CHAPTER 3

EXPERIMENTAL PROGRAMME

3.1 General

This chapter presents the details of the developments in the process of making fly ash based geopolymer concrete. The materials used for making geopolymer concrete, mix proportions, manufacturing and curing of the test specimens are explained. This is followed by a description of number and types of specimens used, parameters tested and the various test procedures adopted.

3.2 Materials

3.2.1 Fly ash

Fly ash is a fine powder recovered from the gases of burning coal during the generation of electricity. These micron-sized earth elements consist primarily of silica, alumina and iron. Fly ash particles are almost totally spherical in shape, allowing them to flow and blend freely in mixtures. Fly ash improves considerably the performance of binder paste and increases the bonding action with aggregate and reinforcement. The properties of fly ash may vary considerably according to several factors such as the geographical origin of the source coal, conditions during combustion and sampling position within the power plant. The major elemental constituents of fly ash are Si, Al, Fe, Ca, C, Mg, K, Na, S, Ti, P and Mn. Nearly all naturally occurring elements can be found in fly ash in trace quantities. Certain trace elements, including As, Mo, Se, Cd and Zn, are primarily associated with particle surfaces. The most abundant species in fly ash extracts are inorganic ions derived from Ca, Na, Mg, K, Fe, S and C.

Fly ash has been widely used as a partial replacement of cement in concrete for over half a century. The benefits include saving of cement and lowering of the heat of hydration in mass concrete (Baoju et al 2005). The use of fly ash in concrete is economical and modifies the properties of concrete in both the fresh and hardened states with improvements to workability, strength, abrasion, heat evolution, shrinkage and reduces water reducing admixture demands. In addition, the storage and disposal problem of fly ash, which is an industrial waste or by-product is also solved by the use of fly ash in concrete; otherwise, the fly ash has to be disposed of in landfills at a considerable cost. It reduces cracking due to autogenous and plastic shrinkage. It increases the packing density of the cementitious system, thus creating a less permeable structure (Malek et al 2005).

Geopolymer concrete in this study was made from low calcium fly ash with a combination of sodium hydroxide (NaOH) and sodium silicate solution (Na_2SiO_3). Fly ash used in this study was low-calcium (ASTM Class F) dry fly ash from Ennore thermal power station, Chennai as shown in Table 3.1. The chemical compositions of the fly ash from all batches as determined by X-Ray Fluorescence (XRF) analysis are given in Table 3.2.

Table 3.1 Type of Fly ash Used for the Study

Material	Class	Source
Fly ash	F	Ennore thermal power station, Chennai, Tamil Nadu, India.

Table 3.2 Test Report on Fly ash Analysis (ASTM Class F)

S.No	Constituents	% Composition
1.	Silica (as SiO_2)	48
2.	Alumina (as Al_2O_3)	29
3.	Ferric Oxide (as Fe_2O_3)	12.7
4.	Calcium Oxide (as CaO)	1.76
5.	Magnesium Oxide(MgO)	0.89
6.	Sodium (as Na_2O)	0.39
7.	Potassium (as K_2O)	0.55
8.	Sulphur (as SO_3)	0.5
9.	Loss On Ignition	1.61

3.2.2 Alkaline Activators

The alkaline liquid used was a combination of sodium silicate solution and sodium hydroxide solution. The NaOH solids were dissolved in water to make the solution. The mass of NaOH solids in a solution depends on the concentration of the solution and is expressed in terms of Molar (M). NaOH solution with a concentration of 8 M consisted of 8 x 40 = 320 grams of NaOH solids (in flake or pellet form) per litre of the solution, where 40 is the molecular weight of NaOH. The mass of NaOH solids was measured as 262 grams per kg of NaOH solution of 8M concentration. Similarly the mass of NaOH solids per kg of the solution for 14M concentration was measured as 404 grams and for 16M mass of NaOH solid is 444 grams. Chindaprasirt et al (2007) found that, to produce a higher strength geopolymer the optimum sodium silicate to sodium hydroxide ratio was in range of 0.67 to 1.00.

Alternatively the concentration of NaOH between 10M and 20M give small effect on the strength. Test result of sodium silicate is shown in Table 3.3.

Table 3.3 Test Result of Sodium Silicate

S.No	Constituents	Percentage Composition
1.	Na_2O	13.7
2.	SiO_2	29.4
3.	Water	55.9

3.2.3 Aggregates

In the absence of the usage of proper alternative aggregates becoming possible in the near future, the concrete industry globally will consume 8-12 billion tons annually of natural aggregates after the year 2010 (Tsung et al 2006). Aggregates are inert granular materials such as sand, gravel or crushed stone which, along with water and Portland cement, constitute an essential ingredient in concrete. Good concrete mix aggregates need to be clean, hard, strong particles free of absorbed chemicals or coatings of clay and other fine materials that could cause the deterioration of the concrete. Aggregates which account for 60 to 75 percent of the total volume of concrete are divided into two distinct categories fine and coarse. Fine aggregates generally consist of natural sand or crushed stone with most particles passing through a 4.75 mm sieve. Coarse aggregates are particles retained in 4.75 mm sieve. Gravels constitute the majority of coarse aggregate used in concrete with crushed stone making up most of the remainder.

3.2.3.1 Fine Aggregate

River sand available in Chennai was used as a fine aggregate and tested as per IS: 2386 (part III). Sieve analysis results of fine aggregate are shown in Table 3.4.

Table 3.4 Sieve Analysis Result of Fine Aggregate

IS Sieve size	Mass Retained on Sieve (gm)	Cumulative Mass Retained (gm)	Cumulative % Mass Retained	Cumulative % Passing
4.75mm	-	-	-	-
2.36mm	70	70	3.5	96.5
1.18mm	98	168	8.4	91.6
600µ	985.5	1153.5	57.68	42.33
300µ	726.5	1880	94	6
150µ	95	1975	98.75	1.25
Pan	25	2000	100	0

3.2.3.2 Coarse Aggregate

The coarse aggregate comprises the portion of the aggregate which has large particle size. It may be either crushed or uncrushed. They generally posses all the essential qualities of a good building stone showing very high crushing strength, low absorption value, least porosity, interlocking textures variety of appealing colors and susceptibility to perfect polish. In the present study granite has been used as a coarse aggregate in concrete. Sieve analysis of the coarse aggregate has been done and the percentage passing at different sieves is presented in Table 3.5. Figure 3.1 shows the materials used in preparing geopolymer concrete. The method of preparation of geopolymer concrete is shown in Figure 3.2.

Table 3.5 Sieve Analysis Result of Coarse Aggregate

IS sieve Size	Mass retained on each sieve (gm)	Cumulative mass Retained (gm)	Cumulative % mass retained	Cumulative % Passing
12.5mm	181.0	181.0	9.05	90.95
10mm	714.3	895.5	44.7	55.2
4.75mm	55.5	1987	99.3	0.6

Figure 3.1 Materials used in Geopolymer Concrete

3.3 Preliminary Laboratory Work

The preliminary laboratory work revealed the following:

The main objectives of the preliminary laboratory work were:

- To familiarize with the making of fly ash-based geopolymer concrete.
- To understand the effect of the sequence of adding the alkaline activator to the solids constituents in the mixer.
- To observe the behaviour of the fresh fly ash-based geopolymer concrete and its workability.
- To develop the process of mixing.

3.3.1 Mixing

Davidovits (2002) has suggested that the preference of a mixture of sodium silicate solution and sodium hydroxide solution together at least one day before adding the liquid to the solid constituents. The fly ash and the aggregates are first mixed together in a mixing pan for about 5 minutes manually as shown in Figure 3.3. After the dry mix is made, the prepared alkaline solution is thoroughly mixed with the dry mix for another 5 minutes to make fresh geopolymer concrete which is shown in Figure 3.4. The fresh concrete was placed in the mould. The specimens were compacted with three – layer placing and tamping using a rod. This was followed by an additional vibration of 10 seconds using a vibrating table. Specimens such as cubes, cylinders and beams were cast and tested. After casting the concrete mix was allowed to settle down in the moulds for 30 minutes. Different batches were adopted for 7 days, 14 days and 28 days of testing. The preparation of geopolymer concrete is shown in Figure 3.2.

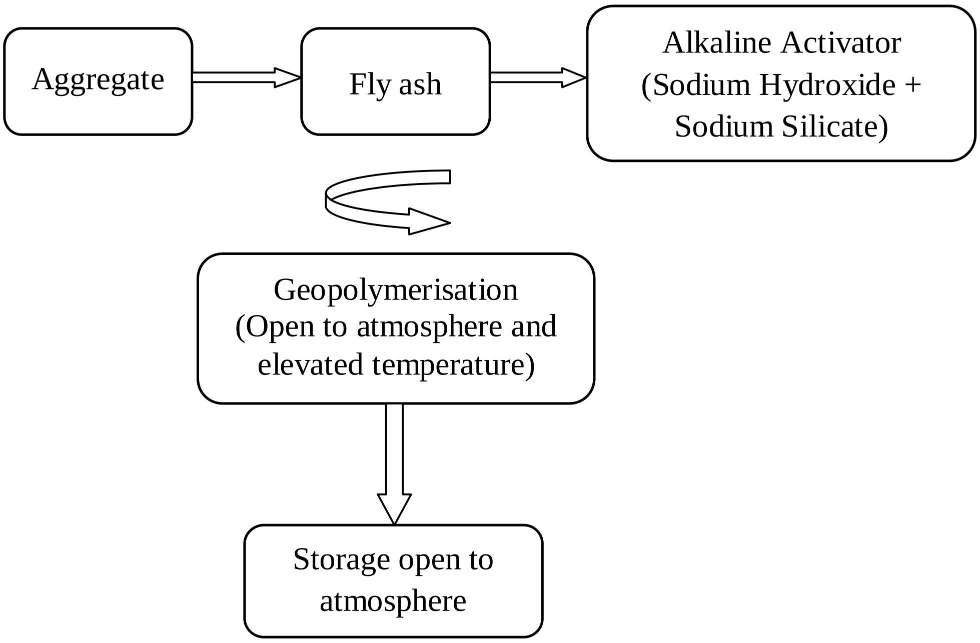

Figure 3.2 Preparation of Geopolymer Concrete

Figure 3.3 Dry Mixing of Materials

Figure 3.4 Fresh Geopolymer Concrete

3.3.2 Curing

There are two types of curing used in this study viz., Curing at room temperature and curing at an elevated temperature of 60ºC in laboratory oven. After casting, the concrete mix is allowed to settle in the mould for 30 minutes. For air curing the specimens were allowed to cool in air, demoulded and kept open until the day of testing as shown in Figure 3.5. The specimens were kept in the hot air oven for curing at 60^{o}C as shown in Figure 3.6. During the curing process, the geopolymer concrete experiences polymerization process. Due to the increase in temperature, polymerization become more rapid and the concrete gain 70% of its strength within 3 to 4 hrs of curing (Kong and Sanjayan, 2008). Heat curing of GPC is generally recommended, both curing time and curing temperature influence the compressive strength of GPC (Mustafa Al Bakri, 2011). With curing temperature in range of 60^{o}C to 90^{o}C within time in 24 to 72 hrs, the compressive strength of concrete can be obtained about 400 to 500 kg/cm^2 (Chanh et al, 2008).

Figure 3.5 Specimen After Curing

Figure 3.6 Oven Curing

3.4 Mix Proportion

3.4.1 Mix Proportion for Geopolymer Concrete Block

The laboratory program conducted in this investigation focused on four basic mixes and these were designated with the molarity of NaOH. The concentration of NaOH used in the experimentation was based on the review of previous research (Hardjito and Rangan 2005). Accordingly the performances of geopolymer concrete block specimens made with 8M, 10M, 12M and 14M of NaOH were evaluated. The ratio of sodium silicate solution-to-sodium hydroxide solution was fixed as 2.5. The ratio of fly ash: sand: coarse aggregate was 1:1.1:2.6 with ratio of activator solution to fly ash as 0.4. The geopolymer concrete mixes were designated as GP1, GP2, GP3 and GP4 respectively. Table 3.6 shows the mix proportion for GPC.

Table 3.6 Mix Proportions for GPC for 1 m³ of Concrete

Specimen	Designation of Mix	Aggregate		Fly ash (kg)	NaOH Solution		Sodium Silicate (kg)	Curing Condition
		C.A (kg)	Sand (kg)		Mass (kg)	Molarity (M)		
GPC Solid Block (150 x150 x 150 mm)	GP1	1274	539	490	41	8M	103	30ºC and 60ºC
	GP2	1274	539	490	41	10M	103	30ºC and 60ºC
	GP3	1274	539	490	41	12M	103	30ºC and 60ºC
	GP4	1274	539	490	41	14M	103	30ºC and 60ºC
GPC Hollow Block (100 x 100 x 250 mm) with one hollows of size 45x 75 x125mm	GP1	1274	539	490	41	8M	103	30ºC and 60ºC
	GP2	1274	539	490	41	10M	103	30ºC and 60ºC
	GP3	1274	539	490	41	12M	103	30ºC and 60ºC
	GP4	1274	539	490	41	14M	103	30ºC and 60ºC

3.4.2 Mix Proportion for Geopolymer Brick

The behaviour of unreinforced geopolymer brick masonry prism is compared with clay brick masonry prism. English bond unreinforced Clay Brick Prism (CBP) and Geopolymer Brick Prism GBP (M1) and GBP (M2) of brick size 225 x 105 x 70 mm were cast using 10M and 12M NaOH concentration with prism dimension of 609 x 220 x 609 mm (h/t = 2.77) and 609 x 220 x 914 mm (h/t = 4.3). Table 3.7 shows the mix proportion for preparing GPC bricks.

Table 3.7 Mix Proportion for Geopolymer Brick

Designation of Prism	Type of Brick	Mix Proportion of Brick	Mix Proportion of Fly ash : Binder)	Mix Proportion of mortar (Cement : Sand)
CBP	Clay Brick	Burnt Clay Moulded	-	1: 4
GBP (M1)	Geopolymer Brick	1:3 (Fly ash: Quarry Dust)	1: 0.54	1: 4
GBP (M2)	Geopolymer Brick	1: 1.1: 2.6 (Fly ash :sand: Coarse Aggregate)	1:0.54	1: 4

3.5 Summary

This chapter enlists the materials used for making geopolymer concrete, mixture proportions, manufacturing and curing of the test specimens. Fly ash used in this study was low-calcium (ASTM Class F) dry fly ash from Ennore thermal power station, Chennai. The alkaline liquid comprises a combination of sodium silicate solution and sodium hydroxide solids. Coarse and fine aggregates used in the local concrete industry were used. The coarse aggregates were crushed granite-type aggregates comprising 20 mm, 14 mm and 7 mm and the fine aggregate was fine sand. The mixture proportions used in this study were developed based on previous study on fly ash-based geopolymer concrete (Hardjito and Rangan, 2005). Molarity of sodium hydroxide (NaOH) solution was chosen in the range of 8M to 14M. Ratio of activator solution-to-fly ash by mass was fixed to be 0.40. Curing at elevated temperatures was done in two different ways, i.e. curing at room temperature and in the laboratory oven at 60ºC. Mix proportion used for geopolymer brick to construct geopolymer prism and clay brick prism is also presented.