CHAPTER 3

RESEARCH METHODOLOGY

3.1 Introduction

This Chapter describes the experimental work performed in this research study. It includes the details of various materials used in the concrete mixtures, design of mix proportions, method of preparation of the concrete mixes, and casting and curing of test specimens. The procedures used to evaluate the fresh properties of SCC such as filling ability, passing ability and resistance to segregation and physical and mechanical properties such as density, water absorption, compressive strength, tensile and flexural strength, modulus of elasticity, Poisson's ratio, creep and drying shrinkage are also described in this chapter. The experimental details of fresh properties and hardened concrete tests are shown in Table 3.1.

3.2 Materials

The materials that were used for making SCGC and OPC control mixes are low-calcium (ASTM Class F) fly ash as source material, silica fume, Type I Portland cement, sodium hydroxide and sodium silicate as alkaline activators, fine and coarse aggregate, superplasticizer and water. The description of each of the material used to prepare the concrete mixes is described in the following sub-sections. In order to minimize the effect of constituent materials on the properties of concrete, all the materials were obtained in single bulk deliveries and stored in airtight containers to prevent deterioration due to ambient conditions.

Table 3.1 Experimental Details of Concrete Tests

Sample Type	Test type	Standard	Type of Measurement	Equipment	Testing Age	Sample size	Number of sample	Unit
Fresh Concrete	Slump flow	European guidelines EFNARC 2005	Filling ability	Slump cone, Base plate & Measuring tape	Fresh state	0.0056 m^3	1 sample/mix	mm
	T_{50} Slump flow		Viscosity/flow ability	Slump cone, Base plate & Stop watch		0.0056 m^3		sec
	V-Funnel		Filling ability	V-Funnel apparatus		0.0112 m^3		sec
	L-Box		Passing ability	L-Box apparatus		0.0142 m^3		-
	J-Ring		Passing ability	J-Ring apparatus		0.0056 m^3		mm
Hardened Concrete	Density	BS EN 12390-7:2009	Unit weight	Weighing Machine	After removing from moulds	100x100x100 mm cube & 100x200 mm cylinder	6 cubes, 6 cylinders/mix	kg/m^3
	Water Absorption	ASTM C 642-96	Permeability characteristics	Oven, Weighing Machine	28 days		3 cubes, 3 cylinders/mix	%
	Compressive Strength	BS EN 12390-3:2002	Crushing strength	2000 KN Digital Compressive & Flexural Testing Machine	3, 7, 28, 90 and 180 days	100x100x100 mm cube	3/mix/age of testing	N/mm^2
	Splitting Tensile Strength	BS EN 12390-6:2000	Tension strength		3, 7, 28 and 90 days	100x200 mm cylinder	2/mix/age of testing	N/mm^2
	Flexural Strength	BS EN 12390-5:2000	Deflection/ Ductility		3, 7, 28 and 90 days	100x100x500 mm prism	2/mix/age of testing	N/mm^2
	Modulus of Elasticity and Poisson's Ratio	ASTM C 469	Stiffness	Universal Compression Testing Machine	3, 7 and 28 days	100x200 mm cylinder	2/mix/age of testing	GPa
	Creep	ASTM C 512	Time-dependent Deformation under load	Creep frames	Up to 1 year	150x300 mm cylinder	6/mix	Micro-strain
	Drying Shrinkage	ASTM C 157	Time- dependent Deformation without load	Mechanical Dial Gauge Comparator	Up to 1 year	76x76x285 mm prism	2/mix	Micro-strain

3.2.1 Fly Ash

Fly ash which contains high percentage of silica and alumina is known to be a suitable source material for making geopolymeric concrete. Therefore, in this research, fly ash was chosen as a source material for making SCGC mixes. Fly ash, complying with BS EN 450-1:2005 [127] was obtained from Manjung power station, Perak, Malaysia. Specific gravity of fly ash was found to be 2.38 and it had Brunauer Emmer Teller (BET) surface area (measured by nitrogen-adsorption method) of 1.5895 m^2/g. The chemical composition of fly ash as determined by X-Ray Fluorescence (XRF) analysis is given in Table 3.1. According to Fernández-Jimenez and Palomo [32], to produce optimal binding properties of geopolymeric concrete, the fly ash should have low calcium oxide (CaO) content and low content of unburned carbon, while the iron oxide (Fe_2O_3) content should not exceed 10%. Table 3.2 shows that the fly ash contained a very low percentage of carbon as indicated by loss on ignition (LOI) value. It can be seen that CaO content is 6.73% and LOI value is 0.88%, while the Fe_2O_3 content is 4.57%, which is lower than 10%. The molar Si/Al ratio was also about 2, which is the same as proposed by Davidovits [6].

Table 3.2 Chemical Composition of Fly ash as determined by XRF

Oxide	(%) by mass	Requirements as per BS EN 450-1:2005 [127]
Silicon dioxide (SiO_2)	51.30	Min. 25%
Aluminium oxide (Al_2O_3)	30.10	-
Ferric oxide (Fe_2O_3)	4.57	-
Total $SiO_2 + Al_2O_3 + Fe_2O_3$	85.97	Min. 70%
Calcium oxide (CaO)	6.73	Max. 10%
Magnesium oxide (MgO)	0.89	Max. 4%
Phosphorus pentoxide (P_2O_5)	1.60	-
Sulphur trioxide (SO_3)	1.40	Max. 3%
Potassium oxide (K_2O)	1.56	-
Titanium dioxide (TiO_2)	0.70	-
Sodium oxide (Na_2O)	0.26	Max. 5%
Loss on ignition (LOI)	0.88	-

3.2.2 Silica Fume

Commercially available silica fume supplied by Elkem materials in dry densified form with Grade 920 complying with the mandatory requirements of BS EN 13263-1:2005+A1:2009 [128] was used in this study. The physical properties and chemical composition of silica fume are presented in Table 3.3. Silica fume was used in SCGC mix as replacing part of the fly ash in order to improve the hardened properties of fly ash-based SCGC. Fly ash was replaced with silica fume at the contents of 5%, 10% and 15% by mass.

Table 3.3 Physical Characteristics and Chemical Composition of Silica fume

Oxide	(%) by mass	Requirements as per BS EN 13263-1:2005 [128]
Silicon dioxide (SiO_2)	90.2	Min. 85%
Aluminum oxide (Al_2O_3)	0.82	--
Ferric oxide (Fe_2O_3)	1.67	--
Calcium oxide (CaO)	1.24	--
Magnesium oxide MgO	0.31	--
Sulphur trioxide (SO_3)	1.40	Max. 2%
Potassium oxide (K_2O)	4.02	--
Sodium oxide (Na_2O)	0.10	--
Loss on ignition (LOI)	0.23	Max. 4%
BET Specific surface (m^2/g)	18.825	Min. 15
Specific gravity	2.22	--

3.2.3 Ordinary Portland Cement

Ordinary Portland cement (OPC), the principal ingredient in Portland cement concrete, is the most common type of cement extensively used around the world for general purpose. Commercially available Type I Portland cement supplied by Tasek Corporation Berhad (4698-W), Ipoh, Perak, was used in the preparation of OPC-based control mix. The chemical composition of the cement is given in Table 3.4. The cement met the requirements of BS EN 197-1:2000 [129]. OPC Type 1 was preferred as the observation on concrete properties could be done in normal hydration process.

Table 3.4 Chemical Composition of Ordinary Portland Cement

Oxide	(%) by mass	Requirements as per BS EN 197-1:2000 [129]
Silicon dioxide (SiO_2)	21.28	-
Aluminum oxide (Al_2O_3)	5.60	-
Ferric oxide (Fe_2O_3)	3.36	-
Calcium oxide (CaO)	64.14	-
Magnesium oxide MgO	2.06	-
Sulphur trioxide (SO_3)	2.14	Max. 3.5%
Potassium oxide (K_2O)	0.49	-
Insoluble residue	0.22	Max. 5%
Total Alkalis	0.05	-
Loss on ignition (LOI)	0.64	Max. 5%

3.2.4 Aggregates

Aggregates are the main constituents of concrete as the properties of aggregates significantly influence the properties of concrete. The quality of aggregates is of considerable importance, since they make up about 70 to 80% of the total volume of concreter. In the present study, locally available crushed granite of angular shape and of rough surface texture with 14 mm maximum size was used as coarse aggregate while natural quartzite sand, obtained from Tronoh, Perak, was used as fine aggregate in the preparation of all test specimens. To remove fine silt particles, coarse aggregate was washed and used in saturated surface dry (SSD) condition. The physical properties of both coarse and fine aggregates are given in Table 3.5. The sieve analysis of aggregates was performed in accordance with ASTM C 136-01 [130] and the results of sieve analysis are presented in Tables 3.6 and 3.7, respectively. It can be seen from Tables 3.6 and 3.7 that both the coarse and fine aggregates complied with the grading requirements as proposed by BS 882: 1992 [131].

Table 3.5 Physical Properties of Coarse and Fine aggregate

Property	Coarse aggregate	Fine aggregate
Type	Crushed	Uncrushed
Maximum size (mm)	14	4.75
Specific gravity	2.66	2.61
Fineness modulus	-	2.76
Water absorption (%)	0.96	1.18
Unit weight (kg/m^3)	1578	1658

Table 3.6 Sieve Analysis of Coarse aggregate

Sieve size	Weight retained (gm)	Percentage weight retained	Cumulative percentage weight retained	Percentage passing	BS 882: 1992 range [131]
20.0 mm	0	0	0	100	100
14.0 mm	116.81	5.88	5.88	94.12	90-100
10.0 mm	798.55	40.20	46.08	53.92	50-85
5.00 mm	995.40	50.11	96.19	3.81	0-10
2.36 mm	20.47	1.03	97.22	2.78	-
Pan	55.20	2.78	100	-	

Table 3.7 Sieve Analysis of Fine aggregate

Sieve size	Weight retained (gm)	Percentage weight retained	Cumulative percentage weight retained	Percentage passing	BS 882: 1992 range [131]
5.00 mm	0	0	0	100	89-100
2.36 mm	157.13	15.85	15.85	84.15	60-100
1.18 mm	172.79	17.43	33.28	66.72	30-100
600 μm	141.46	14.27	47.55	52.45	15-100
300 μm	325.56	32.84	80.39	19.61	5-70
150 μm	183.89	18.55	98.94	1.06	0-15
Pan	10.51	1.06	100	-	

3.2.5 Alkaline Solution

In geopolymerization, high alkaline solutions are required to activate the silicon and aluminium present in the source material to dissolve and form geopolymeric paste. The most common alkaline solution used in geopolymerization is a combination of sodium hydroxide or potassium hydroxide and sodium silicate or potassium silicate [1]. In the present study, a combination of sodium hydroxide and sodium silicate was chosen as the alkaline activator. Sodium-based solutions were preferred because they are cheaper than Potassium-based solutions. In addition, compared to potassium hydroxide, sodium hydroxide promotes higher dissolution of fly ash particles [1].

3.2.5.1 Sodium hydroxide

Sodium hydroxide in pellets form with 99% purity, supplied by Quick Lab Sdn Bhd, Malaysia was used. Sodium hydroxide solution was prepared by dissolving sodium hydroxide pellets in potable water. Figure 3.1 shows the sodium hydroxide used in this study.

Figure 3.1 Sodium hydroxide Pellets

3.2.5.2 Sodium silicate

Sodium silicate (Figure 3.2), Grade A53 in solution form with SiO_2-to-Na_2O ratio by mass of approximately 2 supplied by Malay-Sino Chemical Industries Sdn Bhd,

Malaysia was used in this study. The chemical composition of the sodium silicate solution was Na2O = 14.26%, SiO2 = 29.43% and water = 56.31% by mass.

Figure 3.2 Sodium silicate Solution

3.2.5.3 Preparation of Alkaline solution

Alkaline solution was prepared by mixing sodium hydroxide and sodium silicate solutions. As proposed by Hardjito and Rangan [1], the ratio of sodium silicate to sodium hydroxide by mass was taken as 2.5. The sodium hydroxide solution was prepared by dissolving sodium hydroxide pellets in potable water. The mass of sodium hydroxide solids in a solution varied depending on the concentration of the solution expressed in terms of molar, M. For example, to prepare 1 kg of sodium hydroxide solution with a concentration of 8 M, 294 grams of sodium hydroxide pellets were diluted with 706 grams of water. Similarly, the mass of sodium hydroxide solids per kg of the solution for other concentrations were measured as 10 M: 367 grams, 12 M: 441 grams, and 14 M: 514 grams. Both the liquid solutions were then mixed together and an alkaline solution was prepared.

3.2.6 Superplasticizer

Superplasticizer is an admixture used in concrete mixtures to increase the workability of concrete. In order to attain higher workability and required flowability of the fresh SCGC, a commercially available polycarboxylic superplasticizer, under the brand

name of Sika Viscocrete-3430 (Figure 3.3) supplied by Sika Kimia Sdn Bhd, Malaysia, was used in the SCGC mixes. Superplasticizer had the density of 1.11 kg/litre and pH value of 6.5.

Figure 3.3 Superplasticizer

3.2.7 Water

Water is the most important and least expensive ingredient of concrete and plays a significant role in the concrete mix. Water that is fit for drinking is generally considered satisfactory for use in mixing concrete. In this research study, the tap water available in the concrete laboratory was used in the preparation of concrete mixes.

3.3 Development of Suitable Mix Proportion for SCGC

3.3.1 Design of Mix Proportions

Most of the mix design procedures available for SCC are based on the assumptions and trial & error approaches. In this study, the manufacture of SCGC was also carried out using the traditional trial & error method. Based on the knowledge available in the literature on the mix proportions of geopolymer concrete and SCC, the proportions of constituents for SCGC mixtures were selected. In the beginning numerous trial

mixtures (Appendix A) were prepared. However, to develop a suitable mix proportion for fly ash-based SCGC, a total of seventeen mixtures using constant fly ash content (400 kg/m^3) were formulated by varying the mix parameters such as amount of extra water, curing time, curing temperature, dosage of superplasticizer and concentration of sodium hydroxide. The details of these mixtures are given in Table 3.8. The coarse and fine aggregates constituted about 75% by mass in the mixes. For each mix, the activator to fly ash ratio was kept 0.5 whereas the ratio of sodium silicate to sodium hydroxide solution by mass was kept 2.5 for all mix proportions. Each mix was tested for self-compactability and compressive strength. Finally, a suitable mix proportion fulfilling the self-compactability criteria and having targeted compressive strength was selected for further investigation.

Table 3.8 Details of Mix Proportions

Mix Code	Fly Ash	Fine Aggregate	Coarse Aggregate	Sodium Hydroxide		Sodium Silicate	Alkaline/ Fly ash	Super plasticizer		Extra water		Curing	
												Time	Temp.
	kg/m^3	kg/m^3	kg/m^3	kg/m^3	Mol.	kg/m^3	Ratio	kg/m^3	%	kg/m^3	%	hrs	°C
M_1	400	850	950	57	12	143	0.5	28	7	40	10	24	70
M_2	400	850	950	57	12	143	0.5	28	7	48	12	24	70
M_3	400	850	950	57	12	143	0.5	28	7	60	15	24	70
M_4	400	850	950	57	12	143	0.5	28	7	80	20	24	70
M_5	400	850	950	57	12	143	0.5	28	7	48	12	48	70
M_6	400	850	950	57	12	143	0.5	28	7	48	12	72	70
M_7	400	850	950	57	12	143	0.5	28	7	48	12	96	70
M_8	400	850	950	57	12	143	0.5	28	7	48	12	48	60
M_9	400	850	950	57	12	143	0.5	28	7	48	12	48	80
M_{10}	400	850	950	57	12	143	0.5	28	7	48	12	48	90
M_{11}	400	850	950	57	12	143	0.5	12	3	48	12	48	70
M_{12}	400	850	950	57	12	143	0.5	16	4	48	12	48	70
M_{13}	400	850	950	57	12	143	0.5	20	5	48	12	48	70
M_{14}	400	850	950	57	12	143	0.5	24	6	48	12	48	70
M_{15}	400	850	950	57	8	143	0.5	24	6	48	12	48	70
M_{16}	400	850	950	57	10	143	0.5	24	6	48	12	48	70
M_{17}	400	850	950	57	14	143	0.5	24	6	48	12	48	70

3.3.2 Mixing Procedure

In the production of SCC type of mixtures, the mixing sequence and duration are very important. Therefore, for the production of SCGC, a systematic procedure for batching and mixing was employed to achieve the homogeneity and uniformity in all mixtures. To make all the ingredients mixed properly and uniformly, a rotating pan mixer (Figure 3.4) of 100 L capacity was used. Mixing process was carried out in two stages. Initially, dry fine powdered materials such as fly ash, fine aggregate and/or silica fume were blended manually for about a min. Afterwards, coarse aggregate (SSD condition) was added to the mixer and mechanically mixed for approximately 3 min. At the end of dry mixing, a well-shacked pre-mixed liquid mixture (Figure 3.5) containing alkaline solution, superplasticizer and extra water was added in several steps while mixing and an additional 3 min of wet mixing followed. To ensure the mixture homogeneity, fresh concrete mix was hand mixed for further 1 to 2 min. The freshly prepared concrete (Figure 3.6) was then assessed for the essential workability tests required for characterizing concrete as self-compactable. Due to the highly caustic nature of the mix, appropriate safety measures were taken throughout the mixing procedure.

Figure 3.4 Pan Mixer

Figure 3.5 Liquid Mixture

Figure 3.6 Self-Compacting Geopolymer Concrete

3.3.3 Workability Tests for SCGC Mixes

The functional requirements of fresh SCC are different that required for vibrated fresh concrete, therefore, conventional workability tests are not valid for SCC. In the present study, to characterise the fresh geopolymer concrete as self-compactable, for each mix composition, tests such as slump-flow, T-50 Slump flow, V-Funnel, L-Box and J-Ring were attempted as workability tests. The details of these tests are given in the following sub-sections.

3.3.3.1 Slump Flow Test

Slump flow test (Figure 3.7) is the most widely used test for evaluating the flowability of SCC. This test was performed in accordance with EFNARC 2002 [92]. To perform this test, the slump cone was placed on a rigid and non-absorbent leveled plate and filled with fresh concrete in a single layer without tamping. Prior to testing, the inside of slump cone and the base plate were moistened. After filling the slump cone, any surplus concrete from top of the cone and around base of the cone was removed. The slump cone was then lifted vertically, allowing the concrete to flow across a smooth, horizontal surface. When the concrete patty came to rest, two orthogonal diameter measurements were made and the average of these two was recorded as the slump flow. During the slump flow test, the time from lifting the cone to when the flow spread reached a 500 mm dia circle, termed as T_{50cm} slump flow was also recorded for all mix compositions.

Figure 3.7 Slump Flow Test

3.3.3.2 V-Funnel Test

The V-Funnel test (Figure 3.8) is used to assess the viscosity and filling ability of SCC. This test was performed according to the European Guidelines for SCC, Annex B.2 [11]. To perform this test, at first, the V-Funnel apparatus was cleaned and set on the stand and the inside surfaces of the funnel were moistened. The trap door was closed and a bucket was placed under the funnel. After that, the funnel was completely filled with fresh concrete without any agitation or rodding. After filling

the funnel, the trap door was opened and the time taken for the concrete to flow out of the funnel was measured and recorded as the V-Funnel flow time.

Figure 3.8 V-Funnel Test

3.3.3.3 L-Box Test

The L-Box test is used to assess the filling and passing ability of SCC and also assess the stability of SCC mixes. This test was performed according to the European Guidelines for SCC, Annex B.3 [11]. Before commencing the test, the L-Box apparatus was set on a firm leveled ground and inside surfaces of the box were moistened. After that, the gate separating the vertical and horizontal compartments was closed and the vertical section of the box was filled with fresh concrete. The gate was then lifted and the concrete was allowed to flow through a row of reinforcing bars into the horizontal section of the box. When concrete stopped flowing and movement was ceased, the heights of the concrete at the end of the horizontal section (H_2) and in the vertical section (H_1) were measured. Figure 3.9 shows an L-Box test carried out in the laboratory.

Figure 3.9 L-Box Test

3.3.3.4 J-Ring Test

J-Ring test (Figure 3.10) is used to assess the passing ability of SCC. This test was performed in accordance with EFNARC 2002 [92]. To perform this test, the J-Ring was placed centrally on the base plate around the slump cone. The cone was filled with the concrete in a single layer without any external compaction. The surplus concrete above the top of the cone was struck off, and any concrete remaining on the base plate was removed. The cone was lifted perpendicular to the base plate, and the concrete was allowed to flow out freely to spread horizontally through the gaps between the bars. After that, the difference in height of the concrete inside and that just outside the J-Ring was measured at four locations and the average of the difference in height was recorded as the J-Ring blocking step value.

Figure 3.10 J-Ring Test

3.3.4 Preparation and Casting of SCGC Mix Specimens

After performing the workability tests, fresh concrete was poured in 100 mm x100 mm x100 mm steel moulds without any compaction or vibration and allowed to fill all the spaces of the moulds by its own weight (Figure 3.11). Prior to casting, steel moulds were cleaned and oiled properly. Care was taken that there were no gaps, so as to avoid the possibility of leakage from the slurry. After casting, the top surface of the specimens was scraped to remove excess material and smooth finish was achieved by means of a trowel. Three cubes were prepared for each test variable.

Figure 3.11 Casting of SCGC Mix Specimens

3.3.5 Curing of Test Specimens

Activation of alumino-silicate based materials with alkalis generally requires heat curing for the formation of alkali-activated binders. Heat curing substantially assists the chemical reaction that occurs in the geopolymer paste. Immediately after casting, the test specimens together with steel moulds were placed in the oven (Figure 3.12) for heat curing at a specified temperature for a specified period of time in accordance with the test variables selected. At the end of the oven curing period, the test specimens were removed from the moulds and placed in room temperature for air curing (Figure 3.13) until the specified age of testing.

Figure 3.12 Oven Curing

Figure 3.13 Air-dry Curing

3.3.6 Compressive Strength Test

Compressive strength is a primary measure used to evaluate the quality of hardened concrete. After the completion of specific curing period, the 100 x 100 x 100 mm cubical specimens were tested for compressive strength test. This test (Figure 3.14) was performed in accordance with BS EN 12390-3:2002 [132] using 2000 kN Digital Compressive & Flexural Testing Machine. The test cube was subjected to a compressive force at the rate of 3.0 kN/sec until it failed. A set of three cubes for each test variable were tested at the age of 1, 3, 7 and 28 days.

Figure 3.14 Compressive Strength Test

3.4 Design of OPC Control Mix

3.4.1 Mix Proportion

Based on the proportions of selected mix for SCGC, OPC-based control mixture was designed. All the material quantities such as binder content, proportions of fine and coarse aggregate and water/binder ratio were kept same except that OPC was used as the binder. The details of the mix proportion are given in Table 3.9.

Table 3.9 Mix Proportions

Materials	Selected SCGC Mix	OPC Control Mix
Fly ash (kg/m^3)	400	-
Ordinary Portland cement (kg/m^3)	-	400
Fine Aggregate (kg/m^3)	850	850
Coarse Aggregate (kg/m^3)	950	950
Fine Aggregate/Coarse Aggregate ratio	0.895	0.895
Sodium hydroxide (kg/m^3)	57 (10M)	-
Sodium silicate (kg/m^3)	143	-
Activator/binder ratio	0.5	-
Water/cement ratio	-	0.5
Water (kg/m^3)	48	200
Super plasticizer (kg/m^3)	24	-

3.4.2 Mixing Procedure

For the production of OPC control concrete, mixing was performed in accordance with BS1881-125:1986 [133], using a rotatory drum mixer (Figure 3.15). To begin with, the dry fine aggregate and coarse aggregate in SSD condition were placed in mixer and mixed for 30 sec. After wards, about half of the mixing water was added to the mixer and mixing was continued for further 1 min. The mix was then left for about 5 min so as both coarse and fine aggregates may absorb water. Later on, Portland cement was added in the mixer. This duration was not less than 1 min. Finally, the remaining half of the water was poured into the mixer and mixed for another 2 min. The freshly prepared concrete was hand mixed for a while and then assessed for the workability test.

Figure 3.15 Drum Mixer

3.4.3 Workability Test

After mixing, the fresh concrete was tested for its workability by slump test. This was the only test used to measure the workability of OPC-based control concrete. This test was performed following the procedure prescribed in BS EN 12350-2:2000 [134]. To perform this test, the cone was placed on a firm and smooth surface and filled with freshly mixed concrete in three layers. Prior to test, the inside of the mould and its base were moistened in order to reduce the influence of surface friction on the slump result. Each layer was tamped 25 times with tamping rod. After the last layer, the top

surface was struck off by means of a shredding and rolling motion of the tamping rod. Immediately after shredding, the cone was lifted slowly and the difference in the height of the slumped concrete and top surface of the cone was measured to the nearest 5 mm as its workability. Figure 3.16 shows the measurement of the slump test.

Figure 3.16 Workability Test for OPC-based Control Concrete

3.4.4 Casting and Curing of OPC Control Mix Specimens

After performing the slump test, specimens were cast for determining the compressive strength. The greased moulds were filled with concrete in three layers and compaction was done by poker vibrator (Figure 3.17). After casting, the moulds were left in the casting room for 24 hrs. The OPC control specimens were removed from their moulds after 24 hrs of air curing and put into the water tank (Figure 3.18) at room temperature until the desired day of testing.

Figure 3.17 Casting of OPC Control Mix Specimens

Figure 3.18 Curing of OPC Control Mix Specimens

3.5 Evaluation of Physical and Mechanical Properties of Selected SCGC and OPC Control Mixes

3.5.1 Physical Properties

3.5.1.1 Density

The density of concrete is a measure of its solidity. Probably the easiest and accurate way to calculate the concrete density is to measure some into a container of known volume and weighing it. In this study, the density of both SCGC and OPC mix specimens was determined in accordance with BS EN 12390-7:2009 [135] by weighing six 100 x 100 x 100 mm cubical and six 100 x 200 mm cylindrical specimens after demolding them. The density of each sample was calculated by dividing the weight of specimen to its volume.

3.5.1.2 Water Absorption

To know the relative porosity or permeability characteristics of concrete, water absorption test is carried out. To determine the water absorption characteristics of SCGC and control OPC mix, three 100 x 100 x 100 mm cubical and three 100 x 200 mm cylindrical specimens from each mix were tested at the age of 28 days. This test

was performed in accordance with ASTM C 642-96 [136]. The test specimens were initially placed in the oven at a temperature of 105°C for a period of 24 hrs (Figure 3.19). After that the specimens were taken out from the oven and allowed to cool to room temperature and weighed. The samples were then immersed in a water bath (Figure 3.20) at approximately 25°C for not less than 48 hrs. After removing from the water bath, the specimens were surface-dried and weighed. The water absorption was obtained by using the following expression 3.1.

$$Absorption\ (\%) = [(W_2 - W_1)/ W_1]\ x100 \qquad (3.1)$$

where W_1 = weight of specimen after complete drying at 105°C, and

W_2 = weight of surface dry sample after immersion in water for 48 hrs

Figure 3.19 Concrete Specimens placed in the Oven

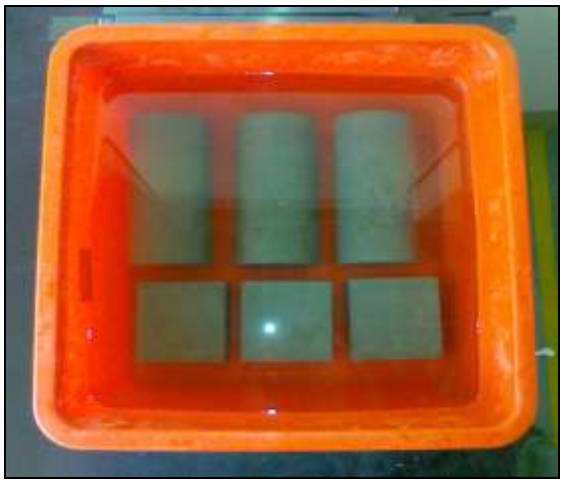

Figure 3.20 Specimens soaked in Water bath

3.5.2 Mechanical Properties

The mechanical properties which are generally considered to be the most important are compressive strength, flexural and tensile strength, modulus of elasticity, and creep and drying shrinkage. To study the mechanical behaviour of SCGC, all of these tests were performed.

3.5.2.1 Compressive Strength

As described in section 3.3.6, this test was performed in accordance with BS EN 12390-3:2002 [132] using 2000 kN Digital Compressive & Flexural Testing Machine. At the end of appropriate curing regime, three 100 x 100 x 100 mm concrete cubes from each formulation were tested at the ages of 3, 7 and 28 days after casting. Compressive strength test at the ages of 90 and 180 days were also carried out to examine the strength development of hardened concrete.

3.5.2.2 Tensile Strength

Tensile strength of concrete can be measured by three different test methods, namely direct tensile test, the modulus of rupture test and the splitting cylinder test. Due to difficulty in applying direct tension to concrete specimen, the split cylinder test and the modulus of rupture tests are widely used. In the present study, tensile strength of concrete specimens was measured by performing the cylinder splitting test on 100 x 200 mm concrete cylinders, following the procedure prescribed by BS EN 12390-6:2000 [137]. The cylinder was loaded uniformly on two diametrically opposite lines at a rate of 0.94 kN/sec without any sudden shock loads. The aligning jig (Figure 3.21) was used to prevent the specimen from rolling to the sides during the test setup. It was also helpful to position the specimen directly beneath the centre of the thrust of the spherical bearing block of the testing machine. Before the application of the load, two hardboard-packing strips of dimensions 38 cm x 1.4 cm x 0.4 cm were placed at both the top and bottom of the specimen to distribute the load along the length of the cylinder. The two diametral lines drawn earlier on the ends of the specimen were used to align the axial plane of the specimen with the vertical axis of the uprights. Later on,

the whole assembly was placed in the testing machine. Two cylindrical specimens from each mix were tested at the ages of 3, 7, 28 and 90 days after casting. Figure 3.22 shows the test set up for tensile strength.

Figure 3.21 Specimen in the Jig

Figure 3.22 Splitting Tensile Strength Test

3.5.2.3 Flexural Strength

Flexural strength, also known as modulus of rupture, is a measure of plain concrete beam to resist failure in bending. Flexural strength is determined either by third-point loading or centre-point loading test methods. Centre-point loading method gives a higher value for the flexural strength than the third-point method. Centre-point

loading also gives more variable values; as a result, this test method is very rarely used. In this research study, the flexural strength values were obtained using third point loading method (Figure 3.23) on 100 x 100 x 500 mm prism specimens. Flexural strength test was performed in accordance with BS EN 12390-5:2000 [138], using 2000 kN Digital Compressive and Flexural Testing Machine as shown in Figure 3.24. The specimens were placed on support blocks of the testing machine and the load was applied constantly without any shock until the specimen was failed. Duplicate set of samples from each mix were tested at the ages of 3, 7, 28 and 90 days after casting.

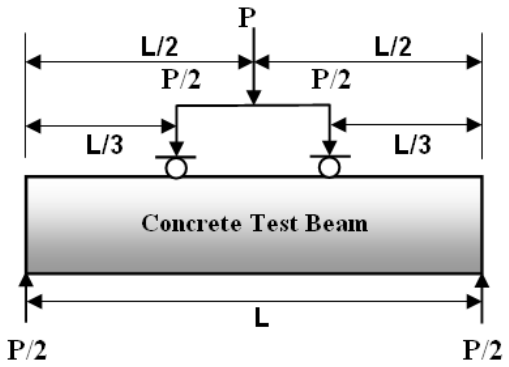

Figure 3.23 Third-point Loading Test Method

Figure 3.24 Flexural Strength Test

3.5.2.4 Modulus of Elasticity

Modulus of elasticity (also known as elastic modulus) is a measure of a material's stiffness or tendency to deform under load. The modulus of elasticity can be measured using either static or dynamic tests. Modulus of elasticity determined from an experimental stress-strain curve, is generally termed as static modulus of elasticity while the modulus of elasticity determined through the longitudinal vibration test is termed as dynamic modulus of elasticity. For determining the values for modulus of elasticity and Poisson's ratio, based on previous findings, two SCGC and one OPC-based control mixture were formulated. The details of these mix proportions are given in Table 3.10. The static modulus of elasticity was determined on 100 x 200 mm cylindrical specimens in accordance with the procedure prescribed by ASTM C 469 [139]. At the end of appropriate curing regime, four concrete cylinders (two for compressive strength and two for modulus of elasticity and Poisson's ratio) from each formulation were tested at the ages of 3, 7 and 28 days. Prior to testing the top and bottom ends of each cylinder were ground to provide a smooth surface.

Table 3.10 Mix Proportions of SCGC and OPC Control Mixes

Materials	SCGC-1 Mix	SCGC-2 Mix	OPC Control Mix
Fly ash (kg/m^3)	400	360	-
Portland Cement (kg/m^3)	-	-	400
Silica fume (kg/m^3)	0	40	-
Fine aggregate (kg/m^3)	850	850	850
Coarse aggregate (kg/m^3)	950	950	950
Sodium hydroxide (kg/m^3)	57	57	-
Sodium hydroxide Concentration (M)	10	10	
Sodium silicate (kg/m^3)	143	143	-
Water/geopolymer solids ratio	0.33	0.33	-
Activator/binder ratio	0.5	0.5	-
Water/cement ratio	-	-	0.5
Water (kg/m^3)	48	48	200
Superplasticizer (%)	6	6	-
Curing time (hrs)	48	48	-
Curing temperature (°C)	70	70	-

3.5.2.4.1 Test Procedure

For determining the values for modulus of elasticity and Poisson's ratio of the designed concrete mixes, two Linear Voltage Differential Transducers (LVDTs) were used to measure the lateral and longitudinal deformations of the concrete specimen during the test. The setup also consisted of a universal compression testing machine and a computer data acquisition system to download the data from the test. To perform the test, the specimen was placed centrally on the lower platen of the testing machine. Using concentric circles inscribed on the lower platen, the axis of the test specimen was carefully aligned. The spherical bearing block was brought down slowly to lightly bear on the specimen. The specimen was loaded and unloaded at least two times. During the first loading and unloading cycle, no data was recorded. This was primarily done to seat the gages. The output from the load cell and the LVDTs were recorded by a data acquisition system and the data for load and displacement was obtained. From the displacement readings, strain values were calculated while the readings from the load cell were used to calculate the stress. A plot of stress Vs strain was developed, and the modulus of elasticity was determined using the following expression 3.2.

$$E = \sigma_2 - \sigma_1 / \varepsilon_2 - 0.000050 \quad (3.2)$$

where,

E = Young' modulus of elasticity, MPa

σ_2 = Stress corresponding to 40% of the ultimate load or ultimate stress, MPa

σ_1 = Stress corresponding to a longitudinal strain of 0.000050, MPa

ε_2 = Longitudinal strain produced by stress σ_2

Before modulus of elasticity test was performed, the companion specimens were tested for compressive strength, in order to calculate the 40% of the ultimate compressive strength. The test set-up for measuring the elastic constants is shown in Figure 3.25.

Figure 3.25 Modulus of Elasticity Test

3.5.2.5 Poisson's Ratio

When a concrete cylinder is loaded in compression, it experiences a shortening in the longitudinal direction and an expansion in the transverse direction. In other words, a longitudinal strain is produced in the direction of the applied load and at the same time a lateral stain of opposite sign is also produced. The ratio of this lateral strain to the longitudinal strain is called Poisson's ratio.

Poisson's ratio for all three mixes was measured and calculated following the procedure outlined in ASTM C 469 [139]. The data required for Poisson's ratio was obtained simultaneously while collecting the data for modulus of elasticity. The values for Poisson's ratio were calculated using the equation 3.3.

$$\mu = (\varepsilon t_2 - \varepsilon t_1) / (\varepsilon_2 - 0.000050) \quad (3.3)$$

where,

μ = Poisson's ratio

εt_2 = average transverse strain at stress σ_2

εt_1 = average transverse strain at stress σ_1, and

ε_2 = longitudinal strain corresponding to the stress σ_2.

3.5.2.6 Creep

All concretes undergo long-term deformations known as creep and shrinkage. Creep is defined as the time-dependent deformation resulting from a sustained stress. It is usually determined by measuring the change with time in the strain of specimens subjected to a constant load and stored under appropriate conditions. As for the modulus of elasticity and Poisson's ratio test, the same three mixes SCGC-1, SCGC-2 and OPC control (Table 3.10) were used for creep and drying shrinkage test. Creep strains were determined by using 150 x 300 mm cylinder specimens. Eight cylinders were prepared from each mix. Three of these cylinders were used for measuring the creep, and three were used as companion specimens to measure the drying shrinkage. The other two cylinders were utilised for compressive strength test. After casting, the SCGC mix specimens along with moulds were placed in the oven at 70°C for 48 hrs while OPC control mix specimens were left in the casting room for 24 hrs. After initial curing, the SCGC mix specimens were removed from the moulds and placed in room temperature for air curing until 7 days whereas the OPC control mix specimens were put into the water tank for water curing at about 23°C until the age of 7 days. Prior to the commencement of the test, the creep specimens and the companion shrinkage specimens were attached with Demountable Mechanical (DEMEC) gauge points. Each cylinder has four gauge points, with two on each diametrically opposite side, separated by 200 mm as shown in Figure 3.26. In addition to test specimens, ASTM C 512 [140] requires plugs to be used above and below the creep testing specimens while they are in the creep frames. This helps to ensure an even stress distribution across the actual creep testing specimen. For this purpose, six 150 mm x 300 mm concrete cylinders (one cylinder for each frame) were cut in half and attached to the test specimens (Figure 3.27). The creep tests were conducted in a laboratory room where the temperature was maintained at about 23°C, but the relative humidity could not be controlled. The relative humidity of the room varied between 56% and 64% during the test.

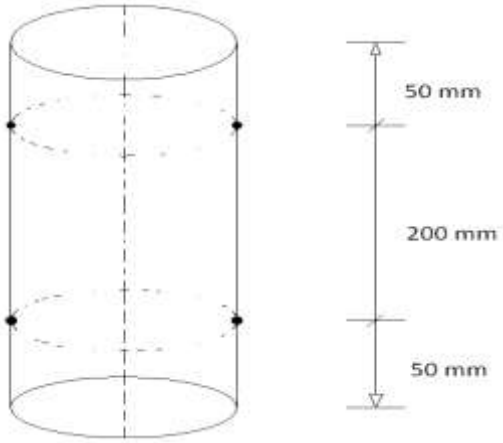

Figure 3.26 Location of Demec Gauge Points on Test Cylinders

Figure 3.27 Creep Test Specimens

3.5.2.6.1 Creep Frames

This study utilized six specially built creep frames in order to test each of the three concrete mixtures of interest. ASTM C 512 [140] provides a basic description of the layout of the required creep frame. It specifies that the frame must be capable of maintaining the applied load within ±2% of the target load even as length change occurs within the test specimens. To do this, the specification suggests the use of springs, which need to be flexible enough to allow reasonable amounts of length change to occur before any significant reduction in load occurs. The springs are

sandwiched between two steel plates and the load applied onto top loading plate is transmitted to the loading plate in the middle through the springs that transfer the applied load into the test specimens. It is a self-equilibrating system where the springs maintain the load when the specimens experience creep. The frames are reloaded periodically to ensure that the specimens stay under constant load. Figure 3.28 shows a schematic of the creep frame with the working principle of the creep frames.

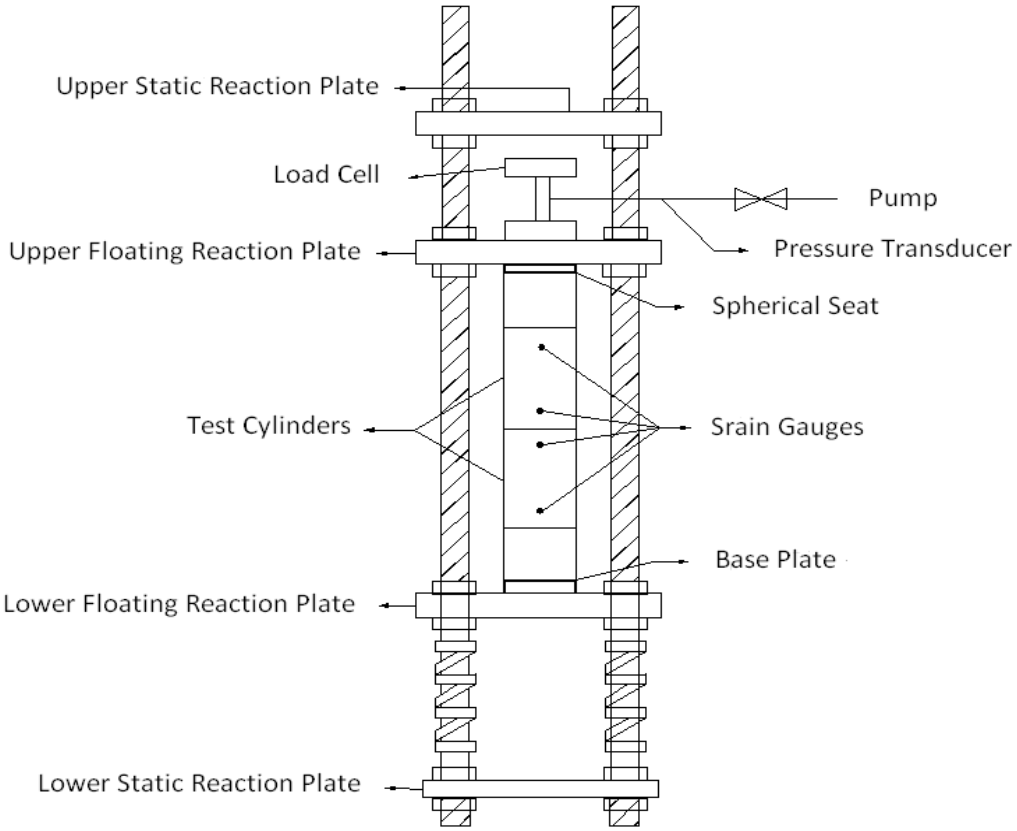

Figure 3.28 Schematic Diagram of Creep Test Frame

3.5.2.6.2 Test Set up and Measurements of Creep Strains

Creep tests were started on the 7th day after casting of the specimens. After the appropriate curing regime for each set of specimens, the test specimens for creep test were stacked in a specially built creep testing frames as shown in Figure 3.29. The creep testing was done in accordance with ASTM C 512 [140]. Before the creep specimens were loaded, the 7th day compressive strength of concrete was determined by testing the two cylinders reserved for the compressive strength test. As required by

the specification, the creep specimens were loaded to 40% of the measured mean compressive strength. This load was maintained as the sustained load throughout the duration of the test. The creep frames were able to sustain the applied load on the cylinders through the use of springs. The creep and drying shrinkage strains were measured by using a DEMEC strain gauge measuring device of 200 mm guage length (Figure 3.30) with DEMEC points fixed on the specimens. The strain readings were taken immediately before and after the loading and then at 2 hrs and 6 hrs after loading. The strain values were then recorded daily in the first week, once a week until the fourth week, once in 2 weeks until the twelfth week and once every four weeks until one year. Strains on companion shrinkage specimens were measured at the same time intervals as the creep strain readings and were taken immediately following the creep measurements. Specimens used for creep and shrinkage testing remained unsealed for the duration of the test, so the creep and shrinkage strains included their autogenous and drying shrinkage and their basic and drying creep portions. The non-loaded specimens underwent only shrinkage while the loaded ones underwent both creep and shrinkage. The loaded specimens included elastic, creep and shrinkage deformations. The elastic portion was captured between the measurements before and immediately after loading. Creep strains were obtained by subtracting shrinkage strain measured on unloaded specimens from the total strain of the loaded specimens. Figure 3.31 shows the test setup for measuring the creep strains.

Figure 3.29 Creep Frames with Specimens

Figure 3.30 Strain Measuring Device

Figure 3.31 Creep Test Set up

3.5.2.7 Drying Shrinkage

Shrinkage is the decrease in volume which is primarily caused by the loss of water during the drying process [13]. Of all the various types of shrinkage, drying shrinkage usually results in the largest volume change. The drying shrinkage of concrete is generally measured following the procedure prescribed by ASTM C 157 [141]. In this method, concrete specimens are tested over a given period of time by measuring the length change under a constant environment. For the present study, in addition to the cylinders for creep test as described in section 3.5.2.6, two rectangular concrete

prisms measuring 76 x 76 x 285 mm were cast from each mix and tested in accordance with ASTM C 157 [141]. Steel studs were embedded at the ends of each prism as shown in Figure 3.32.

Figure 3.32 Specimens for Drying Shrinkage Test

3.5.2.7.1 Test Procedure

The drying shrinkage measurements started on the 7th day after casting the concrete. The prismatic specimens for the drying shrinkage test were kept in the same environment as the creep and shrinkage cylinders, and measured at the same time increments. A mechanical dial gauge comparator (Figure 3.33) was used to measure shrinkage strains. Comparator readings for each specimen were carried out daily in the first week, followed by once a week until the fourth week, once in two weeks until the twelfth week, and then once in four weeks until one year. Figure 3.34 shows the shrinkage specimens kept in the testing room.

Figure 3.33 Mechanical Dial Gauge Comparator

Figure 3.34 Drying Shrinkage Test Set up