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Development of mix design method for geopolymer concrete

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Abstract. This study proposes a mix design method for geopolymer concrete (GPC) using low calcium fly ash and alccofine, with the focus on achieving the required compressive strength and workability at heat and ambient curing. Key factors identified and nine mixes with varied fly ash content (350, 375 and 400 kg/m^3) and different molarity (8, 12 and 16M) of NaOH solutions were prepared. The cubes prepared were cured at different temperatures (27°C, 60°C and 90°C) and tested for its compressive strength after 3, 7 and 28 days of curing. Fly ash content has been considered as the direct measure of workability and strength. The suggested mix design approach has been verified with the help of the example and targets well the requirements of fresh and hardened concrete.

Keywords: geopolymer concrete; low calcium fly ash; alccofine; mix design; AF-GPC graphs; alkaline activator liquid

1. Introduction

The conservation of natural resources and minimization of the environment depletion are the topic of attention among researchers, which led to looking at alternative sustainable construction materials. Globally, the role of the ordinary Portland cement based (OPC) concrete is second only to water, with the cement usage being 4.0 billion tons per annum with a growth rate of 4% per annum (Jewell *et al.* 2014). The enormous usage of OPC based concrete is challenged with environmental and power related issues (Davidovits 1994, Aïtcin 2000, Worrell *et al.* 2001, Gartner 2004, Duxson *et al.* 2007, Kayali *et al.* 2008). The production of Portland cement requires highly energy for production and more significantly it releases one tonne volume of carbon dioxide for one tonne production of cement (Malhotra 1999, McCaffrey 2002). Nowadays, industrial by-products such as ground granulated blast furnace slag, mine waste, fly ash, red mud, rice husk ash etc, and construction waste has become a big problem (Saha *et al.* 2016, Solanki *et al.* 2016), it requires large areas of useful land for disposal and has a huge impact on the environment. Geopolymer concrete is paving a path of better alternative construction material in terms of lowering the greenhouse gases, as it can reduce the CO_2 by 80% which is caused by cement industries itself only (Gartner 2004). GPC is hoped to be a sustainable material which may serve as a better alternative to OPC concrete by effectively utilizing industrial wastes such

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as fly ash, slag, rice husk ash etc. (McLellan *et al.* 2011, Parveen *et al.* 2013, Slaty *et al.* 2015, Jindal *et al.* 2017) and maintaining environmental standards.

Enough literature is available which highlights higher compressive strength of GPC in shorter curing period than conventional concrete (Davidovits 1994, Aïtcin 2000, Worrell *et al.* 2001, Gartner 2004, Duxson *et al.* 2007, Kayali *et al.* 2008). The higher compressive strength in shorter curing period has been obtained at higher curing temperature in presence of fly ash (Amol *et al.* 2014). The results are not encouraging at ambient temperature. The structural behaviour of GPC and conventional concrete is same (Raj *et al.* 2016, Hardjito 2005). The literature which is available on the mix design procedure is based on heat curing and has been discussed in the second part. The heat curing limits the use of GPC in the cast in situ practices, although it can be used for precast industry. Further, workability and strength have not been targeted at the same time. So, a mix design method is required which admires required workability and target mean strength at ambient as well as elevated heat curing. Alccofine, patented by counto microfine private limited along with the Ambuja cement private limited, available all over the globe, have been incorporated to achieve the strength and workability at ambient curing. This paper presents a mix design method by targeting workability and strength for GPC based on low calcium fly ash. However, alccofine has also been added to attain the required compressive strength at ambient temperature and extends the applicability of geopolymer concrete beyond precast concrete industry.

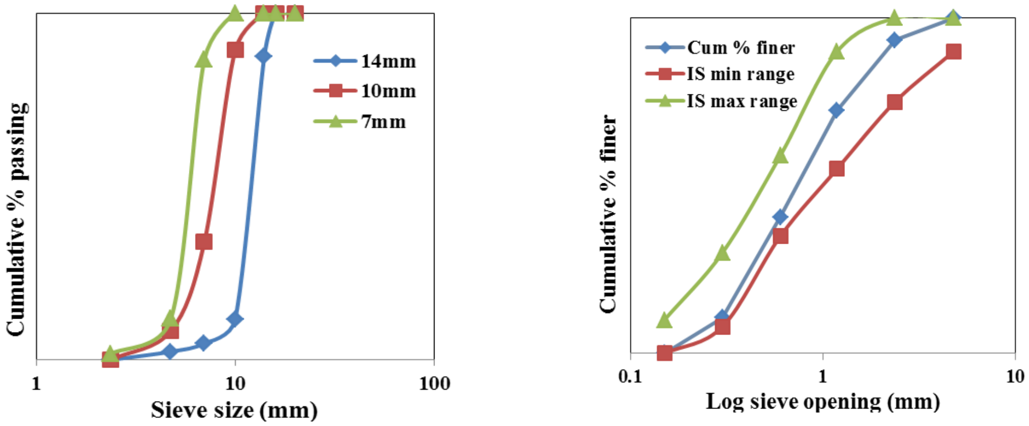
2. Review of mix design methods and their limitations

Lloyd *et al.* (2010) were the first one among the various researchers who proposed mix design methodologies for GPC. In this method, total aggregates content was fixed at 80% and the density of GPC has been assumed as 2400 kg/m^3. The total mass of fly ash and alkaline activator solution was obtained by deducting the total aggregates content from the assumed density of 2400 kg/m^3. Mix proportions were, however, determined without targeting workability and compressive strength.

Anuradha *et al.* (2012) suggested a design procedure for different grades of GPC by using Indian standards developed for conventional concrete. In this method, fly ash content and ratio of activator liquid to fly ash was selected based on the required compressive strength. The fine aggregate percentage was kept constant and correction to fine aggregate percentage was performed based on its zone. Later it was observed that the activator solution content employed is in excess for the corresponding strength reported and even the targeted strength was not achieved.

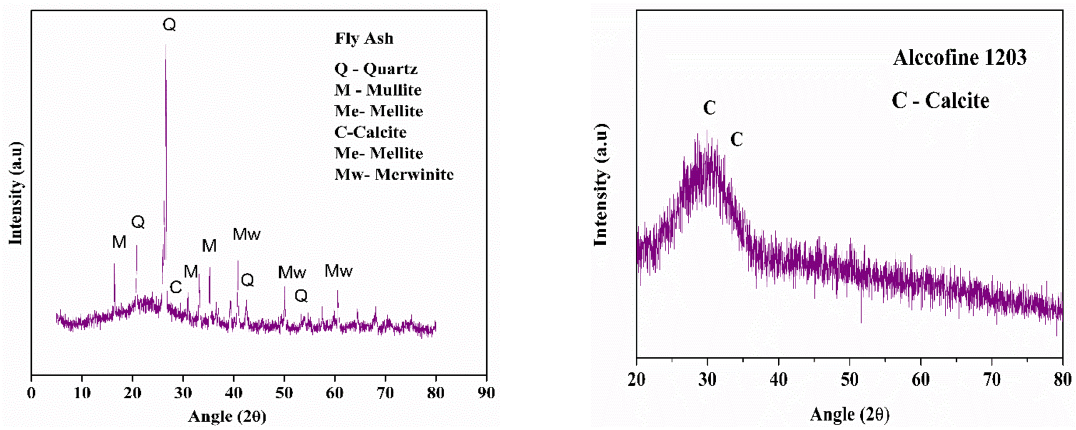
Ferdous *et al.* (2013) suggested a mix design by using the several parameters like specific gravity, workability, air content and concrete density as a variable. However, it was difficult to determine the exact activator solution content with respect to the fly ash by using their design procedure. Further, selection of the alkaline activator to fly ash ratio was another significant issue.

Junaid *et al.* (2015) proposed a mix design approach for low calcium alkali activated fly ash based GPC. In this method, various plots are given for selecting the variables affecting the properties of the GPC and marvellous efforts have been done to achieve compressive strength and workability. Since fly ash concrete requires high curing temperature to achieve required compressive strength and water to geoploymer solid ratio cannot be sole criteria to target compressive strength and workability the method had these limitations. Further, fly ash content was randomly assumed to target the required strength and the same has been focused in the current study along with different curing temperatures and NaOH molarity.



(a) Coarse aggregate (b) Fine aggregate

Fig. 1 Grading curve of (a) Coarse aggregate (b) Fine aggregate



(a) Fly Ash (b) Alccofine 1203

Fig. 2 XRD spectrum of Fly Ash and Alccofine 1203

Pavithra *et al.* (2016) made efforts to develop a mix design methodology for GPC with the main focus on achieving better compressive strength in an economical way for different alkaline solutions to binder proportions. To propose a rational mix design method for GPC; correlation between the alkaline activator solution to binder ratios and 28 day compressive strength had been investigated. This method included different AAS to FA ratios (ranging from 0.4 to 0.8) and a maximum strength of 54MPa had been achieved. Like other methods it did not consider the ambient curing and NaOH molarity was fixed to 16M. Further, in this method ACI and Indian standards had been used which intermingled the properties of materials to be considered.

Hardjito *et al.* (2005) studied the effects of parameters affecting the strength and workability of the GPC and it had been concluded that as the curing temperature increased, the compressive strength of fly ash based GPC also increases. Further, addition of plasticizer improved the workability (Hardjito 2005, Rangan *et al.* 2005). The variables considered in this research were plasticizer amount, aggregate size, NaOH concentration etc. but a systematic approach was not done which can be used for practical usage.

Table 1 Aggregate properties

Property	Fine Aggregates	Coarse Aggregates
Specific Gravity	2.32	2.60
Fineness Modulus	2.82	7.10
Water Absorption	1.50%	0.80%

Table 2 Chemical composition and physical properties of processed fly ash

Sample	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	SO_3(%)	CaO(%)	Na_2O (%)	LOI (%)	Specific surface area m^2/kg
Fly ash	62.55	29.02	4.22	0.22	1.1	0.20	0.52	335.7
Requirement as Per IS:3812-2003	70% min. by mass			3% max by mass	---	1.5% max by mass	5	320.0

Table 3 Chemical composition & physical properties of Alccofine 1203

Chemical Composition		Physical Properties	
Constituents	Composition (%)	Physical Property	Results
SiO_2	35.30	Particle Size Distribution (micro metre)	
MgO	6.20	d10	1.8
Al_2O_3	21.40	d50	4.4
		d90	8.9
Fe_2O_3	1.20	Bulk Density (kg/m^3)	680
SO_3	0.13	Specific Gravity	2.70
CaO	32.20	Specific surface area (m^2/kg)	1200

To make the GPC more economical than OPC based concrete, use of alkaline solution must be minimized by keeping in mind the design strength and required workability as alkaline solution is the costliest ingredient in the GPC. It can be concluded from the above available literature on the design and development of GPC, that there seems to be no specific procedure which considers all the essential parameters such as workability, compressive strength, ambient curing temperature. Therefore, in this method an attempt has been made to propose a mix design procedure which considers the aforesaid drawbacks of the earlier proposed methods. However, in this research major emphasis has been kept to focus on developing and presenting, a systematic approach for GPC mix design by targeting desired strength and workability at ambient and heat curing.

3. Material properties

Fine and coarse aggregates locally available were tested and confirmed the requirements as per Indian Standards (BIS 2386-1963, BIS 383-1970). The results of the aggregates are given in Table 1 and grading curve are shown in Fig. 1. Sodium hydroxide in the form of pellets with 98% purity and sodium silicate solution (Na_2SiO_3) with SiO_2/Na_2O between 1.90 and 2.01 were procured commercially. The NaOH solutions were prepared using solid (pallets or flakes) to liquid (distilled water) proportions identified as 263 g, 361 g and 444 g of NaOH solid flakes in 1 kg of solution for 8, 12 and 16 M respectively (Hardjito *et al.* 2004). Local available siliceous fly ash (FA)

Table 4 Mix proportions used in this study

Mixture	F_A	C_A	FA	M: NaOH	TAS	NaOH	Na_2SiO_3	Extra water	AF	SP	Curing Temp. (^oC)
M1FAGC	533	1243	350	8	157.5	45.00	112.5	24.3	35.0	7.0	27/60/90
M2FAGC	521	1215	375	8	168.7	48.21	120.5	26.2	37.5	7.5	27/60/90
M3FAGC	508	1186	400	8	180.0	51.42	128.5	27.9	40.0	8.0	27/60/90
M4FAGC	531	1239	350	12	157.5	45.00	112.5	30.0	35.0	7.0	27/60/90
M5FAGC	519	1210	375	12	168.7	48.21	120.5	32.3	37.5	7.5	27/60/90
M6FAGC	506	1181	400	12	180.0	51.42	128.5	34.3	40.0	8.0	27/60/90
M7FAGC	530	1236	350	16	157.5	45.00	112.5	34.8	35.0	7.0	27/60/90
M8FAGC	517	1207	375	16	168.7	48.21	120.5	37.3	37.5	7.5	27/60/90
M9FAGC	505	1178	400	16	180.0	51.42	128.5	39.8	40.0	8.0	27/60/90

*F_A-fine aggregates, C_A-coarse aggregates, M-molarity, TAS-total alkaline solution, SP- Superplasticizer, all quantities are taken in kg/cubic meters

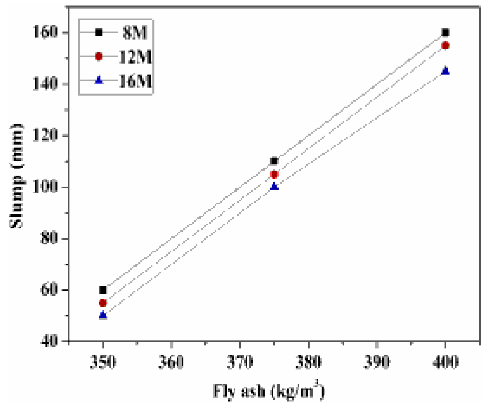


Fig. 4 Slump of GPC with different fly ash content

Table 5 Workability bands used in this study

Fly Ash (FA) Kg/cum	FA<350	350<FA<375	375<FA<400	FA>400
Degree of workability/Slump	Less (<75)	Medium (>75 but<100)	High (>100 but <150)	Very High (>150)

(BIS 3812-2003) (specific gravity 1.95) was procured from Ultratech Ready Mix Concrete plant for all the mixes tabulated in Table 4. X-ray Fluorescence (XRF) method was used to find out the parentage of different chemicals present in the fly ash and given in Table 2. Alccofine 1203 (AF) is a specially processed product based on slag of high glass content with high reactivity obtained through the process of controlled granulation and was procured from Ambuja Cements Limited, Mumbai . The Chemical composition of the alccofine which has been used in this study is shown in Table 3. XRD study was performed on alccofine, which normally consists of calcite and on fly ash which clearly shows the presence of crystalline phase so is indicated by sharp peaks of quartz, mullite, and mellite, shown in Fig. 2. A Naphthalene Sulphonate based water reducing super

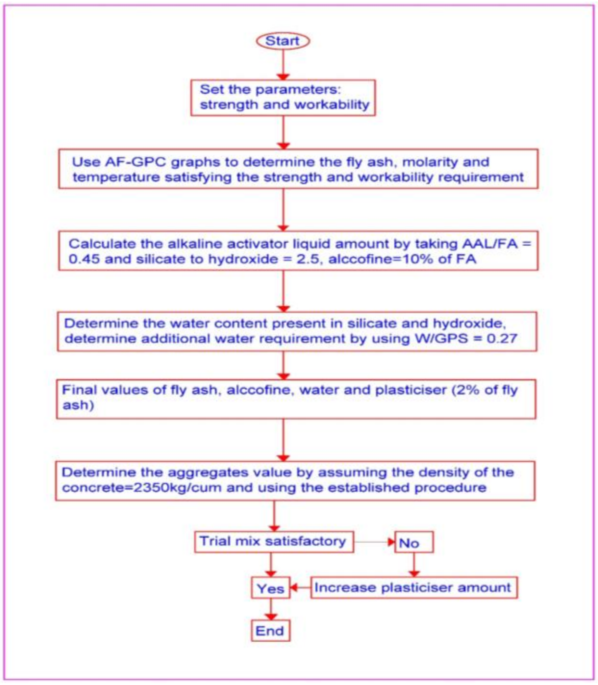


Fig. 5 Flow chart of the proposed mix design process for alccofine based GPC

plasticizer confirming to IS 9103:1999 (BIS 9103-1999) was used to enhance the fresh properties of geopolymer mix.

4. Proposed method for designing geopolymer concrete

4.1.1 AF-GPC Graph and workability bands

AF-GPC-Graphs (Alccofine, Fly ash based Geopolymer-Graphs) have been developed using the experimental data given in the Table 4 for the investigation and these graphs lie at the origin of the developed design mix process. These graphs (Figs. 6 to 8) originally represent the relationship between the fly ash and compressive strength for 8, 12 and 16M NaOH solutions with different curing conditions of ambient (average room temperature 27℃), 60℃ and 90℃. Compressive strength results obtained for the mixes tabulated in Table 4 were used to develop the proposed AF-GPC-Graphs. To choose the exact type of mix, workability bands have been provided in the Table 5. The workability of the fresh GPC was measured using the standard slump (BIS 516-1959). As per literature, high viscosity of alkaline solution led to less workable concrete. Further, higher concentration of alkaline solution speed up the geopolymerisation process due to development of heat in the matrix. This could have been the reason of increased stiffness and therefore, corresponding less values of slump. Also, CaO present in the alccofine led to formation of CSH, in addition to NASH (sodium aluminate silicate hydrate) which is also responsible for less values of slump. Therefore, plasticizer (2%) and alccofine (10%) were introduced in GPC mix in order to target the required slump and compressive strength. However, even for lesser values of slump the GPC placed and well compacted (Rangan 2007). The classification of the slump values was done

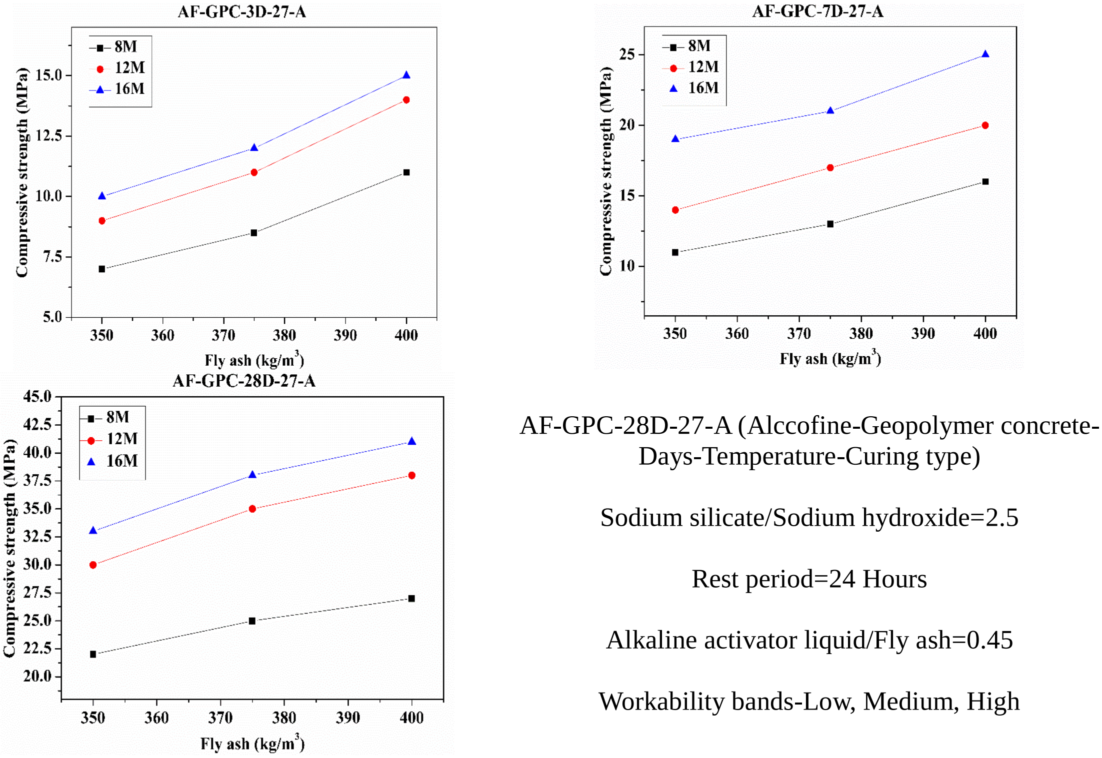


Fig. 6 Compressive strength with varying fly ash content, curing period at 27°C curing temperature

as per the condition of compaction and using the clause 7 of IS 456:2000 (BIS 456-2000). Based on the different slump values GPC was classified in terms of a very highly workable, highly workable, medium workable and low workable (BIS 516-1959, BIS 456-2000). Workability on the similar grounds has also been fixed by British standards and American concrete institute. Slump values observed for the mixes tabulated in Table 4 are plotted in Fig 4 and the above-mentioned criterion has been related to fly ash as shown in Table 5.

4.1.2 Proposed method for designing GPC using fly ash and alccofine

An attempt has been made in this paper, to propose mix design methodology for fly ash and alccofine based GPC in a rational way. By fixing the certain parameters in the production of GPC, it can be made economical and flexibility can be rendered in the design mixes both on the strength requirement and desired activator solution point of view. The essential features of the proposed method are the flexibility to select fly ash content, molarity of the sodium hydroxide and curing temperature required for specific strength and workability. The design procedure of the proposed mix design is outlined in the form of flow chart as stated in Fig. 5 and the step by step procedure is summarized as follows;

As discussed above the GPC set quickly when cured at elevated temperature so, initially a target 7 days for heat curing and 28 days for ambient curing, target compressive strength and workability are set, for the design process (Lloyd *et al.* 2010, Anuradha *et al.* 2012, Ferdous *et al.* 2013, Junaid *et al.* 2015, Pavithra *et al.* 2016). This target strength is then established on the AF-GPC-Graphs (Figs. 4-6), from where corresponding values of fly ash content, molarity of NaOH

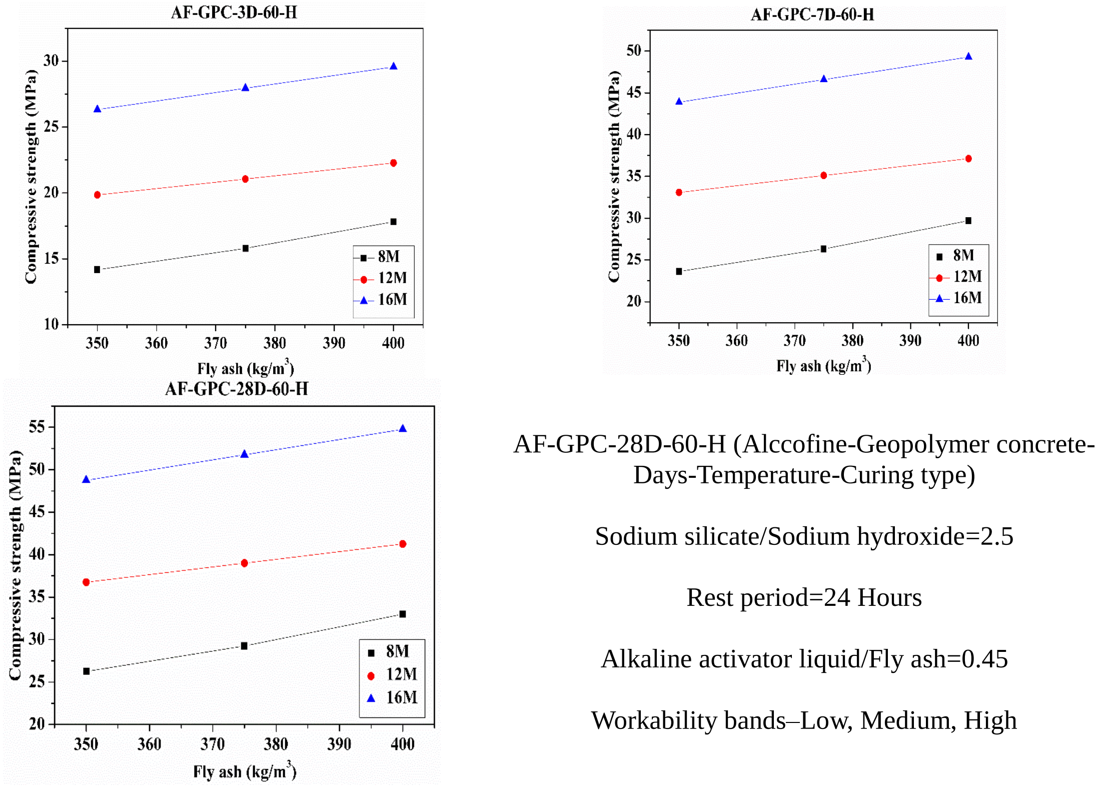


Fig. 7 Compressive strength with varying fly ash content, curing period at 60°C curing temperature

solution and curing temperature are obtained. However, if workability is not achieved for the located fly ash quantity and target strength using the proposed method, then amount of superplasticizer can be increased to reach at the required degree of workability level. The AF-GPC-Graphs presented in this study shows relationship between compressive strength and fly ash. Further, this relationship is based on the fact that 2% and 10% by weight of fly ash should be added as plasticizer and alccofine respectively.

Sodium silicate to sodium hydroxide (Na_2SiO_3/NaOH) ratio by mass has significant effect on the performance and mechanical properties of GPC. From the available literature, it has been found that for practical usage a value between 2.3 and 2.8 is appropriate (Lloyd *et al.* 2010, Anuradha *et al.* 2012, Ferdous *et al.* 2013, Junaid *et al.* 2015). However, keeping in mind the economy of the GPC a middle value of 2.5 has been fixed for this presented research. The next step in the production of the GPC is to select the appropriate alkaline activator liquid (AAL) to fly ash (FA) ratio. It has been found that a value between 0.3 to 0.6 is appropriate for practical usage (Hardjito *et al.* 2005, Junaid *et al.* 2015), however a value of 0.45 has been taken in this study to prepare the AF-GPC-Graphs as literature suggests that with less values of AAL/FA the concrete is not place-able. Calculate the value of NaOH and Na_2SiO_3 solutions by using the following equations

$$\frac{\text{AAL}}{\text{FA}} = \frac{\text{Sodium Silicate solution} + \text{Sodium hydroxide solution}}{\text{Fly Ash}} = 0.45 \ldots\ldots\ldots \qquad (1)$$

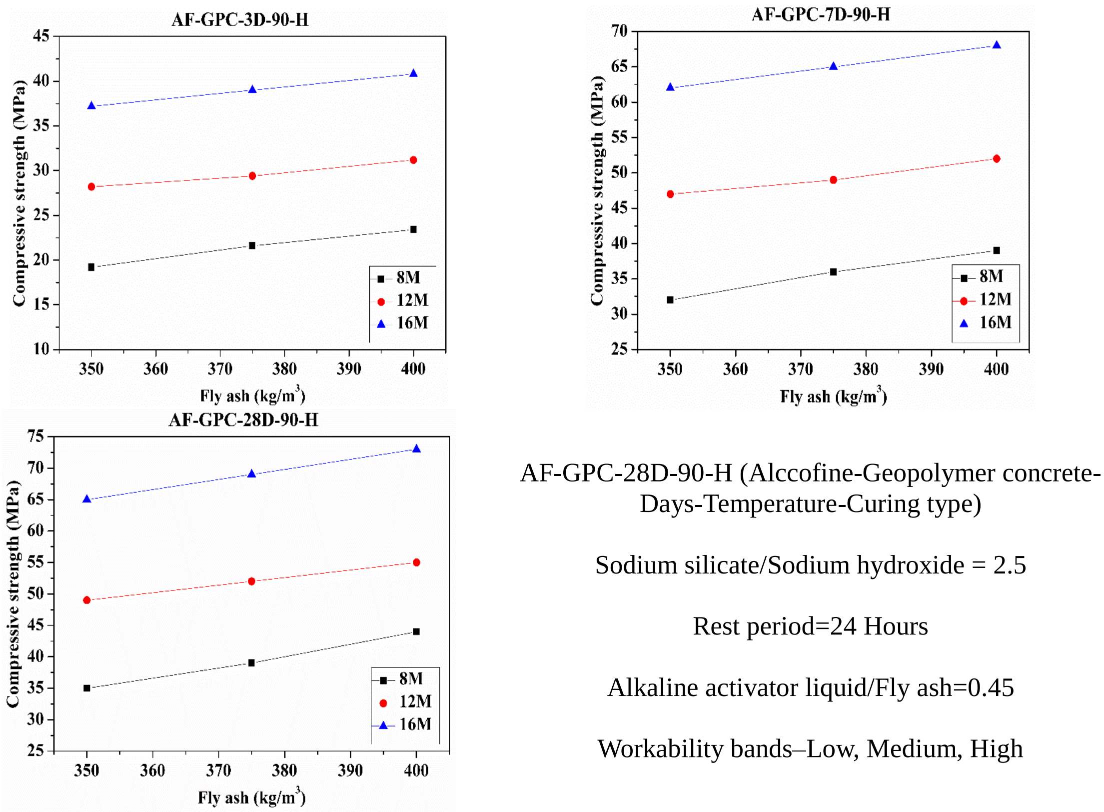


Fig. 8 Compressive strength with varying fly ash content, curing period at 90°C curing temperature

From which; AAL=0.45×Fly ash

$$\frac{\text{Sodium Silicate}}{\text{Sodium hydroxide}} = 2.5 \dots\dots\dots \tag{2}$$

From which; NaOH solution=AAL/3.5 and Na_2SiO_3 solution=2.5×sodium hydroxide solution water (sum of masses of additional free water and water used while preparing Na_2SiO_3 and NaOH) to geopolymer binder (sum of masses of fly ash, alccofine, NaOH solids and Na_2SiO_3 solids) ratio (W/GPB) has been kept 0.27 for this research (Hardjito *et al.* 2005, Lloyd *et al.* 2010, Anuradha *et al.* 2012, Ferdous *et al.* 2013, Ferdous *et al.* 2015, Junaid *et al.* 2015, Pavithra *et al.* 2016). These are named as geopolymer solids as they participate in the geopolymerisation process. Generally, the values of the water to geopolymer binder's ratio varies from 0.23 to 0.30, however decision has been taken to kept a value 0.27 by keeping in mind the workability and economy of the mixes. Knowing the above values, it becomes easy to calculate the exact amount of water and solids used in alkaline activator solution (AAL). Additional free water quantity can be calculated using the Eq. (3).

$$\frac{W}{GPB} = \frac{WOH + WSI + Wextra}{AF + FA + SolidsNaOH + SolidsNa2SiO3} = 0.27 \dots\dots\dots \tag{3}$$

where WOH, WSI, Wextra is the water present in the hydroxide solution, silicate solution and any additional free water in the system respectively. AF, FA, Solids NaOH, and Solids Na_2SiO_3, is the alccofine, fly ash and solids present in the NaOH solution, Silicate solution respectively. In this study, the density of the GPC has been considered as 2350 kg/m^3 on experimental basis and 70-75% of the mass of the GPC has been make up for the saturated surface dry aggregates. The aggregates used are 14, 10 and 7 mm in size and in the proportion of 45, 35 and 20% respectively. Further, the ratio of coarse and fine aggregates was considered 70:30 out of the total aggregates.

4.1.3 Proposed method for sample preparation

Various researchers have tried different methods for the sample preparation of the geopolymer concrete and suggested that the compressive strength of the GPC is not affected by mixing (Junaid *et al.* 2015). The procedure suggested for the preparation of the GPC samples is as discussed below:

1. NaOH is prepared before 24 hrs of the casting and uniformly mixed with the Na_2SiO_3 1 hr prior to the mixing of the ingredients of the GPC.

2. All the ingredients of GPC mixture are then mixed for at least 5 min in the Pan mixture, then poured into 150 mm size standard cube moulds and compaction was done on a vibrating Table for about 4-5 minutes. Sealed samples are then placed at room temperature for 24 hours rest period.

3. The samples now are heat cured at 60^o and 90^{o}C for 24 hours in an electric oven after a rest period of one day. After heat curing, the samples are returned to room temperature till the time of testing. However, the ambient cured samples are kept at the room temperature (27^{o}C) till the time of testing.

5. Verification of the mix methodology using an example

To validate the proposed method, an example is undertaken here to design the mix of GPC at heat and ambient curing for ordinary and precast members.

Assuming, the required compressive strength and workability for ordinary and precast GPC members is 25 MPa, 75 mm and 35 MPa, 75 mm respectively. The next step is to calculate the target mean strength. For this study, the target mean strength has been calculated as per Indian standard (BIS 10262-2009) and equals to $1.65 \times S + F_{ck} = F_{ck'}$, where $F_{ck'}$ is target mean compressive strength, F_{ck} is characteristics compressive strength (28 days for ambient curing and 7 days for heat curing), S is standard deviation. Several NaOH molarity and curing temperatures combinations could be used to achieve this target strength which are presented in AF-GPC-Graphs and Table 5 can be used to target the required workability. Nevertheless, and for the sake of illustration here, the AF-GPC-Graph (Fig. 6) is chosen and calculations are illustrated for mix TM-25. Fly ash quantity can be chosen from the AF-GPC-28D-27-A Graph for the required target mean strength and from Table 5 whichever is more, using this the corresponding values of alccofine, NaOH, Na_2SiO_3, plasticizer and aggregates can be calculated as explained below.

Alkaline activator liquid (AAL) is calculated using Eq. (1):

$0.45 \times 375 = 168.75$ kg/m^3.

Alccofine=$0.10 \times 375 = 37.5$ kg

Using Eq. (1) and Eq. (2) the values of the NaOH and Na_2SiO_3 can be calculated.

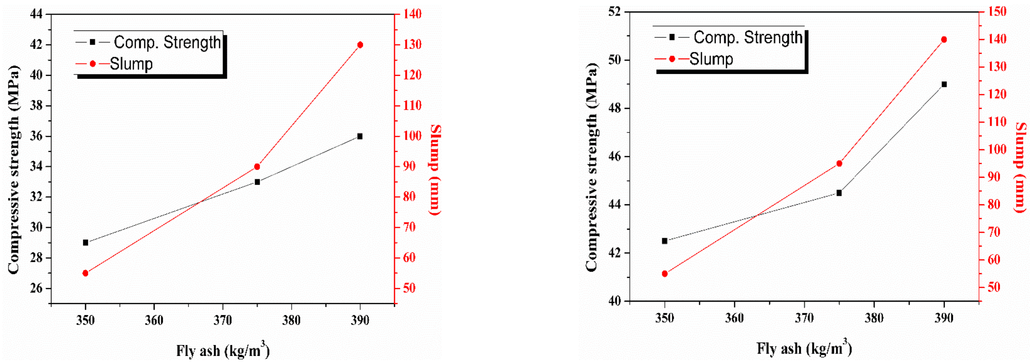
NaOH solution=$168.75/3.5 = 48.21$ kg

Na_2SiO_3 solution=$2.5 \times 48.21 = 120.53$ kg

The mass of solids and water present in the NaOH and Na_2SiO_3 can be calculated by using the

Table 6 Mix design quantities (kg) carried out using the above method for one cubic meter

Quantities	TM-25	TM-25	TM-25	TM-35	TM-35	TM-35
Target strength (MPa)	25	31.6	35	40	43.25	50
Required slump (mm)	75	75	75	75	75	75
Fly ash	350	375	390	350	375	400
Alccofine	35	37.5	39	35	37.5	40
Coarse Aggregates	1239	1210	1193	1236	1207	1178
Fine aggregate	531	519	511	530	517	505
NaOH	45	48.21	50.14	45.0	48.2	51.4
Molarity (M)	12	12	12	16	16	16
Na_2SiO_3	112.50	120.53	125.25	112.5	120.5	128.6
Extra water	30.1	32.2	33.5	30.1	37.3	39.8
Plastisizer	7	7.5	7.8	7	7.5	8
Curing	Ambient (28 Days) @ 27°C	Ambient (28 Days) @ 27°C	Ambient (28 Days) @ 27°C	Heat (24 Hours) 60°C	Heat (24 Hours) @ 60°C	Heat (24 Hours) @ 60°C
Average strength (MPa)	29.0	33.0	36.0	42.5	44.5	49.0
Slump (mm)	55	90	130	55	95	140



(a) TM-25 (b) TM-35

Fig. 9 Compressive strength & Slump and various fly ash content

composition of NaOH (36.1% solids by weight for 12M) and Na_2SiO_3 (44.1% solids by weight as per supplier's specifications) by weight and is given below.
Mass of solids in NaOH=(36.1/100)×48.21=17.40 kg
Mass of water in NaOH=48.21-17.40=30.81 kg
Mass of solids in Na_2SiO_3=(44.1/100)×120.53=53.15 kg
Mass of water in Na_2SiO_3=120.53-53.15=67.38 kg

Moreover, the AF-GPC-Graphs has been developed for W/GPB equals to 0.27. So, extra water quantity can be calculated using the Eq. (3).

(Wextra+30.81+67.38)/(37.5+375+17.40+53.15)=0.27; from which Wextra=32.2 kg.

By using the volumetric analysis, the other parameters like total aggregate can be calculated. Generally, in OPC based concrete the fine aggregate to coarse aggregate ratio is 30-70 and the same has been considered in this example. Commercially available 2% high dosage super-plasticizer's is added and if needed the percentage of super-plasticizer is slightly altered to satisfy the workability requirements. Similarly, the values for the mix TM-35 has been calculated. In addition, two more trial mixes were made with the target compressive strength of 25, 35 and 40, 50 for TM-25 and TM-35 mix respectively, to make the geopolymer concrete economical. The same practice is also followed while preparing the conventional concrete mix design in the laboratory before proposing the final values of the materials. The final values of the materials and the strength achieved using the above proposed method are listed in Table 6 below for mix TM-25 and TM-35 (TM-25: trial mix- target strength in MPa).

Fig. 9 shows the relationship between compressive strength & slump values with various fly ash content achieved from the Table. 6 for both the mixes, TM-25 and TM-35. By referring the Fig. 9, final values of the fly ash content for TM-25 and TM-35 could be in between 360 to 365 kg/cum, which will target the slump and compressive strength. The only difference is of being type of curing for the trial mixes.

6. Conclusions

Based on the data and observations presented above the following conclusions can be arrived upon:

1. A methodology for designing the geopolymer concrete has been demonstrated for implementation of ecofriendly material on large scale.
2. Proposed mix design method, targets the required compressive strength and workability.
3. The suggested method can be confidently used for alccofine and fly ash based geopolymer concrete.
4. It is possible to produce geopolymer concrete with alccofine for general purpose at room temperature (27^{o}C).
5. By inclusion of alccofine, workable and high strength concrete can be produced which can serve the cast in situ and precast concrete industry demands.

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