial Mix Proportion of Geopolymer Mortar by varying Molarity of NaOH, dosage of superplasticizer, curing temperature and rest period

S.No	Mix ID	Flyash (gm)	NaOH		Na_2SiO_3 (gm)	Sand (gm)	Liquid to Flyash Ratio	Super Plasticizer (%)	Silicate to Hydroxide ratio	Curing Temperature (ºC)	Rest period (Hours)	Curing Time (Hours)
			Mass (gm)	Molarity (M)								
1.	GM1	238.00	27.00	10	68.00	300.00	0.40	1	2.5	60	24	24
2.	GM2	238.00	27.00	12	68.00	300.00	0.40	1	2.5	60	24	24
3.	GS1	238.00	27.00	10	68.00	300.00	0.40	1.5	2.5	60	24	24
4.	GS2	238.00	27.00	10	68.00	300.00	0.40	2.0	2.5	60	24	24
5.	GT1	238.00	27.00	10	68.00	300.00	0.40	1	2.5	80	24	24
6.	GT2	238.00	27.00	10	68.00	300.00	0.40	1	2.5	100	24	24
7.	GR1	238.00	27.00	10	68.00	300.00	0.40	1	2.5	80	0	24
8.	GR2	238.00	27.00	10	68.00	300.00	0.40	1	2.5	80	12	24
9.	GR3	238.00	27.00	10	68.00	300.00	0.40	1	2.5	80	48	24

Table 4: Trial Mix Proportion of Geopolymer Mortar by varying curing period

S.No	Mix ID	Flyash (gm)	NaOH		Na_2SiO_3 (gm)	Sand (gm)	Liquid to Flyash Ratio	Super Plasticizer (%)	Silicate to Hydroxide ratio	Curing Temperature (ºC)	Rest period (Hours)	Curing Time (Hours)
			Mass (gm)	Molarity (M)								
1.	GC1	238.00	27.00	10	68.00	300.00	0.40	1	2.5	80	24	1
2.	GC2	238.00	27.00	10	68.00	300.00	0.40	1	2.5	80	24	3
3.	GC3	238.00	27.00	10	68.00	300.00	0.40	1	2.5	80	24	6
4.	GC4	238.00	27.00	10	68.00	300.00	0.40	1	2.5	80	24	9
5.	GC5	238.00	27.00	10	68.00	300.00	0.40	1	2.5	80	24	12
6.	GC6	238.00	27.00	10	68.00	300.00	0.40	1	2.5	80	24	15
7.	GC7	238.00	27.00	10	68.00	300.00	0.40	1	2.5	80	24	18
8.	GC8	238.00	27.00	10	68.00	300.00	0.40	1	2.5	80	24	21

Liquid to fly ash ratio and the ratio of silicate to hydroxide plays a vital role in strength prediction as the liquid stimulates fly ash to undergo polymerization reaction at higher rate. Normally the range of liquid to fly ash and silicate to hydroxide ratio are taken from 0.3 to 0.5 at an interval of 0.05 and the later from 1 to 4 at an interval of 1.5.

The strength value goes on increasing from 0.3 to 0.4 and there is a decrease in the strength beyond 0.4. This is mainly due to the fact that more amount of alkaline solution decrease the reaction rate of fly ash. From the Figure 3, it is well understood that the higher strength value of 18.16 MPa has been obtained only while taking the liquid to fly ash ratio as 0.4 and liquids at a ratio of 2.5.

B. Concentration of NaOH

Concentration of sodium hydroxide is an important parameter that influences the compressive strength of geopolymer. From the Figure 4, it is viewed that increase in the concentration of NaOH will increase the strength of geopolymer in the range of 3.69% and 6.50% when compared to 8M. This is mainly due to the fact that increase in NaOH concentration increases the total solid content of NaOH in the solution thereby it reduces the water content which increases the strength of geopolymer. For economical purpose, it is suggested to use 10M concentration of NaOH throughout the process.

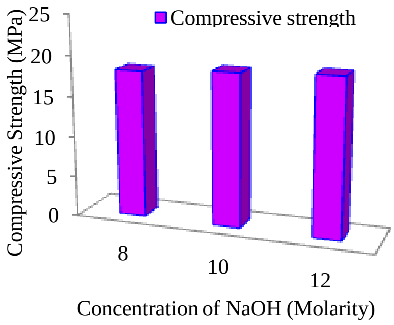

Figure: 4 Variation of compressive strength with the concentration of NaOH

C. Dosage of Super Plasticizer

Addition of super plasticizer improves the workability of the geopolymer mix without increasing the water content. Even though, it improves the workability there is limiting percentage in its addition. Here commercially available super plasticizer (Conplast SP430) was used and it is added at a range from 1 to 2 at an interval of 0.5. Figure 5 clearly shows that there is a continuous increase in strength with the addition of more amount of super plasticizer.

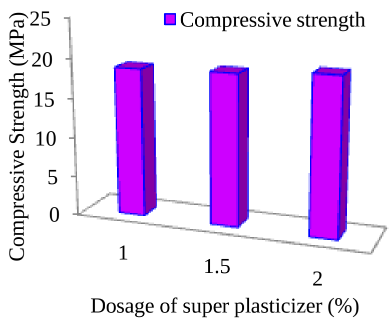

Figure: 5 Variation of compressive strength with super plasticizer

But normally chemical admixtures are added only at a lower amount. Hence it is recommended to use 1% super plasticizer by weight of fly ash for the entire process as it gives a required strength of 18.83MPa.

D. Curing Temperature

As geopolymer gains its strength only through polymerization process when subjected to heat, curing temperature is considered as an important factor. Figure 6 shows that the increase in temperature substantially increases the strength in the range of 6.27% and 9.67% when compared to the specimens subjected to 60ºC. For the purpose of energy consideration it is better to cure the specimens below 100ºC. Hence the specimens are cured at 80ºC in hot air oven for the purpose of strength gaining.

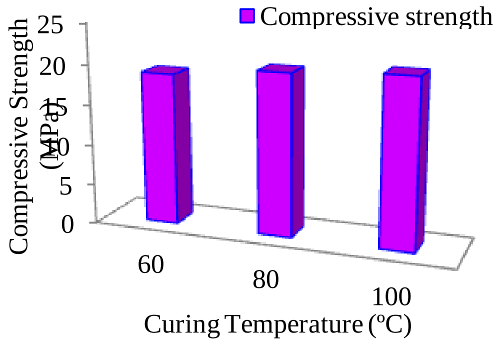

Figure: 6 Variation of compressive strength of geopolymer mortar subjected to different curing temperature

E. Rest Period

Rest period is the time interval between the completion of casting of geopolymer specimens to the start of the curing process. Here the specimens are left for about 0, 12, 24 and 48 hours in the room temperature after casting. Figure 7 obviously shows the increase in strength is obtained in the range of 7.78%, 28.68% and 63.47% and it is found that allowing the specimens to a rest period of 24 hours gives an optimum result with more strength and less time consumption when compared to all other periods.

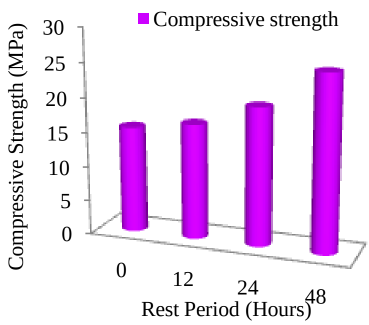

Figure: 7 Variation of compressive strength after subjecting to various rest periods

This is mainly due to the fact that geopolymer do not get hardened quickly after casting and it requires about 1day for setting without any nail impression.

F. Curing Time

It is the time taken by fresh geopolymer mix to harden and to gain strength through heating process. Here the specimens are placed separately from 1 to 24 hours at an interval of 2 hours. From the Figure 8, it has been found that increase in compressive strength of about 35.93% is achieved while curing the specimens at 6 hours when compared to 24 hours. Beyond 6 hours, there is a slight decrease in the strength as the process of geopolymerization is completed at a period of 6 hours itself.

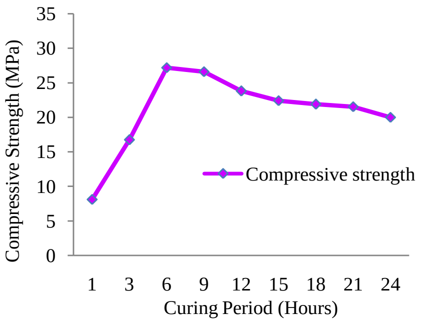

Figure: 8 Variation of compressive strength at various curing periods

IV CONCLUSION

From the experiment, the following conclusions can be drawn:

- Increase in liquid to fly ash ratio and silicate to hydroxide ratio; spontaneously increases the compressive strength of fly ash-based geopolymer mortar. Hence it is recommended to use the former as 0.4 and the latter as 2.5 to achieve higher strength value of about 18.16MPa through adequate polymerization reaction.
- In the process of mix design, increasing the concentration of NaOH will automatically increase the compressive strength of geopolymer mortar. In economical point of view, it is suggested to use 10M concentration of NaOH which gives higher strength of about 3.69% when compared to 8M concentration.
- As chemical admixture should be added only at a minimum rate to enhance the performance of the mix. It is recommended to use 1% of super plasticizer along with the alkaline liquid to achieve required strength of 18.83MPa.
- When the curing temperature is raised from 60°C to 100°C, the strength is also increased from 0 to 9.67%. For energy efficient purpose, the specimens are cured at an optimum temperature of 80°C in the hot air oven to gain strength of about 6.27%.
- The samples are allowed to rest in the ambient temperature without any disturbances for 24 hours to obtain strength of about 28.68% when compared to the sample subjected to immediate curing after casting process.

While subjecting the specimen to a curing period of 1-24 hours at an interval of 3 hours, sufficient strength of 35.93% has been attained when the samples are cured for 6 hours instead of 24 hours.

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