

Development and Determination of Mechanical properties of fly ash and slag blended geopolymer concrete

Sundar Kumar, S. ^x, Vasugi, J. [#], Ambily, P. S. ^{*} and Bharatkumar, B. H. ^{*}

Abstract

This paper summarizes the development of low concentration alkali activator geopolymer concrete mixes and the results of tests conducted to determine the mechanical properties such as compressive strength, split tensile strength and flexural strength of fly ash and slag based geopolymer concrete. Tests were also conducted to determine the elastic modulus of the geopolymer mixes developed. Steel fibres were introduced in the mix to determine the residual strength and enhancement in the flexural strength. Tests were also conducted to determine the fracture energy. The results have been compared with an ordinary Portland cement based concrete.

Keywords: Cement less binder, Geopolymer, Alkali activation, Fly Ash, Ground granulated blast furnace Slag, fibre reinforced concrete, room temperature curing.

1 INTRODUCTION

Ordinary Portland cement has evolved over decades and various forms of blended cements are available in the market today. These cements cater to a variety of specific requirements like quick setting, low heat of hydration, under water concreting etc apart from usual requirement of strength and durability. However the main reason for worry in the present scenario is the carbon footprint of this material. The manufacture of cement produces approximately equal quantity of green house gases. This makes the search for a more environmentally friendly material more relevant. Concrete researchers across the world have been working on alkaline activated cement composites for nearly past sixty years. Davidovits (1991) made significant contribution in this field, he introduced the term 'Geopolymer', research has been more streamlined and geopolymer has been able to find more applications due to the efforts of researchers like Davidovits. various models to account the reaction mechanisms and final product have been proposed. However signification work needs to be carried out so that geopolymer can be as user friendly as the conventional Portland cement based concrete. In most of the developing economies the success of geopolymer needs it to be made more user friendly. From civil engineers point of view fly ash and blast furnace slag turn out to be two fit candidates for alkaline activation. Their chemical compositions make them suitable for activation and the fact that they are available in abundant make them favorable candidates. Fly ash based geopolymers require high concentration alkaline activators which make it difficult to be made at site or at

existing ready mix batching plants. In most applications very quick or very high strength gain is not mandatory.

The history of alkaline activation dates back around 634 BC when alkaline activation was used in making bricks with lime and ashes, however, in modern times the first literature available on alkali activation dates back to late 1930's when blast furnace slag was activated with alkalis (Roy, 1999), investigations on ancient Roman and Egyptian constructions have revealed their composition as aluminosilicate calcium hydrates (Torgal et. al., 2008), Glukhovskiy (1965) has carried out some pioneering work in this field of alkaline activation of slags, he introduced a binder which he called 'soil-cement' since the cementitious material he developed looked like a rock he used the term soil-cement. However even after more than 70 years of active research in this area, controversy still exists about the reaction mechanism and the end products of the alkaline activation. Materials which mainly consist of silicate and calcium when activated with alkalis results in the formation of calcium silicate hydrate, a low concentration of the alkali is sufficient for the reaction to take place (Brough & Atkinson, 2002). However when the material to be activated mainly consists of silicates and aluminates a higher concentration (Xu, 2002) of alkalis is required and it results in a polycondensation reaction forming a chain of aluminosilicate molecules. Davidovits termed this reaction product as 'geopolymer'.

If geopolymer mixes with normal to medium strength can be achieved in reasonable time such as 7 to 14 days it would find a variety of applications. It is important to have a simple or preferably ambient curing regime so that it can be used with existing facilities. In this study the main objective was to develop geopolymer concrete mix of strength around 40 MPa at 28 days with low concentration of alkaline activator. The curing was normal ambient air curing. Basic parameters such as

- Sundar Kumar, S., and Ambily, P. S., are scientists and Bharatkumar, B. H., is a senior principal scientist at CSIR - Structural Engineering Research Centre, Chennai, India. E-Mail: ssk@serc.res.in, ssk.serc@gmail.com.
- Vasugi, J., was a project student at CSIR - Structural Engineering Research Centre, Chennai, India.

compressive, tensile and flexural strength were determined. Stress strain curve for the geopolymer concrete mixes in compression were determined. Studies were extended by incorporating a fixed dosage of steel fibre into the matrix. Residual strength and fracture parameters were determined for the fibre reinforced geopolymer concrete. The tests results have been compared with a Portland cement based concrete which was proportioned to achieve strength similar to that of the geopolymer mix

2 EXPERIMENTAL INVESTIGATIONS

2.1 Development of geopolymer concrete mix

Unlike Portland cement based concrete till date there are no standard method to design or proportion geopolymer concrete mixes. However, ample research has been carried out on the mechanism of geopolymerization and its relation to ingredients in the raw materials. Several trials were carried out to arrive at the final mix proportion. The basic criterion was to develop a mix with 28 days average cube compressive strength of around 40 MPa under ambient air curing and at a lower concentration of alkalies.

Fly ash

Low calcium, class F fly ash was used in the entire experimental work. The fly ash was procured from a thermal power plant near Chennai (Madras), India. The oxide composition and physical characteristics of fly ash used in the study is presented in Table 1.

Slag

Ground granulated blast furnace slag (GGBS) a byproduct of iron smelting industry was used in the present study. The oxide composition and physical characteristics of GGBS used in the study is presented in Table 1.

Compound	Fly Ash	GGBS
SiO ₂	62.1	43.4
Al ₂ O ₃	27.44	12.5
Fe ₂ O ₃	4.57	-
CaO	0.83	40.3
MgO	0.55	1.5
Na ₂ O	0.04	0.9
K ₂ O	1.17	0.6
TiO ₂	1.09	-
Mn ₂ O ₃	0.04	-
SO ₃	0.4	-
Specific gravity	2.2	2.9
Fineness (m ² /kg)	419	400
LOI	0.76	2.1

Alkali activator

Sodium based activator was used to activate fly ash and GGBS. Sodium hydroxide flakes and sodium silicate in liquid form of commercial grade were used in the present study. The physical and chemical properties of the sodium silicate solution are given in Table 2. Distilled water was used to prepare the activator solution. Since dissolution of NaOH in water is a exothermic reaction a substantial amount of heat is generated, this temperature need to be reduced to ambient temperature before being used in concrete preparation. Hence required amount of NaOH was dissolved in water one day prior to casting, however sodium silicate was dissolved in sodium hydroxide solution just before casting. The details of the trial mixes and strength development are given in Table 3.

Table 2 Physical and chemical properties of sodium silicate solution

Color and Appearance	Clear
Specific gravity	1.54
Na ₂ O (% by mass)	14.34
SiO ₂ (% by mass)	32.23
Molar ratio	1:2.26

Initially a geopolymer mix with 100 percent fly ash was tried out. A 10 Molar (M) sodium hydroxide was used to activate the fly ash. The workability of the mix was low and it took more than 48 hours (hrs) to set in ambient temperature, however under oven curing (65°C for 24 hrs) setting was under 8 hrs. It yielded a 28 day strength of 20 MPa. For the next mix an 8 M NaOH Solution was used, the consistency and setting time of the mix was similar to that of the previous mix but it yielded a compressive strength of 16 MPa. In the third trial mix 10 percent of the fly ash was replaced with GGBS and the liquid to binder ratio reduced by 0.1, all other parameters were kept as that of the second trial. Since the mix set quite faster and began to harden within 24 hrs hot air curing was avoided. The mix yielded a 28 days compressive strength of 38 MPa. Since replacement of 10% fly ash with slag resulted in a substantial increase in the compressive strength with better characteristics, it was decided to further increase the slag content in the subsequent mixes 25 and 50 percent of the fly ash was replaced with GGBS. At 50 percent fly ash replacement a 28 day compressive strength of 50 MPa was achieved. When the same mix was subjected to hot air curing at 65°C for 24 hrs there was a further enhancement of around 5 MPa in the compressive strength. However since the target strength was around 40 MPa it was decided to avoid hot air curing. Hence the mix no 4 shown in Table 3 was adopted for further studies.

2.2 Development of Portland cement concrete mix

In order to compare the behavior of the geopolymer concrete properties, a Portland cement based concrete

mix with compressive strength similar to that of geopolymer concrete was developed. ACI method of mix proportioning was used arrive at the initial proportion, the final proportion adopted after a few trial castings is shown in Table 4.

Compressive strength for each mix was determined at 7 and 28 days of age. Cubes were brought to surface dry condition and tested in a compression testing machine with constant loading rate.

Split tensile strength

Mix No	Composition in %		Mix Proportion GPS: S: CA: L/B	Molarity (M) of NaOH	Curing	Comp. strength (MPa)	Na ₂ O/SiO ₂	Al ₂ O ₃ /SiO ₂
	FA	GGBS						
1	100	00	1: 1.35: 3.2: 0.7	8	Oven	16	0.15	0.34
2	100	00	1: 1.35: 3.2: 0.7	10	Oven	20	0.19	0.34
3	90	10	1: 1.35: 3.2: 0.6	10	Air	38	0.18	0.34
4	50	50	1: 1.35: 3.2: 0.64	3	Air	50	0.28	0.33
5	50	50	1: 1.35: 3.2: 0.64	3	Oven	55	0.28	0.33
6	75	25	1: 1.35: 3.2: 0.64	3	Air	42	0.19	0.34

GPS = geopolymer solids, S = sand, CA = coarse aggregate, L/B = Liquid/Binder

Steel Fibres

It is well known that steel fibres enhance the performance of concrete in terms of cracking, flexure energy absorption and many such parameters. Hence investigations were also carried out with 0.75% crimped steel fibres added to the concrete matrix. The length of the fibres were about 20mm and the aspect ratio was 100.

Mix No	Mix Proportion (C:S:CA)	Water/ Cement
1	1:1.67:2.77	0.47

C = cement, S = sand, CA = coarse aggregate

Casting of the specimens

Cubes of size 100mm were cast to determine the compressive strength, cylinders of 100mm and 150mm diameter and diameter/height of 2 were cast to determine the split tensile strength and the stress strain behavior under axial compression. Prisms of 100x100mm cross section and 500mm length were cast to determine the flexural & residual strength and fracture parameters. All the Portland cement concrete specimens were cured under water for 28 days and geopolymer concrete specimens under ambient air curing. All the tests were carried out after 28 days. Table 5 gives the details in terms of quantity of materials per cubic metre of concrete (assuming the density as 2400 kg/m³) of the final 4 mixes that were adopted for detailed investigations.

2.3 Testing of Specimens

Compressive Strength

Since tensile strength is relatively very low when compared to compressive strength and due to the brittle nature of the material, it is extremely difficult to test under direct tensile loading. Hence its tensile strength is determine indirectly, a vertical compressive stress is applied on a cylinder along it height, this in turn results in the development of tensile stress along a line which is normal to the line of application of the load.

Flexural Strength

Flexural strength of concrete was determined by applying a two point load on a prism of 100x100x500mm size. Figure 1 shows testing of prisms to determine flexural strength.



Figure 1 Flexural Strength Test Setup

Residual Strength

Residual strength is a concept used to define the performance of fibre reinforced concrete beyond its initial cracking load. Tests performed are similar to that carried out to determine the flexural strength. However closed

loop displacement controlled testing is required to control the rate of application of load. Prisms of 100x100x500mm size were tested to determine the residual strength. Third point loading was adopted and a constant rate of loading at 0.05mm/min.

Table 5 Quantity of material per cubic metre of concrete in kg

Mix ID	Cement	Fly ash	GGBS	Water	Sand	Coarse aggregate	NaOH	Na2SiO3	Fibre
M1	406	-	-	191	678	1125	-	-	-
M2	406	-	-	191	678	1125	-	-	59
M3	-	194	194	174	523	1241	24.8	49.6	-
M4	-	194	194	174	523	1241	24.8	49.6	59

Note: M1 and M2 are Portland cement concrete, M3 and M4 are geopolymer concrete mix

Fracture Parameters

The fracture parameters are helpful in predicting the fracture energy of the concrete mixes which in turn signifies the energy absorbing capacity of the concrete mix which was improved due to the addition of fibres. The fibres in the concrete enhance the ductility and thereby the failure is not sudden, the cracks were initiated in the specimens in the form of notch.

The fracture properties were determined from the prism specimens of dimension 100x100x500 mm. A notch of 30 mm depth was cut at bottom exactly at mid span. The span length was 400 mm. The experimental set up is shown in Figure 2. The prisms were loaded with a single point mid span loading at a constant rate. The fracture parameters are determined using Linear – Elastic Fracture – Mechanics (LEFM) approach.



Figure 2 Three point bending test on notched prism specimen

The load vs mid span deflection plots are used to determine the fracture energy of the concrete. The area

under the load – deflection plot indirectly measures the fracture energy. The area for consideration is limited up to the deflection limit of $l/500=0.8\text{mm}$ where l is the span of the specimen. In this case, the span length is taken as 400 mm. For the GPC and OPC mixes without fibre this deflection limit was less than 0.8 mm, in such case, the area under the plot is considered up to the maximum deflection.

The fracture energy (GF) or specific fracture energy is the energy needed to create a crack of unit area and is given

Table 6 Experimental test results

Specimen ID	Compressive strength MPa		Young’s modulus (GPa)	Split tensile strength (MPa)	Flexural strength (MPa)	Residual strength _{maxdefln} (MPa)	GF (J/m ²)
	7 day	28 day					
M1(OPC)	46	57	33	3.81	5.20	1.295	139.42
M2(OPC-F)	55	58	39	4.68	6.42	4.623	419.18
M3(GPC)	36	52	18	3.44	4.62	1.446	46.613
M4(GPC-F)	38	53	19	4.33	6.35	3.249	407.32

by (RILEM committee FMC-50(1985)) as,

$$G_F = \frac{(W_F + W_S \delta_o)}{A_{lig}} \quad \text{in J/m}^2$$

Where, W_F is the work of fracture (equal to the area under the load deflection plot) in Nmm

W_S is the sum of the weight of the specimens and fixtures in N

δ_o is the displacement caused due to the self-weight of specimens and fixtures in mm.

A_{lig} is the area of the ligament that was intact before the test.

3 RESULTS AND DISCUSSIONS

Table 6 summarises the test results of the final four mixes that are mentioned in Table 5. It can be seen that both OPC as well as GPC mixes were able to achieve a 28 days compressive strength in excess of 50 MPa though OPC mixes had a slightly more strength. A major difference was noticed in the young's modulus, GPC mixes exhibited a significantly lower modulus of elasticity. The reason behind this deviation is an active area under investigation by many people. The difference should essentially be due to the aluminosilicate network as the aggregate used is same for both OPC and GPC concrete. More investigations need to be carried out before concluding on this aspect. The effect of fibres was marginally higher in geopolymer concrete. The split tensile strength was enhanced by 1.26 times and the flexural strength by 1.37 times. However it is to be noted that the residual strength of fibre reinforced geopolymer concrete was lesser than that of fibre reinforced OPC concrete. This may be a point on which further studies can be taken up, 'the performance of fibre in cracked geopolymer concrete sections' as most of the concrete structures are cracked at service loads. Similar trend has also been observed in the fracture energy calculation.

4 CONCLUDING REMARKS

The experimental works prove that geopolymer concrete with moderate to moderately high strength can be developed at very low concentration of alkaline activator and ambient air curing.

Use of a fly ash in combination with slag is found to be more reactive and the strength gained is more. However further morphological investigations will be useful in determining the type of network (microstructure) that develops when a mixture of fly ash and slag are activated.

Specialized curing regimes such as steam curing, hot air curing are required only when accelerated strength gain is required. When the design strength is required only at 28 days normal air curing is sufficient.

Additions of steel fibres have improved the flexural, residual and fracture properties as expected.

Further detailed studies on geopolymers with low concentration of alkali activators would help geopolymer concrete to be produced in situ with the available facilities and manpower itself.

5 ACKNOWLEDGEMENT

This paper is being published with the kind permission from 'The Director' CSIR- Structural Engineering Research Centre, Chennai. The authors would like to thank the staff of Advanced Materials Laboratory, CSIR- Structural Engineering Research Centre, Chennai for the help rendered during the experimental work.

6 REFERENCES

- Davidovits, J. 1991. "Geopolymer: inorganic polymeric new materials", *Journal of Thermal Analysis*, no. 37, pp 1633-1656.
- Roy, D. M. (1999). "Alkali-Activated Cements, Opportunities and Challenges." *Cement and Concrete Research* 29(2), pp 249-254.
- Torgala, F.P., Castro-Gomes, J., Said Jalalic (2008). "Alkali-activated binders: A review: Part 1. Historical background, terminology, reaction mechanisms and hydration products." *Construction and Building Materials* 22(7), pp 1305-1314.
- V. D. Glukhovskiy (1965). "Soil Silicates: Their Properties, Technology and Manufacturing and Fields of Application"; Ph.D. Thesis. Civil Engineering Institute, Kiev, Ukraine.
- A.R Brough and A Atkinson (2002). "Sodium silicate-based, alkali-activated slag mortars: Part I. Strength, hydration and microstructure" *Cement and Concrete Research* 32(6), pp 865-879.
- Xu, H. and J. S. J. van Deventer. (2000). "The geopolymerisation of aluminosilicate minerals" *International Journal of Mineral Processing*, 59(3), pp.247-266.