CHAPTER 3

EXPERIMENTAL INVESTIGATIONS

3.1. Overview of Chapter

This chapter mainly discusses the experimental approaches which are used in this research. The main focus of this research is the mix design procedures that were conducted to utilize the full efficiency of the constituent materials so that the ultimate concrete would be a green concrete. Figure 3.1 shows flow of the project.

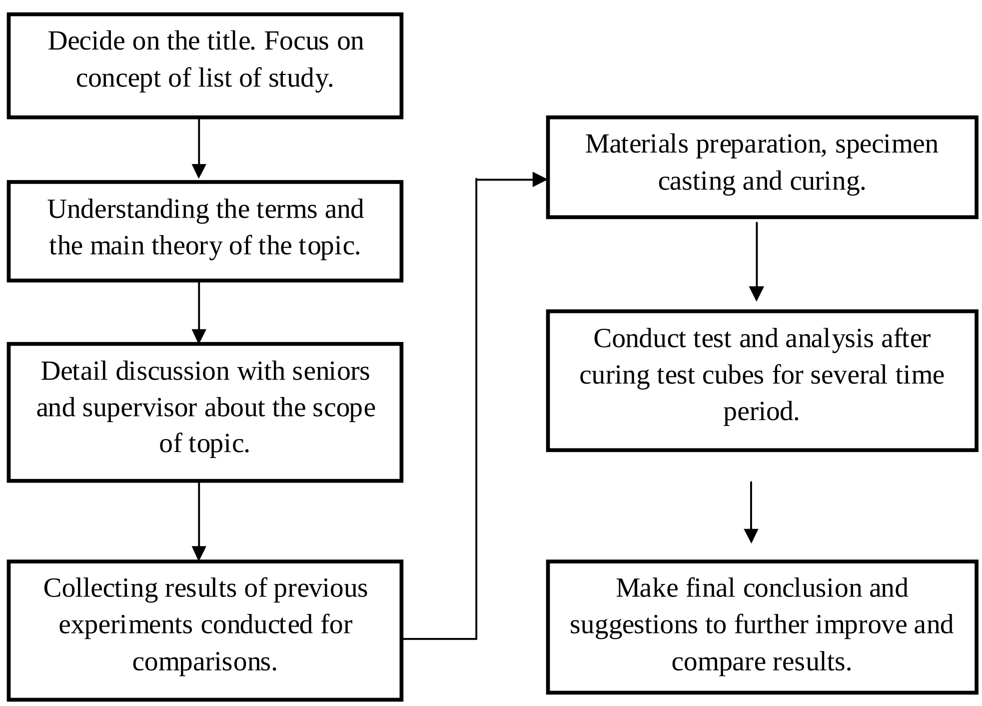

Figure 3.1: Project Flowchart

3.2. Concrete Mix Designs.

The concrete mix design process aims to determine the optimum proportions of the components of concrete in order to achieve the intended requirements throughout the life of concrete structure. For developing concrete mix design, three important stages of concrete are considered; the fresh concrete, the early age of hardened concrete of first 28 days and the entire service life.

These three stages of concrete are accessed by the five clusters of the concrete properties which consist of workability, consistency, density, strength and durability. These five clusters have complex and non-linear relationship among each other. Due to the number of complexities involved, a trivial approach for mix design proportion is adapted that is based on material selection and their properties, types and nature of construction and many other considerations.

For this research, a trial mix design approach was adapted focused to maximize the efficiency of all constituent materials such as aggregates, cement, and additives. With that approach, the end product (concrete) was obtained as the environmental friendly and green concrete. In the following sub-sections, material properties and trial mix design procedures are discussed.

3.2.1. Material Properties and Selections

In order to achieve the highest efficiency of concrete and all its constituents, the experimental investigation is based on 5 components of the concrete system as below:

1. Aggregates (Fine aggregates and coarse aggregates)
2. Cement (Ordinary Portland Cement (OPC) Type 1)
3. Cement Replacing Materials (CRM). For this research, Silica Fume (SF) was chosen as the CRM.
4. Water
5. Superplasticizer

The properties and the characteristics of the materials used are discussed in the next sections. Materials used in this research were selected according to the specifications that were suitable with the objectives of this research and meet the requirement of the appropriate standards that is the British Standards and ASTM Standards.

3.2.2. Trial Mix Proportions.

The main focal point of the mix proportion was to maintain the maximum cement content within the range of 250 kg/m³-400 kg/m³ obtained from various research catering for lower and higher range of cement consumption that would be able to achieve 28 days cube strength between 50 MPa-80 MPa as it was highly demanded by contractors in the construction industry. The details of the trial mix proportions are given in Table 3.1.

Table 3.1: Details of Trial Mix Proportions

Mix Series	OPC (kg/m³)	FA (kg/m³)	CA (kg/m³)	WATER (kg/m³)	SF (%)	SP (%)
250CM	250	860	1290	125	0	3
250SF5	250	860	1290	119	5	3
250SF10	250	860	1290	113	10	3
275CM	275	850	1275	138	0	3
275SF5	275	850	1275	131	5	3
275SF10	275	850	1275	124	10	3
350CM	350	840	1260	175	0	3
350SF5	350	840	1260	166	5	3
350SF10	350	840	1260	158	10	3
400CM	400	830	1245	200	0	3
400SF5	400	830	1245	190	5	3
400SF10	400	830	1245	180	10	3

From the trial mix proportions, each type of concrete mixes included a control mix which contains 100% OPC, 5% SF and 10% SF. The dosages were chosen based on several literature reviews and previous research conducted so to confirm with the objectives of this research. The materials mainly cement (OPC Type 1), sand (fine aggregates) and gravels (coarse aggregates) was proportioned by weight ratio of 1:2:3. The w/c used was 0.5 (by weight) throughout the research to look upon the overall range of performance of the concrete. SP is fixed at 3% of the total mix. The slump in this research was observed and was expected to be in the range of ±50-70 mm for HPC (Silica Fume Society Association, 2008).

3.3. Materials Preparations.

In the following sub-sections, the mechanical, physical and chemical properties of materials used in this research are discussed. Besides this, materials preparations are also discussed. The preparations included tests that were conducted according to the appropriate standards procedures.

3.3.1. Aggregates (Fine aggregates and coarse aggregates)

The coarse aggregates used were gravels of nominal maximum sizes 20mm while the fine aggregates used were natural sands having 3.35mm nominal maximum sizes. Both aggregates were selected conformed to BS882:1992. for this research. Coarse aggregates were prepared as Saturated Surface Dry (SSD) aggregates by washing the aggregates. The wet aggregates were then left in the open air and sheltered from direct sunlight for 12 hours. Figure 3.1 shows the coarse and fine aggregates that were used in the research. Fine aggregates were divided into 2 types; 'Designed' and 'As-supplied' graded aggregates which produced 'Designed' mixes and 'As-supplied' mixes.


Figure 3.2: Coarse Aggregates & Fine Aggregates

In this research, quality control is also very important into obtaining quality concrete properties. The Sieve Analysis Test, X-ray Fluorescence Spectrometry (XRF) Test and X-Ray Diffraction Spectrometry (XRD) Test were conducted. The test procedures were discussed for each test.

3.3.1.1. Sieve Analysis Test

For this research, the sieve analysis is conducted with accordance to BS 882: 1992 for the fine aggregates (sand) and coarse aggregates (stones, max. 20mm). The purpose of this test is to obtain well graded and finely distributed input of raw materials so to have quality mix without taking into consideration of cement content in mixes. Thus to fulfil the main objective that well graded and finely distributed aggregates affects contribution to high strength in concrete materials, two type of mixes that is the 'Designed' and 'As-supplied' mixes were prepared.

3.3.2. Chemical Composition of OPC and SF

For this research, the chemical composition is determined for OPC and SF from two ways which were:

1. X-Ray Fluorescence Spectrometry (XRF) Test.
2. X-Ray Diffraction Spectrometry (XRD) Test

The purpose of the tests was discussed in the following sub-sections.

3.3.2.1 X-Ray Fluorescence Spectrometry (XRF) Test.

XRF Test was conducted on samples of SF and OPC used throughout this research study. The purpose of this test is to determine the chemical composition and trace any harmful elements contained in the SF and OPC. Chemical compositions of both of these samples were obtained and analyzed.

The XRF machine, BRUKER AXS S-Pioneer (Refer to Figure 3.3) was used for this test which is supplied by BRUKER AXS as mentioned in S. Harun (2007).

Figure 3.3: XRF Machine (BRUKER AXS S-Pioneer)

3.3.2.2 X-Ray Diffraction Spectrometry (XRD) Test

The XRD Test was carried out using Diffractometer of Bruker Axs D8 Advance to analyze the crystalline properties of SF sample and to detect the presence of various crystal system of SiO_2 in SF. The sample was taken from SF that will be incorporated into the concrete mixture. The XRD results were used to describe the effect of SF into the concrete properties during the maturing period. The X-Ray Diffractometer used in this research in Figure 3.4.

Figure 3.4: Bruker Axs D8 Advance Diffractometer

3.3.2. Ordinary Portland Cement (OPC) Type 1

Ordinary Portland Cement (OPC) Type 1 which conformed to the requirements of BS EN 197-1 2000 was used with the physical and chemical properties listed in Table 3.2. OPC Type 1 was preferred because the observation on concrete properties can be done in normal hydration process hence the advantages of SF usage in concrete can be optimized.

Table 3.2: Physical and Chemical Properties of OPC Type 1.
(Cement Industries of Malaysia Berhad, CIMA)

Modulus	Lime Saturation Factor	0.96
	Silica Modulus	2.37
	Iron Modulus	1.58
Compressive Strength (N/mm^2)	3 Days	38
	7 Days	46
	28 Days	56
Chemical Ingredients (%)	SiO_2	19.98
	Fe_2O_3	3.27
	Al_2O_3	5.17
	CaO	63.17
	MgO	0.79
	SO_3	2.38
	Total Alkalis	0.9
	Insoluble Residues	0.2

3.3.3. Silica Fume (SF)

Silica Fume (SF) was supplied complimentary by SIKA Kimia Sdn.Bhd. The SF used was of the finest quality and no additives are added during the mixing and casting process. SF was obtained from Elkem materials in dry densified form with Grade 920 confirming to the mandatory requirements of ASTM C1240.

3.3.4. Water

The water used in the mix needed to be free from harmful chemicals, oil, chloride, silt or any harmful ingredients that could affect the performance of the concrete. The water used in the concrete mixes was tap water.

3.3.5 Superplasticizer

In this research, superplasticizer (SP) was supplied by SIKA Kimia Sdn.Bhd. used in a fixed dosage of 3%. SP used was high range water-reducing concrete admixtures that met the requirement of BSEN934-2:2001. It was polycarboxylate type, has the density of 1.11kg/l and pH value of 5.5. Figure 3.5 shows the superplasticizer that was used in this research.

Figure 3.5: Superplasticizer

3.4. Concrete Mixing and Samplings.

The procedure of machine mixing that was used in this research was in accordance to BS1881-125:1986. Mixing of concrete ingredients was performed in the laboratory using a 105 liter capacity concrete mixer. The dry ingredients which included cement, sand and gravel was first mixed for 1 minute in the mixer prior to water addition. Cement replacing material (CRM) such as Silica Fume (SF) and superplasticizer (SP) that was diluted in water were added to the dry ingredients in the mixer. After addition of the water to the dry ingredients, it was mixed for 1 minute to achieve homogeneous concrete and then left for 8 minutes. Next cement was added and mixed for 1 minute and finally the remaining water was added into the concrete mix and mixed for another 1 minute. After the fresh concrete was mixed homogeneously, it was tested for determination of slump.

3.5. Fresh Concrete Testing.

After mixing concrete, the fresh concrete was tested for finding slump as recommended by BS1881: Part 102:1983. The mix was filled into a clean truncated mould (diameter at top: 100mm, diameter at bottom: 200mm, height: 300mm) by four equal layers and each layer was rodded 25 times with a round steel rod. After the top layer was rodded, the excess concrete at the top of the mould was wiped out. Then, the mould was lifted carefully in vertical direction and the differences between the height of the slumped concrete and mould were measured as its workability. This can be observed in Figure 3.6.

Figure 3.6: Measurement of slump

3.6. Hardened Concrete Properties.

Following sub-sections elaborates on the details of hardened concrete tests performed in this study.

3.6.1. Compressive Strength Test.

Concrete cubes were cast in standard steel mould of dimension 150×150×150 mm. Concrete in moulds were laid in three layers of approximately the same thickness. After laying each layer, compaction was done by applying vibration according to the specifications defined in BS8110: 1997. Concrete specimens were tested at the age of 3, 7, 28, 56 and 120 days.

The concrete specimens in the moulds were then covered with polythene sheet to prevent evaporation and left for 24 hours. Eventually, all concrete specimens were removed from the moulds and then transferred into the water bath at room temperature for curing until the desired age of testing.

3.6.1.1. Compressive Strength Test Procedure

The main objective of this test was to obtain the concrete strength (crushing strength). Observations were made on factors of cube density and failures.

All cubes were stripped out of the moulds after 24 hours and shifted into the curing tank. The crushing test complied with the BS: 1881, Part 4, and MS 26: 1971. Prior to the test, the cubes or specimens were weighed to obtain density and placed onto the lower steel platen plate with both smooth surfaces facing the top and bottom platen plates. The load weight was applied at the rate of 5 KN/sec. until the specimen failed. Figure 3.7. illustrates the testing arrangements.

Figure 3.7: Compressive Strength Test

The load at the failure of concrete cube that is referred as the cube crushing load was recorded and divided by the area of the test surface i.e. 150 × 150 × 150 mm to calculate the compressive strength. At every age, three cubes were tested and the mean of value of strength was referred as the compressive strength.

3.6.2. Porosity Test.

The total porosity test was performed using vacuum saturation method (N. Shafiq, 1999). Plain concrete planks of 400×400×40 mm dimensions were cast in wooden moulds that were fabricated in Concrete Technology Laboratory in the department of Civil Engineering at Universiti Teknologi PETRONAS. 40 mm diameter cores were drilled out from the planks for total porosity measurement. The cores were then transferred into the water bath at room temperature for curing until the desired age of testing that was 3, 7, 28, 56 and 120 days.

3.6.2.1. Porosity Test Procedure

The equipment used to determine the total porosity was the vacuum saturation apparatus available in the Concrete Laboratory, Civil Engineering Department at Universiti Teknologi PETRONAS. The apparatus was similar to the method developed by RILEM (1984) for measuring the total porosity. The purpose of pressure saturation apparatus was to achieve total absorption or full saturation of dense mortar and concrete to enable the estimation of porosity. The apparatus is illustrated in Figure 3.8.

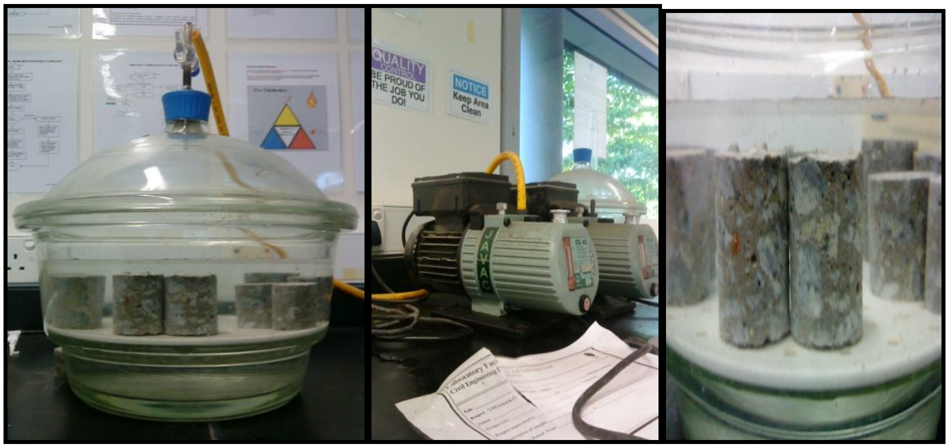

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Figure 3.8: Apparatus for Total Porosity Test

1. Vacuum Saturation Tank
2. Pump
3. Specimens (Fully Submerged condition)

The specimens were placed in vacuum for 30 minutes in a descicator, then water was added in the descicator and immediately vacuum saturated in water for 6 hours (Figure 3.7.1). After 6 hours, pump was turned off and the specimens were left for 24 hours at fully saturated condition. After 24 hours, the specimens were weighed in the air (W_{SA}) and in the water (W_{SW}). Then the specimens were dried in an oven at 105ºC for 24 hours and weighed in the air, W_d, as reported in N. Shafiq (1999). The weighing scale is shown in Figure 3.9.

Figure 3.9: Specimens weighed in air and water

3.6.2.2. Calculations of Porosity Test

The total porosity, P of concrete specimens was calculated using the following equation:

$$P = \frac{W_S - W_d}{W_S - W_W} \times 100 \qquad \text{(Eq. 3.1)}$$

Where:

P	= Total porosity (%)
W_S	= Weight of saturated samples measured in the air (g)
W_W	= Weight of saturated samples measured in water (g)
W_d	= Weight of oven dry samples measured in the air (g)

3.6.3. Split-cylinder Test (Split Tensile Strength)

Concrete cylinders of 200 mm in height and 100 mm in diameter were cast, cured and tested at the age of 28 and 120 days. The test was carried out in accordance with the ASTM C39-86 Vol. 04.02, 1986. The British Standard of BS EN12390-6:2002 was also referred. The test results were used to determine the tensile strength of concrete.

3.6.3.1. Split Cylinder Test Procedure

The split cylinder test was carried out using a standard cylinder specimen by applying a line load because the sides are not smooth enough and would induced high compressive stresses at the surface. Therefore, a narrow loading strip made of soft material was used. The application can be observed from Figure 3.10.

Figure 3.10: Split Cylinder Test

The stress at failure was taken as the tensile strength of the concrete. The specimens were tested by using Universal Hydraulic Testing Machine with a maximum capacity of 2000 kN. During the test, concrete cylinder was loaded with 9.4 KN/s constant loads without any sudden shock loads. The tensile strength of split cylinders was obtained from the testing machine.

3.6.4. Modulus of Elasticity (Flexural Tensile Strength)

Concrete prism of dimensions 500×100×100 mm were cast in standard moulds, cured and tested at the age of 28 days with two specimens for each testing age in accordance with ASTM C469-02el, 1986. The British Standard of BS 1881-109, 1989 was also referred. The results were used to determine the modulus of elasticity.

3.6.4.1.. Flexural Strength Test

The specimens were tested by using the Universal Hydraulic Testing machine with a maximum capacity of 100 kN. The results were as displayed by the machine. During the test, concrete prism was loaded with a point load of 9.4 KN/s without any sudden shock loads which is shown in Figure 3.11:

Figure 3.11: Universal Hydraulic Testing Machine

3.6.4.2. Modulus of Elasticity Calculation

Equation 3.2 was used to calculate the Modulus of Elasticity, E_f from flexural test

$$E_f = \frac{L^3 m}{4 b d^3} \quad \text{(Eq.3.2)}$$

Where:

- L = Support span, (mm)
- b = Width of test beam, (mm)
- d = Depth of tested beam, (mm)
- m = The gradient (slope) of the initial straight-line portion of the load deflection curve, (N/mm)

3.6.5. Chloride Migration Test (Durability in Marine Environment)

Concrete cubes of dimensions 100×100×100 mm were cast in standard moulds, cured and tested at the age of 28, 120, and 180 days. After 28 days, concrete samples are put in the oven to dry for 24 hours. They were then let to cool for half hour before being placed into the salt water. Salt water with salt concentration of 3% was prepared in a curing tank of water capacity of 200 gallons. Sea water from the sea can also be used. The test procedure was referred to N. Shafiq et.al (2007).

At the desired age for testing, the cubes were split into two halves. On the cut surface, silver nitrate solution was sprayed to distinguish the depth of salt water penetration.

3.6.5.1. Chloride Migration Test Procedure

For this research the test is desired to measure the chloride ingress in concrete samples with respect to time. Concrete samples of dimensions 100 × 100 × 100 mm were prepared.

The test was conducted by cutting the concrete sample into two pieces and sliver nitrate ($AgNo_3$) sprayed onto the surface of the broken samples. As shown in Figure 3.12, $AgNo_3$ will show the area of the concrete sample affected by the chloride diffusion. Chloride profiles were then measured. The results were obtained for the samples of the ages of 28, 120 and 180 days.

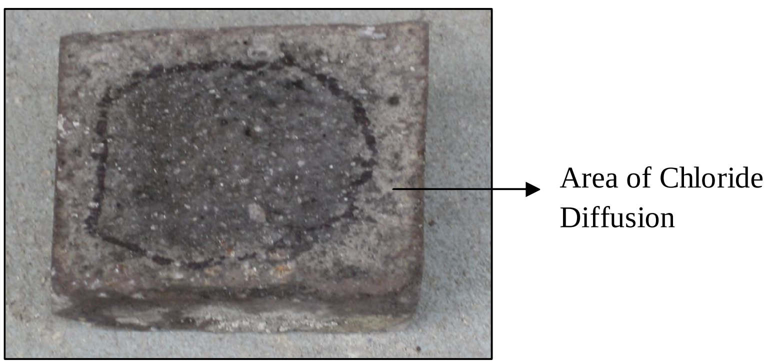

Figure 3.12: Chloride Diffusion in Concrete

The penetration depth was measured and recorded. The results were then analyzed and appropriate graphs were plotted for better comparison. The unit measurement for the depth of chloride penetration is in millimeters (mm).

3.7. WORKSHEETS APPLICATIONS

For this research, 3 worksheets are produced using the Microsoft Excel Software to help to ease in calculations and to determine the following elements;

1. The amount of cement (OPC) used in research for every mix series in tons and kilogram (kg).
2. The efficiency of research compared to other research from the amount of cement consumption for each mix series in percentage (%).
3. The amount of carbon dioxide (CO_2) emitted from each mix series in percentage (%).
4. The effectiveness of research in CO_2 emission compared to other research in percentage (%).

The worksheets are to cater for both ‘Designed’ and ‘As-supplied’ mixes. Standard units, tables and formulas are used in the calculation processes. The examples of the worksheets can be observed in Appendix A, B and C.