CHAPTER 3:
MATERIALS, SAMPLE PREPARATION AND METHODOLOGY

3.1 GENERAL

This chapter exhibits the materials used, preparation of samples and experimental methodology to consider the impacts of admixing RSA and MS on the properties of various cementitious systems. The present investigation aimed to partially replace cement with RSA or the composite of RSA and MS, which can be utilized for making M40 grade PQC. Various approaches which were adopted to study the effects of RSA, MS and their composite on the various properties of cement paste, mortar and concrete are described in detail in the current chapter.

The methodology adopted in this research is shown in Figure 3.1 and has been divided into 6 parts, as mentioned below:

Part I - Properties of materials. The primary aim of this part was to determine the various physical and chemical properties of the materials which were used in the various experiments. It was done to validate the usage of RSA and MS as an SCM.

Part II - Physical properties of the paste. In this part, various proportions of SCMs (RSA and MS) by weight of OPC in the paste were decided based on their properties found in Part I. The primary aim of this part was to analyse the effectiveness of RSA, MS and combination of both in improving the different properties of cement paste. The mineralogical analysis was performed on these cement paste. Also, the optimum dosages of HRWR (for cement mortar with different proportions of RSA and MS) were evaluated.

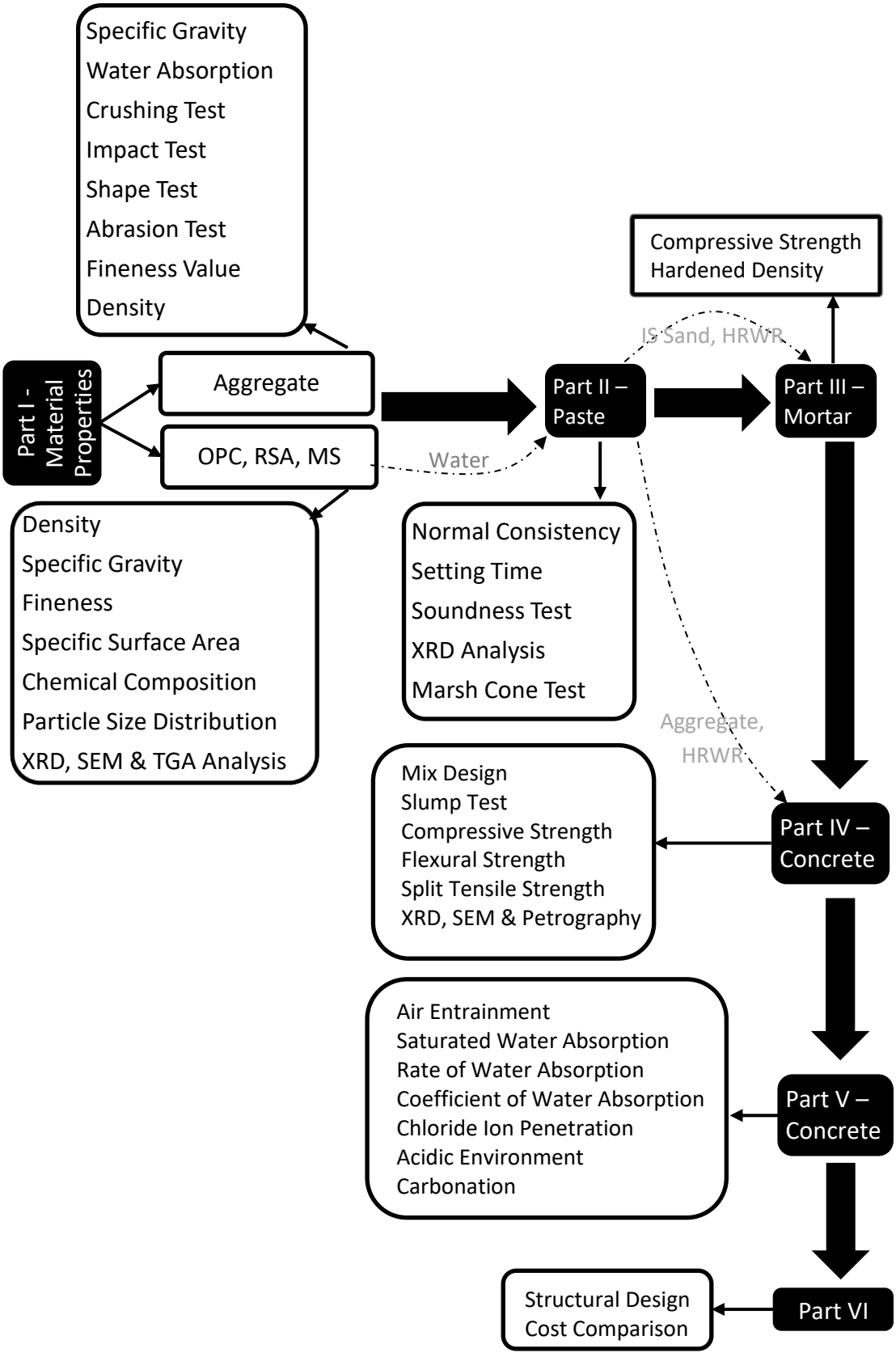


Figure 3.1 Flowchart of the Research

Part III - Physical properties of mortar. In this part, the effects of RSA and MS on physical properties of cement mortar were observed. Also, based on the results of testing on cement mortar admixed with RSA and MS, various proportions of SCMs (RSA and MS) by weight of OPC in the concrete were decided.

Part IV- Strength properties of concrete. In this part, mix design for M40 grade PQC was carried out. Also, the different dosage of HRWR for concrete with different proportion of SCMs (RSA and MS) was decided based on the slump test. The primary aim of this part was to analyse the effectiveness of RSA, MS and combination of both in improving the strength properties of concrete. Also, microstructural and mineralogical analysis of different samples of concrete was carried out.

Part V - Durability properties of concrete. In this part, the effects of RSA and MS on durability properties of concrete were observed. Also, the relations between various durability properties and age of curing of concrete were formulated based on the results of experiments.

Part VI- Structural design and cost comparison. Based on the results of experiments performed in Part I to Part V, few concrete mixes containing a different proportion of RSA and MS were chosen and evaluated for their usage in M40 grade PQC as per the structural design. Also, the cost comparisons of rigid pavement involving these mixes were made.

3.2 MATERIALS

The materials which were used in the experiments and methodology adapted to determine their properties are mentioned below.

3.2.1 Aggregates

Grain size analysis - The grain size analysis of coarse and fine aggregates was done in accordance with IS 2386 (I) [225] and considered in terms of IRC 44 [226]. The aggregates were washed and dried in the oven at 110 ^{0}C temperature until a constant mass was achieved. The sieves were arranged in the order of decreasing size. The gradation curve was plotted between sieve sizes on x-axis (log scale) and percentage finer on the y-axis. The fineness modulus was calculated by dividing the total cumulative percentage of weight retained from 100. The ratio of coarse (20 and 10 mm) and fine aggregates was balanced appropriately to accomplish the combine grading requirement of IS 383 and IRC 44.

Crushing value test - The crushing strength test of coarse aggregates was performed as per IS 2386 (IV) [227]. The aggregates were washed and then dried in the oven at 110 ^{0}C for 4 hours. The aggregates passing through 12.5 mm IS sieve and retained on 10 mm IS sieve were chosen for the test. The crushing load was applied at the rate of 40 kN per min. using a compression testing machine over a period of 10 minutes (total load = 400 kN). The crushed aggregates passing through 2.36 mm IS sieve was expressed as a percentage of the initial weight.

Impact value test – It was performed as per IS 2386 (IV) [227]. The oven dried aggregates passing through 12.5 mm IS sieve and retained on 10 mm IS sieve were filled in the cylindrical mould in three layers, and each layer was given 25 blows by a tamping rod. The aggregates in the mould were subjected to 15 blows of 14 kg metal hammer from the height of 380 mm in the impact test apparatus. The tested aggregates were then sieved through 2.36 mm IS sieve, and the weight of the finer material was expressed as a percentage of the initial weight.

Specific gravity and Water absorption test – These tests for coarse as well as fine aggregates were performed as per IS 2386 (III) [228]. Wire basket method was adopted for coarse aggregates while pycnometer method was used for fine aggregates. The specific gravity and water absorption were calculated using Equation 3.1 (a) and (b) respectively.

$$\text{Specific gravity} = \frac{C}{B-A} \qquad 3.1\ (a)$$

$$\text{Water absorption} = \frac{C-D}{D} \times 100 \qquad 3.1\ (b)$$

where, A = weight of aggregate in water

B = weight of surface saturated dry aggregate

C = weight of oven dried aggregate

Shape test – The shape test of the coarse aggregates was performed as per IS 2386 (I) [225]. 200 pieces of each size of 25 – 20 mm, 20 – 16 mm, 16 – 12.5 mm, 12.5 – 10 mm and 10 – 6.3 mm were weighed. All the pieces of a particular size were gauged through thickness and length gauge and weighed. The ratio of the total weight of the pieces which passed through thickness gauge and which retained on length gauge to the total weight of the pieces considered was reported as the flakiness index and elongation index respectively.

Abrasion value test – Los Angeles abrasion machine was used to determine the abrasion value of the coarse aggregates as per IS 2386 (IV) [227]. 5 kg of 20 mm size aggregate (grading B) and 5 kg of 10 mm size aggregate (grading C) were put inside the drum of the abrasion machine with 11 and 8 steel balls of total weight 4.584 kg and 3.33 kg respectively. The drum was rotated for a total of 500 revolutions with 30 to 33 revolutions per minute. The weight of the material passing 1.7 mm IS sieve was expressed as the percentage of the initial weight.

Density – The bulk density of the aggregates was measured as per IS 2386 (III) [228]. The mass of aggregate required to fill a given volume of the cylindrical mould was noted as 'V'.

3.2.2 Cement, Rice Straw Ash and Microsilica

Density and Specific gravity – The density and specific gravity of cement/RSA/MS were measured as per ASTM C188-17 [229]. The Le Chatelier flask, as shown in Figure 3.2, was filled with kerosene up to 0 to 1 ml mark. The initial mass of the flask with kerosene was noted as 'A'. The flask was then transferred to the water bath with constant temperature, and the height of kerosene (in ml) in the flask was recorded as 'B'. Nearly 64 g of cement/RSA/MS was added to the flask through funnel such that the particles do not stick to the inner body of the flask. The mass of the flask after the addition of cement/RSA/MS was recorded as 'C'. The opening of the flask was closed by glass stopper, and the flask was gently whirled until all the entrapped air was removed. The final height of the kerosene in the flask after the addition of cement/RSA/MS was recorded as 'D'. A constant temperature in the water bath and flask was maintained throughout the experiment. The density and specific gravity were calculated as per Equation 3.2 (a) and (b) respectively. The difference between the height of kerosene in the flask before and after the addition of cement/RSA/MS (B and D respectively) was representative of the equal volume of kerosene displaced (in cm^3).

$$\text{Density} = \frac{\text{Mass of the cement/RSA/MS used}}{\text{Volume of the kerosene displaced}} = \frac{\text{C}-\text{A}}{\text{D}-\text{B}} \qquad 3.2\ (a)$$

$$\text{Specific gravity} = \frac{\text{Density of cement/RSA/MS}}{\text{Density of water at 4 °C}} \qquad 3.2\ (b)$$

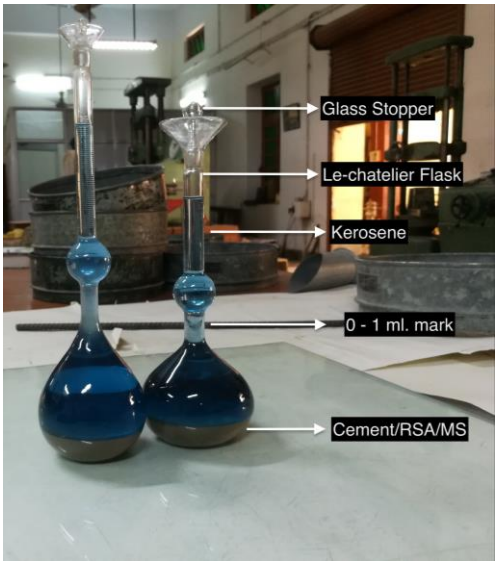


Figure 3.2 Le Chatelier flask for determining density/specific gravity of OPC/RSA/MS

Fineness - The fineness by dry sieving of cement/RSA/MS was measured as per IS 4031 (I) [230]. 10 gm of the material was sieved through 90 µm sieve. The percentage mass of the material retained on the 90 µm sieve w.r.t total mass of material sieved, was considered as a fineness value.

Specific surface area – The specific surface area of the cement was determined by Blaine air permeability method as per IS 4031 (II) [231]. The Blaine air permeability apparatus is shown in Figure 3.3. The specific surface area of the RSA and MS was determined by BET Surface Area Analyzer.

Chemical composition – The chemical composition of cement, RSA and MS was determined by X-Ray Fluorescence (XRF) test. It was performed on cement, RSA and MS particles by using ARL OPTIM X X-Ray Analyzer, as shown in Figure 3.4.

Particle size distribution – The particle size distribution of cement, RSA and MS were determined by CIS-50 ANKERSMID particle size analyser, which is shown in Figure 3.5.

Figure 3.3 Blaine air permeability apparatus for specific surface area of OPC

Figure 3.4 ARL OPTIM X X-Ray Analyzer for XRF analysis of OPC/RSA/MS

Figure 3.5 CIS-50 ANKERSMID particle size analyser for particle size distribution of OPC, RSA and MS

3.2.3 Mineralogical, Microstructural and Thermo Gravimetric analysis of Cement, Rice Straw Ash and Microsilica

The X-ray diffraction (XRD) analysis or mineralogical analysis was performed using RIGAKU Ultima IV X-Ray Diffractometer (Figure 3.6) to determine the crystalline phases of OPC, RSA and MS following ASTM C1365 - 18 [232]. The oven-dried samples of OPC, RSA and MS were subjected to X-Rays of monochromatic copper Kα radiation with wavelength (ƛ) of 1.5418 Å with a scan step size of 0.02^0 and scanning rate of 5^0 per minute. The range of 2θ was 20^0 to 80^0 for OPC and RSA samples and 10^0 to 80^0 for MS sample.

Approximately 10 g of the OPC/RSA/MS powder was fixed to a metal stub using double-sided conductive tape. Further, the stub was mounted in the SEM using a mechanical attachment following ASTM C1723 - 16 [233]. Subsequently, the SEM images were acquired using the FEI QUANTA 450 Scanning Electron Microscope (Figure 3.7).

For thermogravimetric analysis (TGA), 42 g of RSA was placed in a sample pan of NETZSCH Thermo Gravimetric Analyser (Figure 3.8). Further, the temperature was increased gradually up to 1200 ^{0}C at a rate of 20 ^{0}C per minute. Additionally, a graph was also plotted between variations in the mass of RSA concerning temperature and duration of heating to determine the rate of loss of mass.

Figure 3.6 Rigaku Ultima IV X-Ray Diffractometer for XRD analysis

Figure 3.7 FEI QUANTA 450 Scanning Electron Microscope for SEM analysis

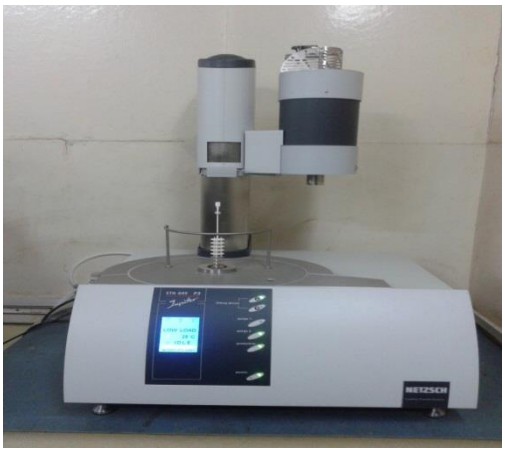


Figure 3.8 NETZSCH Thermo Gravimetric Analyser for TGA analysis of RSA

3.3 STUDY ON PHYSICAL PROPERTIES OF CEMENT PASTE

3.3.1 Normal Consistency

The normal consistency of the cement paste with various proportions of RSA and MS was performed using Vicat apparatus as shown in Figure 3.9 in accordance with IS 4031 (IV) [234]. In the experiment, the total binder content of 400 gm was mixed with water (25% by weight of binder content) to form a cementitious paste. The time taken to form a paste was between 3 to 5 minutes. The Vicat mould kept above the plate was filled with the cementitious paste. The top surface of the paste was brought on a level with the top surface of the Vicat mould. The whole assembly was put under the rod bearing plunger. The plunger was lowered till it touched the top surface of the paste and was then released quickly such that it penetrates the paste. The penetration depth of the plunger from the bottom of the mould was noted. The amount of water was varied in trials of different paste samples until the penetration depth of plunger was between 5 to 7 mm from the bottom of the Vicat mould, and this amount of water (in %) was considered as the consistency (‘*P*’) of the cementitious paste.

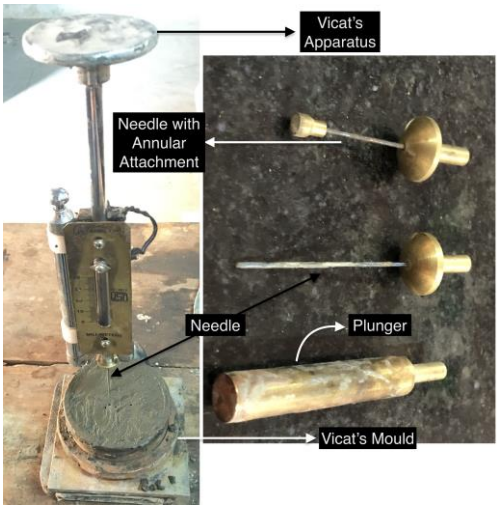


Figure 3.9 Vicat’s apparatus with different attachments for consistency and setting times tests

3.3.2 Initial and Final Setting Time

The initial and final setting time of the cementitious paste was performed using the Vicat apparatus, as shown in Figure 3.9, in accordance with IS 4031 (V) [235]. The methodology adopted was similar to that of the consistency. However, the water content to prepare a cementitious paste with different ratios of cement, RSA and MS was kept fixed at 0.85 times the consistency of that particular mix. Also, in place of a plunger, a needle and a needle with annular attachment were used for determining the initial and final setting time respectively. The time 'T_1' was recorded when water was mixed with cementitious materials. The time at which the needle was able to penetrate the paste up to 5 mm from the bottom of the mould was recorded as 'T_2'. The time at which the needle (with annular attachment) made an impression on the top surface of paste while the annular part did not, was recorded as 'T_3'. The difference between 'T_2' and 'T_1' was reported as the initial setting time, while the difference between 'T_3' and 'T_1' was declared as the final setting time of the cementitious paste.

3.3.3 Soundness Test

The soundness test of the cementitious paste was performed using Le Chatelier apparatus as per IS 4031 (III) [236]. Cementitious paste prepared with different proportions of cement, RSA and MS and w/b ratio was kept fixed at 0.78 times the consistency of that particular mix. The oiled mould was kept on an oiled glass sheet and filled with the prepared cementitious paste. The edges of the mould were kept together while filling of cementitious paste. The mould was covered with another oiled glass sheet, and a small weight was kept over the glass sheet to maintain its position. The whole assembly was immersed in water at 27 ^{0}C for 24 hours. After 24 hours, the distance between the two indicator points was measured and recorded as 'L_1'. The

mould was again submerged in water, and its temperature was increased to 100 ^{0}C in 30 minutes. The temperature of water at 100 ^{0}C was maintained for another 3 hours. The mould was removed from the water and allowed to cool down. The distance between the two indicator points was measured and recorded as 'L_2'. The difference between two distances was indicative of the soundness of the different cementitious paste.

3.3.4 Mineralogical Analysis

XRD analysis of cementitious paste using RIGAKU SmartLab X-Ray Diffractometer (Figure 3.10 (a)) was done to study the formation of hydrated compounds. The rectangular cementitious samples (approximately 50 x 50 mm size) having approximate height of 20 mm were cast with respective normal consistency as w/b ratio (Figure 3.10 (b)). They were kept in the casting room for 24 hours. Further, they were transferred to the closed chamber with RH > 90% and a temperature of 27 ^{0}C for 28 days, followed by drying in an oven at 50 ^{0}C for 24 hours. The samples were then kept in the sealed container at a temperature of 27 ^{0}C for 3 days. Upon cooling, the paste samples were powdered for their XRD analysis as per the procedure mentioned in Section 3.2.3 but with different 2θ range (5^0 to 80^0) and scanning rate (2^0/min.).

(a) **(b)**

Figure 3.10 (a) RIGAKU SmartLab X-Ray Diffractometer for XRD analysis of different specimens **(b)** Cementitious paste cake prepared for their XRD analysis

3.3.5 Marsh Cone Test

The Marsh cone test was performed to determine the optimum dosages of HRWR for cement mortar with different proportions of RSA and MS. It was performed on the cement paste slurry with varying RSA and MS content. The w/b ratio was found out as per IS 4031 (VI) [237]. The w/b ratio and total binder content (2 kg) were kept constant for every mix while the dosage of HRWR in the paste was varied. The predetermined amount of water and HRWR (% by weight of total binder content) were mixed with the binder in two steps. 70% of the total amount of water was mixed with the binder for 2 minutes. Remaining water along with the HRWR was mixed with the paste for another 1 minute. The time taken by 1 litre of paste slurry to pass through Marsh cone, as shown in Figure 3.11 was noted. The above mentioned steps were repeated while gradually increasing the HRWR dosage. The amount of HRWR after which further increase in its dosage did not cause a considerable reduction in the flow time of the paste slurry was recorded. This HRWR dosage was adopted while casting of cement mortar with the same proportion of RSA and MS as was in the respective paste slurry. Similar steps were adopted for various mixtures with different proportion of RSA and MS (% by weight of cement).

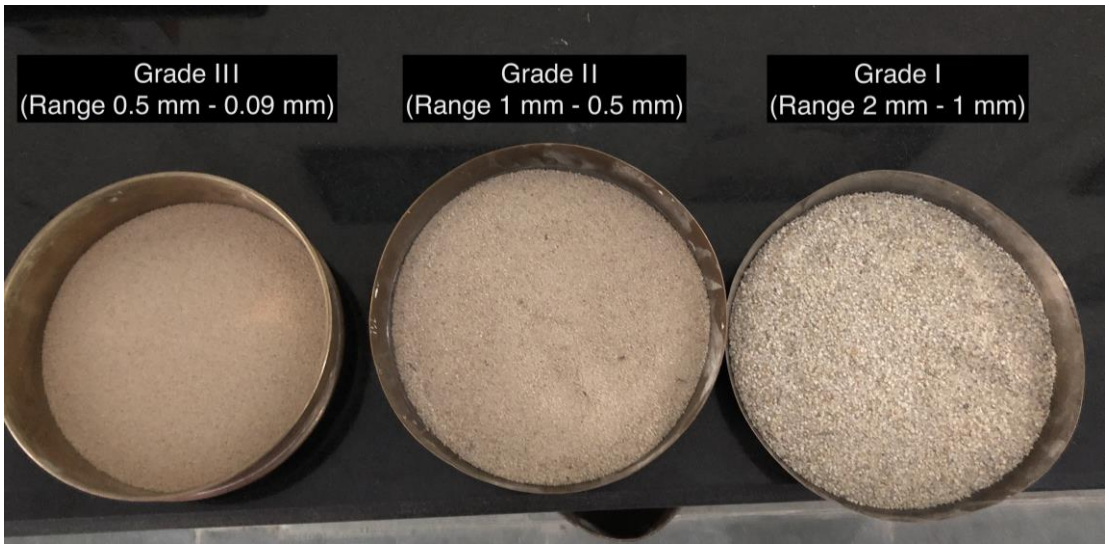
Figure 3.11 Marsh cone test

3.4 STUDY ON PHYSICAL PROPERTIES OF CEMENT MORTAR

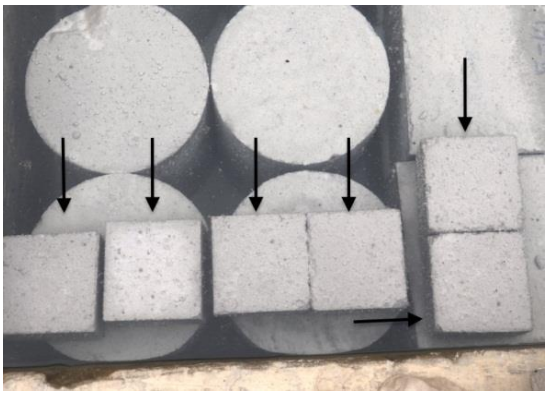
3.4.1 Compressive Strength

The compressive strength of the mortar cubes was determined as per IS 4031 (VI) [237]. The total binder content (200 gm), IS sand (600 gm) and w/b ratio (as per IS 4031 (VI)) were kept constant for all the mix. Three grades of IS sand (Grade I, II and III) as shown in Figure 3.12 (a) were used in equal proportion of 200 gm each in the mixture. The particles of Grade I, II and III IS sand were in the range of 2 mm – 1 mm, 1 mm – 0.5 mm and 0.5 mm – 0.09 mm respectively. The HRWR was not used in a mortar containing 100% cement also known as control mortar while HRWR dosage (% by weight of cement) in the mortar with RSA and MS (% by weight of cement) was decided on the basis of Marsh cone test. The binder content, IS sand, water and HRWR were mixed, and the mixing time was kept between 3 to 4 minutes. After mixing, the mortar was placed in the cube mould of size 7.06 cm. Before placing the mortar in the mould, the inside walls of mould were oiled properly. The mortar in the mould was prodded 20 times in about 8 seconds for removal of entrapped air. The mould was kept on the vibrator machine set at 12,000 ± 400 vibrations per minute. After vibrating the mould for 1 minute, its top surface was levelled with a trowel. Subsequently, the moulds were kept in the moist room (temperature = 27 ^{0}C, RH > 90%) for 24 hours. After 24 hours, the cubes were removed from the mould carefully and were kept in the water curing tank (Figure 3.12 (b)). The temperature of water in the curing tank was maintained at 27 ^{0}C. The water was renewed every week. After completion of 3, 7, 28, 60, 90 and 365 days of curing, the mortar cubes were surface dried and tested for their compression strength in CTM, as shown in Figure 3.12 (c). The load was gradually increased at a rate of 175 KN/min. Three mortar cubes of each mix were cast for

respective day of curing. The average of the three compressive strength values was reported as the compressive strength of mortar.



(a)



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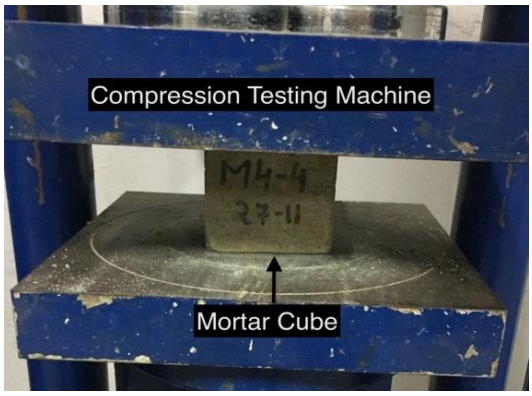


(c)

Figure 3.12 (a) Indian standard sand of different grades **(b)** Curing of mortar cubes **(c)** Compressive strength test of mortar cube

3.4.2 Hardened Density

The hardened density of the mortar was determined as per IS 516 [238]. The excess water on the surface of the mortar was dried by using a cotton cloth after completion of the curing period. The mortar cubes were left in the room with a temperature of 27 ^{0}C for 30 minutes. The saturated surface dry weight of the mortar cubes was recorded for the calculation of hardened density.

3.5 MIX DESIGN OF M40 GRADE PAVEMENT QUALITY CONCRETE

The mix design was done as per IRC 44 [226] and IS 10262 [239]. The final concrete mix design and w/b were confirmed after several trial mixes. In the mix design of concrete, the quantity of materials per unit volume of concrete, the ratio between coarse aggregates and fine aggregates in the concrete, the ratio between 20 mm and 10 mm nominal size coarse aggregates were decided.

3.6 SLUMP TEST

The slump test was done to determine the workability of concrete and also to determine the different dosages of HRWR in the concrete with different proportion of RSA and MS. It was performed in accordance with IS 1199 [240]. The slump mould was placed above the base plate. It was filled with concrete in 4 layers with each layer being tamped 25 times. The internal body of the mould was oiled before filling with concrete. The top surface of the mould was levelled with a trowel, and excess concrete was removed. The mould was gradually raised in the vertical direction. The slump was calculated as the difference between the height of mould and the height of concrete after the mould was raised entirely. If the slump was as decided in the mix design, the dosage of HRWR was fixed for that mix. However, if the slump was lower/higher as compared to mix design, the above-mentioned procedure was repeated with different dosage of HRWR until the slump was equal to the predetermined value. The assembly used in the slump test is shown in Figure 3.13.

Figure 3.13 Slump test of concrete

3.7 STUDY ON MECHANICAL STRENGTH OF CONCRETE

3.7.1 Compressive Strength

The concrete cubes of size 150 mm were cast (Figure 3.14 (a)) as per the mix design of concrete. They were left covered in the casting room for 24 hours. Following 24 hours of casting, the cubes were removed from the mould (Figure 3.14 (b)) and put in the curing tank for curing in water (Figure 3.14 (c)) as per ASTM C192 [241]. The curing water was restored every week, and its temperature was maintained at $27^{o} \pm 2^{o}$ C. After completion of 3, 7, 28, 60, 90 and 365 days of curing, cubes were removed and examined for their compressive strength in CTM as shown in Figure 3.14 (d) as per IS 516 [238]. The load was gradually increased at a rate of 310 KN/min. Three concrete cubes of each mix were cast for respective day of curing. The average of the three compressive strength values was reported as the compressive strength of concrete (f_c).

(a) **(b)**

(c) **(d)**

Figure 3.14 (a) Casting **(b)** Demoulding **(c)** Curing **(d)** Compressive strength test of 150 mm size concrete cubes

3.7.2 Flexural Strength

The concrete prisms of size 500 x 100 x 100 mm were cast as per the mix design of concrete. They were left covered in the casting room for 24 hours. Following 24 hours of casting, the prisms were removed from the mould and placed in the curing tank for curing in water as per ASTM C192 [241]. The curing water was restored every week, and its temperature was maintained at 27 ± 2 ^{o}C. After completion of 3, 7, 28, 60, 90 and 365 days of curing, prisms were removed and examined for their flexural

strength in Flexural Strength Test Machine (Three-point bending) as shown in Figure 3.15 (a) as per IS 516 [238]. The position of the rollers was fixed at markings on the beam, as shown in Figure 3.15 (b). The load was gradually increased at a rate of 1.8 KN per minute. Three concrete prisms of each mix were cast for each day of curing. The average of the three flexural strength values was reported as the flexural strength of concrete at respective day of curing. The flexural strength (f_r in N/mm^2 or MPa) was calculated as per Equation 3.3 (a) and (b).

$$f_r = \frac{P \times L}{b \times d^2} \quad \text{if a > 133 mm} \qquad 3.3\ (a)$$

$$f_r = \frac{3P \times a}{b \times d^2} \quad \text{if 110 mm < a < 133 mm} \qquad 3.3\ (b)$$

where, P = maximum applied load (in N)

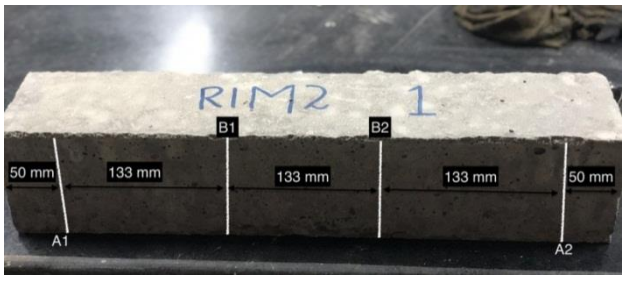
L = span length = 500 mm

b = width of beam = 100 mm

d = depth of beam = 100 mm

a = distance between the line of fracture and the nearest support roller

(a)



(b)

Figure 3.15 (a) Flexural strength test setup **(b)** Position of support and load rollers marked on the beam

3.7.3 Split Tensile Strength

Similar to the casting of concrete cubes and prisms, the concrete cylinders of size 150 mm dia. ('D') and 300 mm height ('H') were cast and cured in water. After completion of 3, 7, 28, 60, 90 and 365 days of curing, cylinders were removed and examined for their split tensile strength in CTM as per IS 5816 [242]. The load was applied on the horizontal axis (height) of the cylinder as marked in Figure 3.16. The loading rate applied was as per Equation 3.4 (a). The split tensile strength ('f_t' in N/mm^2 or MPa) was calculated as per Equation 3.4 (b).

$$\text{Loading Rate} = (1.2 \text{ to } 2.4) \times \frac{\pi}{2} \times \text{H} \times \text{D} \ (\textbf{in N/min.}) \qquad 3.4 \text{ (a)}$$

$$f_t = \frac{2 \times Maximum\ Load\ (\boldsymbol{in\ N})}{\pi \times H \times D} \qquad 3.4 \text{ (b)}$$

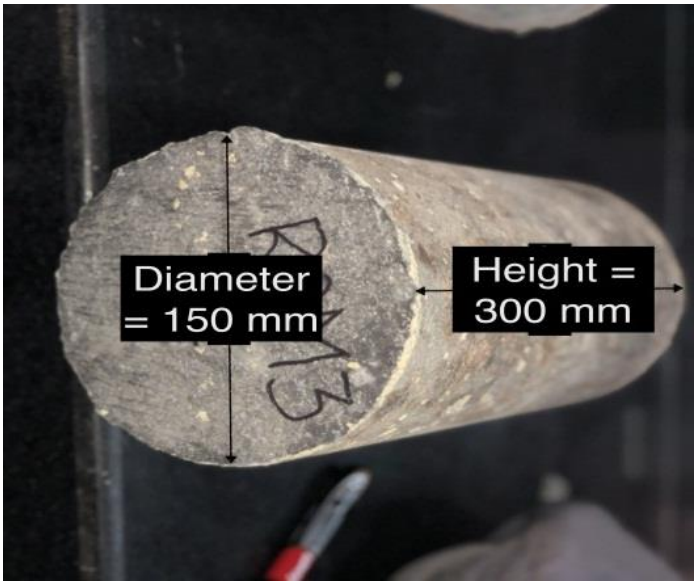


Figure 3.16 Dimensions of concrete cylinder and load position

3.7.4 Microstructural and Mineralogical Analysis

The selected samples of hardened concrete after 28 days of curing in water were pulverized into fine powder form for their XRD analysis to study the formation of

major chemical compounds and to contemplate the way these chemical compounds effect the strength of concrete. XRD analysis of these powdered samples was done as per the procedure mentioned in Section 3.3.4 using RIGAKU SmartLab X-Ray Diffractometer shown in Figure 3.10 (a).

The thin sections of hardened concrete after 7, 28 and 90 days of curing in water were prepared for their SEM analysis. The thin sections of concrete were prepared by mounting a concrete sample on a glass slide using a strong adhesive. After 24 hours, samples were thinned using diamond saws. Further, the polishing of the sample was done by lapping equipment until an ultra-thin section was formed. The SEM analysis was done using FEI Nova NANOSEM 450, as shown in Figure 3.17 to study the effect of microsilica and RSA on the microstructure of hardened concrete. The SEM images of the hardened concrete were acquired at a similar resolution to aid the comparison between different samples of concrete. However, few images with higher resolution and magnification were also obtained for the in-depth microstructural study of few concrete samples.

Similar to the SEM study, the petrographic study also helps in understanding the groundmass of the hardened concrete. However, the images obtained in petrography study had lower resolution and contrast as compared to the SEM images. The thin sections of hardened concrete at 28 days of curing in water were prepared for their petrographic analysis as per ASTM C856 [243] using LEICA optical microscope (Figure 3.18) under ordinary light (O.L.) and at a resolution of 2.5 times (2.5x) zoom and 5 times (5x) zoom.

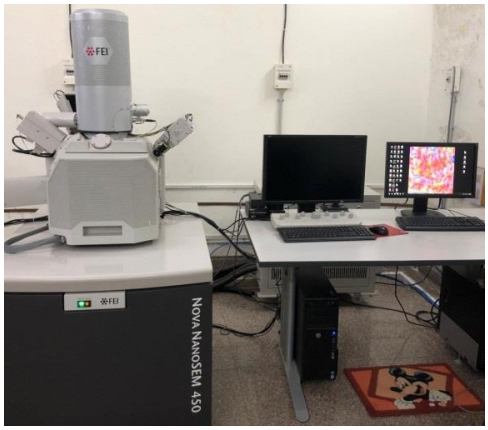


Figure 3.17 FEI Nova NANOSEM 450 Scanning Electron Microscope for microstructural analysis of concrete specimens

Figure 3.18 LEICA optical microscope for petrographic study of concrete specimens

3.8 STUDY ON DURABILITY PROPERTIES OF CONCRETE

3.8.1 Air Entrainment in Fresh Concrete

The air entrainment in fresh concrete was measured using the pressure method (Figure 3.19) as per ASTM C231 [244]. The fresh concrete was filled in the measuring bowl in 3 layers, and each layer was rod 25 times by tamping road. After filling of each layer, the sides of the bowl were tapped for removal of trapped large air bubbles, if any. The excess concrete from the top surface of the measuring bowl was stroked off until the bowl was just full. The measuring bowl was pressure-tight sealed. The water was filled in the tube for level slightly above the zero-mark. The water level was brought back to the zero-mark by tapping the measuring bowl. The operating pressure 'P' was determined by the calibration test. The pressure slightly more than the operating pressure 'P' was applied on the concrete. The sides of the bowl were tapped lightly to bring the pressure to 'P'. The height of water level in the tube was noted as 'h_1'. The pressure on the concrete was lowered through a vent, and subsequently, the sides of the

bowl were tapped for 1 minute. The height of the water level was recorded as 'h_2'. The difference between 'h_1' and 'h_2' was noted as the air content in the fresh concrete.

Figure 3.19 Set-up for air entrainment of fresh concrete

3.8.2 Saturated Water Absorption

Concrete cubes of 150 mm size were cast and cured in water for 3, 7, 28, 60, 90 and 365 days. After completion of the curing period, concrete cubes were subjected to saturated water absorption test as per ASTM C642 [245]. After drying in an oven for 24 to 48 hours at 105 ^{0}C, the hardened cubes were kept in a desiccator at a temperature of 22.5 ± 2.5 ^{0}C to cool down. Subsequently, the dry weight of the cubes was noted as 'A'. Afterwards, the cubes were immersed in water for 48 hours. The temperature of the water was maintained at 21 ± 1 ^{0}C. After 48 hours, the surface-dried weight of the cube was noted as 'B'. The saturated water absorption was calculated as per Equation 3.5.

$$\text{Saturated Water Absorption} = \frac{\text{B}-\text{A}}{\text{A}} \times 100 \qquad 3.5$$

3.8.3 Rate and Coefficient of Water Absorption

For the rate of water absorption and coefficient of water absorption test, concrete cylinders (10 cm dia. & 20 cm height) were cast and cured in water for 3, 7, 28, 60, 90 and 365 days. The test was performed as per ASTM C1585 [202]. After curing, the specimens were dried in an oven at 50 ^{0}C for 3 days. They were then sealed in a chamber at 23 ^{0}C for 15 days. After 15 days, the side and top surface of the concrete cylinders were covered with a polythene sheet. The polythene sheet was tightened in place by a flexible cord and water-proof duct tape. The bottom surface of the cylinders was left uncovered to permit the movement of water in one direction only. The initial mass of the prepared sample was taken. They were submerged in the water to the extent of 3 mm from the bottom surface on support rods, as shown in Figure 3.20. The mass of absorbed water by the prepared specimens was taken at specified intervals (1, 5, 10, 20, 30 and 60 minutes; 2, 3, 4, 5 and 6 hours; 1, 2, 3, 4, 5, 6 and 7 days). The mass of water absorbed ('M') at time 'T', the open surface area of sample ('A'), and density of water ('D') was obtained to calculate the rate of water absorption ('I') as per the Equation 3.6. According to ASTM C1585 [202], the slope of the line best fit to 'I' plotted against the square root of time 1, 5, 10, 20, 30, 60 minutes; 2, 3, 4, 5 and 6 hours was taken as the initial rate of water absorption and against the square root of time 1, 2, 3, 4, 5, 6 and 7 days was taken as the secondary rate of water absorption. The quantity of water ('Q') absorbed by the concrete cylinders in 1 hour was also obtained for calculating the coefficient of water absorption ('K'). 'K' was determined using Equation 3.7.

$$\mathrm{I} = \frac{\mathrm{M}}{\mathrm{A} \times \mathrm{D}} \qquad 3.5$$

$$\mathrm{K} = \frac{\mathrm{Q}}{\mathrm{A} \times \sqrt{\mathrm{T}}} \qquad 3.6$$

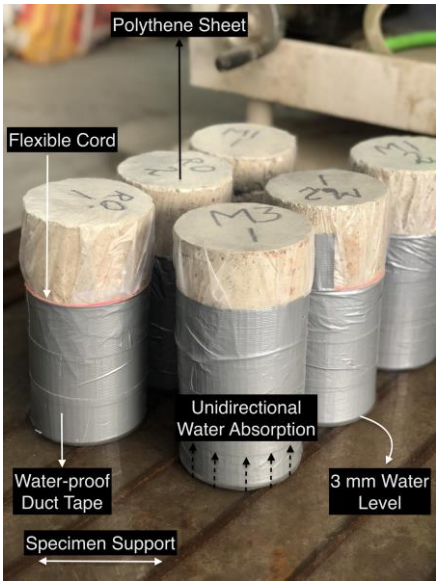


Figure 3.20 Setup for rate and coefficient of water absorption test of concrete

3.8.4 Chloride Ion Penetration

The chloride ion penetration test of concrete slabs (300 mm^2 & 75 mm thick) was conducted as per ASTM C1543 [246]. The slabs were cast as shown in Figure 3.21 (a) and cured in water for 3, 7, 28, 60, 90 and 365 days. After completion of curing, the sides of the slab were covered with closed-cell polystyrene foam, and the bottom surface was left uncovered for proper circulation of air. The polystyrene foam was held together by water-proof duct tape. A rapid-setting epoxy adhered to the vertical surface of polystyrene foam and the horizontal surface of the slab for water-proofing. The ponding solution (3% reagent grade NaCl by weight in water) was maintained up to 2 cm, as shown in Figure 3.21 (b). The top surface was capped with a glass film to avoid excessive dissipation of water through evaporation from the ponding solution. After 90 days of ponding, the NaCl solution was removed, and the surface of the slab was left to dry. The salt crystals formed on the surface of the slab were removed using the wire scrub. The powdered samples were procured from the depths of 10-20 mm and 25-35 mm from the top surface of the concrete slab, as shown in Figure 3.21 (c), and the

chloride ion percentage was obtained as per ASTM C1152 [247]. According to ASTM C1543 [246], concrete cylinders of size 200 mm height and 100 mm diameter were cast from the same mix of concrete which was used to prepare slabs for chloride ion penetration test. These cylinders were then cured in water just like the slabs. After the completion of the curing period, the cylinders were tested for their background chloride content as per ASTM C1152 [247]. The background chloride content of the cylinder was then subtracted from the chloride content obtained at each depth of the ponded slab to determine the penetrated chloride value.

(a)

(b)

(c)

Figure 3.21 (a) Slab casting **(b)** Chloride ion penetration test of the concrete slab by ponding method **(c)** Drilled holes of 10-20 mm and 25-35 mm depth to obtain samples for chloride ion penetration test

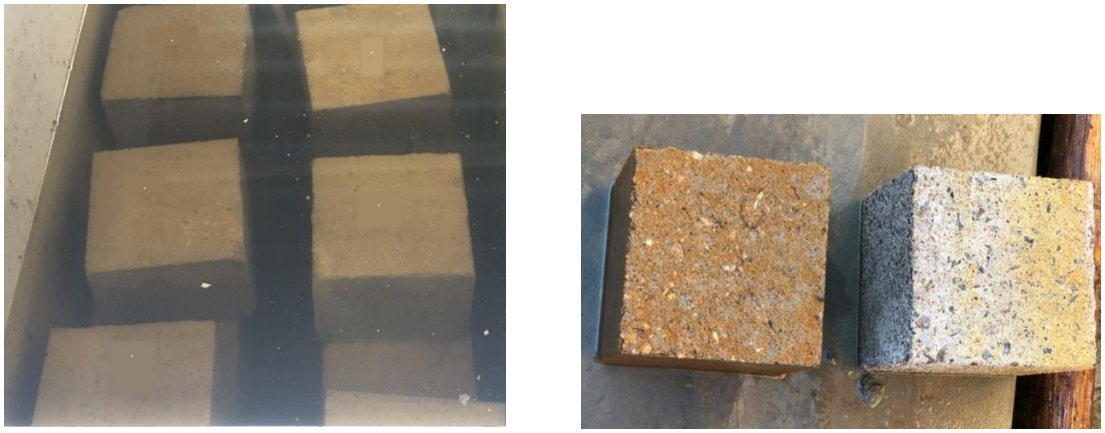
3.8.5 Acidic Environment

For observing the effects of the acidic environment on concrete admixed with RSA and MS, 18 cubes of each mix (150 mm size) were cast and left covered in the

casting room for 24 hours. The cubes were removed from the mould after 24 hours of casting and were placed in the curing tank for water curing as per ASTM C192 [241].

Out of 18 cubes, 12 cubes (6 cubes each) were kept in the water curing tank for 28 days and 365 days. These 12 cubes were removed from the water curing tank after their respective curing time and dried in an oven for 24 hours at 105 ^{0}C. Upon cooling, their initial mass was noted. They were immersed in two acidic solutions having concentration of 2.5% hydrochloric acid and 2.5% sulphuric acid for a period of 30 days (3 cubes for each acidic solution after each age of water curing) (Figure 3.22 (a)). After 30 days of immersion in the acidic solution, the cubes were washed with potable water to remove loose or deposited materials as shown in Figure 3.22 (b) and were placed in an oven at 105 ^{0}C for 24 hours. Upon cooling, their final mass was noted to draw a comparison between the initial and final mass of concrete. These cubes were then subjected to the compression strength test of concrete as per IS 516 [248].

Remaining 6 cubes (3 cubes each) were kept in the water curing tank for 58 days and 395 days until the time of compressive strength test as per IS 516 [248]. To determine the loss of compressive strength due to acidic exposure, the compressive strength of the cubes (28 days water curing + 30 days acidic immersion) was compared with compressive strength of cubes at 58 days of curing in water. Similarly, the compressive strength of the cubes (365 days water curing + 30 days acidic immersion) was compared with compressive strength of cubes at 395 days of curing in water.



(a) **(b)**

Figure 3.22 (a) Concrete cubes under acidic solution **(b)** Concrete Cubes washed with potable water after HCl solution (left) and H_2SO_4 solution (right) immersion

Acidic solutions having concentrations of 2.5% HCl (= 0.291 M) and 2.5% H_2SO_4 (= 0.455 M) in distilled water were recorded to have pH values of 0.54 and 0.34, respectively. The curing water and acidic solution were restored after every 7 days and 10 days respectively, and the temperature of curing water was maintained at 27 ± 2 ^oC. It must be noted that the above-mentioned number of cubes tested are for one concrete mix only. A similar procedure was adopted for testing of remaining mixes. From here on, 'immersion in HCl or H_2SO_4' should be referred to as 'immersion in 2.5% conc. HCl(aq) or H_2SO_4(aq)', respectively.

For SEM analysis, thin sections of the exposed surface of hardened concrete were prepared from the concrete cubes after 365 days of water curing and subsequent immersion in acidic solutions for 30 days. The thin sections of the different concrete mix was prepared as per the procedure mentioned in Section 3.7.4. These thin sections were studied under the FEI Nova NANOSEM 450 Scanning Electron Microscope (Figure 3.17) at various resolutions to observe the effects of acidic solution on the cementitious systems of hardened concrete and interpret the factors responsible for the deterioration of concrete.

For XRD analysis, RIGAKU SmartLab X-Ray Diffractometer was used (Figure 3.10 (a)). Hardened cement mortar was separated from coarse aggregates of hardened concrete for understanding the improvement in the resistance of cementitious systems to an acid environment (separation of fine aggregates was not possible due to its finer size). The hardened cement mortar was ground to a fine powdered form. The powdered samples were placed on the flat and thin glass slide using adhesive material. Similar procedure as mentioned in Section 3.3.4 was adopted for XRD analysis.

3.8.6 Carbonation

For observing the effects of Accelerated Carbonation Curing, 3 cubes (150 mm size) for compressive strength, 3 prisms (500 x 100 mm x 100 mm) for flexural strength and 3 cylinders (300 mm height x 150 mm dia.) for split tensile strength were cast for each mix of concrete. After casting, all the specimens were left covered in the casting room for 24 hours. The specimens were removed from the mould after 24 hours of casting and were placed in the water curing tank as per ASTM C192 [241].

These samples of concrete (cube/prism/cylinder) were kept in the water curing tank for 14 days. Then, the samples were removed from the curing tank and kept in the oven for 24 hours at 60 ^{0}C. Subsequently, they were kept in the carbonation chamber for another 14 days for accelerated carbonation curing (Figure 3.23). The concentration of CO_2, temperature and relative humidity in the carbonation chamber were maintained at 5%, 27 ± 2 ^{o}C and 65 ± 5% respectively as per other studies [153], [179], [192], [249]. After the completion of ACC, these samples of concrete were subjected to compression strength test and flexural strength test as per IS 516 [248] and split tensile strength test as per IS 5816 [242]. The strength (compressive/flexural/split tensile) of

the concrete samples at 28 days of combined curing (water + ACC) were compared with strength at 28 days of water curing to study the effect of ACC.

The cubes subjected to ACC were cut vertically into two parts, and phenolphthalein indicator was sprayed on the cut faces. The phenolphthalein indicator was prepared by mixing 1g of phenolphthalein with 90 ml of ethanol. It was diluted to 100 ml with water. The carbonation affected zone was colourless while the unaffected zone (where CO_2 was not able to penetrate) turns pink in colour. The depth of CO_2 penetration in each cube was measured.

The curing water was restored after every 7 days, and the temperature of curing water was maintained at 27 ± 2 ^oC. It must be noted that the above-mentioned number of specimens tested are for one mix only. A similar procedure was adopted for the testing of remaining concrete mixes.

Figure 3.23 Concrete prisms in a carbonation chamber for ACC

3.9 STRUCTURAL DESIGN AND COST COMPARISON

Based on the results of mechanical and durability properties, few concrete mixes containing different proportions of RSA and MS were chosen. The structural design of the selected combinations of concrete for rigid pavement was done as per IRC 58 [250]. They were evaluated for their usage in M40 grade PQC as per their flexural strength. The design thickness of the concrete slab of the selected mix was computed. Also, the cost comparison of rigid pavement involving the concrete of selected mix was done. The cost comparison was done on the basis of the total cost of 1 m^3 of admixed concrete and also on the basis of the total cost of admixed concrete required for construction of 1 km of National Highways (2 lanes, one way).

3.10 CHAPTER SUMMARY

Chapter 3 provides a brief discussion about the methodology adopted to determine various properties of materials used in the current investigation. Subsequently, the methods utilized to identify the physical properties of cement paste and cement mortar containing a different proportion of RSA and MS were discussed in detail. Also, the process adopted to study the results related to mechanical and durability properties of concrete of these mixes were discussed. The procedure chosen for mineralogical and microstructural analysis of selected admixed concrete mixes was explained. Chapter 3 also shows the instruments used for various tests and the standard codes which were followed during the experiments. Finally, the procedure adopted for comparison between the structural design and cost of the rigid pavement incorporating different proportions of RSA and MS was explained.