CHAPTER 4

EXPERIMENTAL PROCEDURES

4.1 EXPERIMENTAL DESIGN

The purpose of this research is to find the suitability of quarry dust as an alternative material for the river sand in concrete making and to determine its effectiveness in mechanical and durability properties. Also it is proposed to experimentally determine the effects of mineral admixtures and inhibitors in further enhancing the strength and durability properties of quarry dust concrete. The investigations are proposed to be carried out in four phases.

Phase I

In this phase, the fresh concrete properties, mechanical strengths and corrosion resistance properties of concrete containing quarry dust as fine aggregate will be experimentally determined and compared with the conventional concrete containing river sand as fine aggregate.

Phase II

In this phase, the influence of fly ash on strength and corrosion resistance properties by partial replacement (10%, 20%, 30%, 40% and 50%) of cement in quarry dust concrete will be studied and the optimum percentage of fly ash will be arrived.
Phase III

In this phase, the significance of partial replacement of cement by ground granulated blast furnace slag in quarry dust concrete will be studied. Cement will be partially replaced by various percentages of GGBFS (10%, 20%, 30%, 40% and 50%) and the effects on the strength and corrosion resistance properties of quarry dust concrete will be studied. The optimum percentage of GGBFS to be added for obtaining maximum strength and corrosion resistance will be arrived.

Phase IV

In this phase, some of the organic and inorganic inhibitors at different percentage levels (1%, 2%, 3% and 4%) will be added to quarry dust concrete and their effects on the various mechanical strengths and corrosion inhibition will be studied. The organic inhibitors proposed to be used are triethanolamine, diethanolamine and diethylamine and the inorganic inhibitors to be adopted are calcium nitrite, calcium nitrate and sodium nitrate. The optimum dosage to be added will be determined individually for each inhibitor.

4.1.1 Details of the Tests to be Conducted in all Four Phases

1. Tests on fresh concrete
   i) Slump test
   ii) Compacting Factor test
   iii) Vee Bee test

2. Strength tests
   i) Compressive strength test
   ii) Split tensile strength test
   iii) Flexural strength test
   iv) Bond strength test
3. Microstructural properties
   i) Water absorption
   ii) Percentage of voids
   iii) Bulk density

4. Corrosion tests
   i) Impressed voltage test
   ii) AC impedance technique
   iii) Weight loss measurement
   iv) Scanning Electron Microscopy

4.1.2 Details of the Number of Concrete Specimens to be Cast for Each Test

A minimum of three specimens will be cast for testing at a time for any test and the average value obtained by testing the specimens will be considered. A total number of 438 cubes of size 150mm for compressive strength test, 438 cylinders of 150mm diameter and 300mm long for split tensile strength test, 438 numbers of beams of size 500mm x 100mm x 100mm for flexural strength test, 438 numbers of 150mm diameter and 300mm long concrete cylinders with centrally placed steel rod of diameter 16mm and length 70mm, 108 cubes of size 150mm for water absorption, 108 cubes of size 150mm for determining percentage of permeable voids, 108 cubes of size 150mm for bulk density and 396 numbers of 75mm diameter and 150mm long concrete cylinders with centrally placed steel rod of diameter 16mm and length 150mm for corrosion tests has been proposed to cast for the conduct of all the experiments. Table 4.1 shows the details of the specimens required for each test.
Table 4.1 Number of specimens to be cast

<table>
<thead>
<tr>
<th>Identification</th>
<th>Cubes for Comp. test</th>
<th>Cubes for split tensile test</th>
<th>Beams for flexural test</th>
<th>Cylinders for bond strength</th>
<th>Cubes for water absorption test</th>
<th>Cubes for % of permeable voids</th>
<th>Cubes for bulk density</th>
<th>Cylinders for corrosion tests</th>
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<tr>
<td>Conventional concrete</td>
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<td>Quarry dust concrete</td>
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<td>Fly ash blended quarry dust concrete</td>
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<tr>
<td>GGBFS blended quarry dust concrete</td>
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<td>Quarry dust concrete with calcium nitrate</td>
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4.2 TESTS ON FRESH CONCRETE

The definition of workability given in ACI: 116R-90 is ‘the property of freshly mixed concrete or mortar which determines the ease and homogeneity with which it can be mixed, placed, consolidated and finished’. In essence, workability is defined as the ease of placement with resistance to segregation. Therefore, the workability of concrete is associated with terms such as flow ability, mobility, stability, resistance to segregation, and pumpability. Each of these terms has a specific meaning but all of them are related to workability.
The workability of various concrete mixes used in this study was measured by,

i. Slump test

ii. Compacting Factor Test

iii. Vee Bee Consistometer Test

The detailed procedures of the above tests are given in appendix 1

4.3 STRENGTH TESTS

4.3.1 Compressive Strength

The compressive strength of concrete is one of the most important tests conducted on hardened concrete because most of the desirable characteristic properties of concrete are qualitatively related to its compressive strength. The compression test is carried out on cubic specimens of size 150mmx150mmx150mm as per IS: 10086-1982 in compression testing machine (CTM) of 2000kN capacity shown in Figure 4.1.

![Figure 4.1 Compression test](image)

4.3.2 Split Tensile Strength

This test is an indirect tensile strength test. In this test, the tensile strength of concrete was determined by applying diametrically opposite
compressive forces on a plane passing through the center of the cylinder in an universal testing machine (UTM). Split tensile strength test was carried out on cylindrical specimens of 150 mm diameter and 300 mm length as shown in Figure 4.2.

4.3.3 Flexural Strength

Beams of size 500 x 100 x 100 mm were cast for determining the flexural strength of concrete. The testing is carried out in UTM of capacity 40T and the system of loading is two point loading as per IS 516-1959. The experimental setup is shown in Figure 4.3.
4.3.4 Bond Strength

Bond strength between paste and steel reinforcement is of considerable importance and is mainly dependent on the blending materials used in making concrete. The corrosion rate has a direct effect on the bond strength and hence, it is one of the chief factors to be considered. Cylinders of size 150mm diameter and 300 mm length with high yield strength deformed (HYSD) rods of 70cm length placed at the centre, were used for determination of bond strength. Bond strength is given by the formula $P/A$, where $P$ is the ultimate load and $A$ is the total area of contact of the rod with concrete i.e. surface area and cross sectional area.

![Figure 4.4 Bond strength test](image)

The detailed procedures of the above tests and specimen cast are given in appendix.

4.4 MICRO STRUCTURAL PROPERTIES

Micro structural properties like water absorption, percentage of permeable voids and bulk density of concrete were also determined.
4.4.1 Water Absorption (ASTM-C642-81)

Water absorption of hardened concrete specimens was calculated based on ASTM C642-81. Cubes of size 150X 150 X 150mm were cast and after 28 days curing the cubes were taken out and dried in an oven at 105°C for 24 hours. The dried specimens were cooled to room temperature (25°C), weighed accurately and noted as dry weight \( W_d \). Dry specimens were immersed in water and the weight of the specimens at predetermined intervals was taken after wiping the surface with dry cloth. This process was continued till constant weight is obtained in two successive observations \( W_w \).

The percentage of water absorption is given by,

\[
W = \frac{(W_w - W_d)}{W_d} \times 100,
\]

where \( W_w \) is the weight of the saturated specimen and \( W_d \) is the weight of the dried specimen.

Figure 4.5 Water absorption test set up

4.4.2 Percentage of Voids

The cube specimens after 28 days of water curing were dried in an oven at a temperature of about 105°C for 24 hours. The specimens were allowed to cool in dry air and weighed. This procedure was repeated until the
difference between the two successive readings did not exceed 0.5% of the lowest weight obtained. This weight was designated as A.

The specimens were then placed in a container filled with tap water and boiled for about 5 hours and were allowed to cool by natural loss of heat for not less than 14 hours to a final temperature of about 20 to 25°C. The surface moisture was removed with a towel and the specimens were weighed. The soaked, boiled, surface dried weight was designated as C. The specimens were then suspended by a wire in water and the weights were taken. This weight was designated as D.

Percentage of voids = \([\frac{(C - A)}{(C - D)}] \times 100\).

where 

A - Weight of the oven dried concrete sample.

C - Weight of the surface dried concrete sample in air after immersion and boiling

D - Weight of the concrete sample in water after immersion and boiling.

4.4.3 Bulk Density

Bulk density of the concrete specimens were calculated as follows

Bulk density of the specimen \(\gamma_D\) = weight / volume of specimen (gms/cm\(^3\))

4.5 DURABILITY TESTS

4.5.1 Impressed Voltage Test (ASTM-C876)

Impressed voltage test is based on electrochemical polarization principle. To assess the corrosion protection efficiency under accelerated test conditions, concrete cylinders of size 75 mm diameter and 150 mm length
were cast with a high yield strength deformed (HYSD) steel bar of 16mm diameter embedded centrally into it. The steel rods were cleaned with pickling acid and degreased and then embedded in such a way that a constant cover is maintained all round and also the protruding rod was insulated by PVC sleeve. After 28 days curing, all the triplicate specimens were taken out and dried for 24 hours then subjected to acceleration corrosion process in order to accelerate reinforcement corrosion. Each test specimen was immersed in the saline media (3% Sodium chloride solution). The rebar projecting at the top is connected to the positive terminals of the power pack (anode) and the stainless steel plate is connected to the negative terminal (cathode). The test specimens were subjected to a constant voltage of 6 volts from the D.C power pack. This setup forms an electrochemical cell. The experimental setup is shown in Figures 4.6 to 4.8. The applied voltage is kept constant continuously and the current response is monitored with respect to time.

Figure 4.6 Test set up for impressed voltage technique
4.5.2 AC Impedance Technique

AC impedance spectroscopy is being used as non-destructive technique for quantifying corrosion of steel rebar embedded in concrete. In this technique an AC signal is applied to the embedded rebar and the response is monitored in terms of phase shift of the current and voltage components and their amplitude. Cylindrical concrete specimens of size 75 mm diameter and 150 mm long were cast with centrally placed steel rod of diameter 12mm. The steel rod is placed in such a way that an equal cover of 31.5mm is maintained all-around. The potential of the rebar was measured periodically using a high input impedance multimeter. Impedance measurement was made using three electrode arrangements. Stainless steel electrode of size
10mmx80mm was used as an auxiliary electrode and saturated calomel electrode was used as a reference electrode. Rebar embedded in concrete acted as a working electrode. The experimental set up is shown in Figures 4.9 and 4.10.

**Figure 4.9 Test set up for AC impedance technique**

Chloride solution was used as a contacting solution to reduce the contact resistance between the electrode assembly and the concrete. A sinusoidal voltage signal of 20mV was applied over a suitable frequency range and the real and imaginary parts of the impedance values were plotted on the Nyquist plot as shown in Figure 4.11. From the Nyquist plot, the charge transfer resistance ($R_{ct}$) and double layer capacitance($C_{dl}$) values were calculated. The $C_{dl}$ values were obtained from the relationship,

$$C_{dl} = \frac{1}{2\pi f_{\text{max}}} \times R_{ct}$$
where,

\[ C_{dl} = \text{Double layer capacitance} \]
\[ R_{ct} = \text{Charge transfer resistance} \]
\[ f_{\text{max}} = \text{Frequency at } Z'' \text{ value maximum} \]

Figure 4.12 shows the image of AC impedance spectroscopy used in this study.

**Figure 4.10** AC Impedance circuit diagram

**Figure 4.11** AC impedance Nyquist plot
4.5.3 Weight Loss Measurement

Steel embedded concrete cylinders were cast with 1 to 4 percentages of inhibitors and without inhibitor. High yield strength deformed (HYSD) rods of size 16 mm diameter and 150 mm long, were immersed in the pickling solution (Hydrochloric acid + water in equal parts) for 15 minutes to remove the initial rust. The initial weight ($W_1$) of the rod was measured and embedded in the center of cylindrical concrete specimens of size 75 mm diameter and 150 mm long. The specimens were subjected to 28 days curing in fresh water. After the completion of curing period the cylinders were immersed in 3% NaCl solution under alternate wetting (3days) and drying (3days) conditions over a period of 90 days. At the end of 90 days, the cylinders were broke open and the final weight of the specimens was taken. The difference between the initial and final weight gives the weight loss of the specimen. From the weight loss, corrosion rate can be calculated using the following formula:
Corrosion rate in mmpy = 87600 \times \frac{W}{DAT}

where

W = weight loss in grams (W_1 - W_2)

D = Density of steel gm/ cm^3

A = Area of the embedded rebar in cm^2

T = Time in hours

Figure 4.13 Specimens subjected to alternate wetting and drying for weight loss measurement
4.5.4 Scanning Electron Microscopy (SEM)

SEM is a powerful tool for examining and interpreting microstructures of materials, and is widely used in the field of material science. Scanning Electron Microscopic studies have been carried on the surface of the rebar embedded in the concrete. From the embedded portion of the rebar, 2 cm length has been cut and examined using scanning electron microscope for interpreting microstructures of materials on the surface of the specimen.

\[ T = \text{Time in hours} \]

Figure 4.14 Embedded steel rods – SEM Specimens

Figure 4.15 Image of scanning electron microscope
4.6 Energy Dispersive X-ray Spectroscopy (EDX)

EDX spectra can be acquired over short time-periods and be displayed almost simultaneously, providing a near instant visual representation of the chemical analysis. Qualitative analysis determines what elements are present in a sample by identification of the peaks in the spectrum, whilst quantitative analysis is used to derive the relative abundance of the elements from their corresponding peak intensities, either compared to other elements present in the spectrum or to standards. In this study, EDX analysis was used mainly to determine the bulk composition of the sample materials.