CHAPTER 7

MINERALOGY AND MICROSTRUCTURE OF CONCRETE

7.1 X-Ray diffractometer (XRD) Test

The X-ray diffraction (XRD) technique offers a convenient way to determine the mineralogical analysis of crystalline solids. If a crystalline mineral is exposed to X-rays of a particular wavelength, the layers of atoms diffract the rays and produce a pattern of peaks which is characteristic of the mineral. The horizontal scale (diffraction angle) of a typical XRD pattern gives the crystal lattice spacing and the vertical scale (peak height) gives the intensity of the diffracted ray, when the specimen being X-rayed contains more than one mineral, the intensity of characteristic peaks from the individual minerals are proportional to their amount.

The XRD test is used by Seligmann, P. and Greening, N. R. (90) and Khudsen, T. (91) to identify the crystalline phases and also to study the crystal structures of some of the phases and of differential thermal analysis of quantitative analysis is also possible.

The XRD test is widely used for determining the mineralogical and chemical composition of the cementitious materials. This test is based on the principle of Bragg’s law reported by Narendra Babu, G. C., (92).

A crystal lattice is a regular three-dimensional distribution (Cubic, Rhombic etc.) of atoms is space. These are arranged so that they form a series of parallel planes separated from one another by a distance ‘d’ which varies according to the nature of the material. For any crystal, planes exist in a number of different orientations-each with its own specified ‘d’ spacing.

**Diffraction- Bragg’s law**

When a monochromatic x-ray beam with wavelength ‘λ’ is projected on to a crystalline material at an angle ‘θ’, diffraction occurs only when the distance traveled by the rays reflected from successive planes differs by a complete number ‘n’ of wavelengths.

By varying the angle ‘θ’ the Bragg’s law conditions are satisfied by different ‘d’ spacing in polycrystalline materials. Plotting the angular positions and intensities of the resultant diffracted peaks of radiation produces a pattern, which is characteristic of the
sample. Where a mixture of different phases is present, the resultant diffractogram is formed by addition of the individual patterns.

Philips XRD model X’pert Pro was used in the present research work. The representative concrete samples were powdered down to ≈300 mesh (< 53 micron size) and subjected to CuKa radiation generated at 40 KV and 30 mA. The powdered samples are pressed into the aluminum holder and subjected to radiations between 0-60° (2θ). A scanning speed of 5° is maintained throughout.

7.2 Scanning Electron Microscope (SEM) Test

The SEM test is widely used for studying the microstructure of concrete; to know the bonding between the cement paste and aggregate interfacial zone.

The type, amount, size, shape and distribution of phases present in a solid constitute its structure. The gross elements of the structure of a material can readily be seen, whereas the finer elements are usually resolved with the help of microscope. The term macrostructure is generally used for the gross structure, visible to human eye. The limit of resolution of the unaided human eye is approximately one-fifth of a millimeter (200μm). The term microstructure is used for the microscopically magnified portion of a macrostructure. The magnification capability of modern electron optical microscopes is of the order of 10^5 times; thus the application of transmission and scanning electron optical microscopy techniques has made it possible to resolve the structure of materials to a fraction of a micrometer.

Progress in the level of material has resulted primarily from recognition of the principle that the properties of a material originate from its internal structure; in other words, the properties can be modified by making suitable changes in the structure of a material. Although concrete is the most widely used structural material, its structure is heterogeneous and highly complex. The structure-property relationships in concrete are not yet developed; however, an understanding of some of the elements of the concrete structure is essential to understand the factors influencing the important engineering properties of concrete such as strength, durability, cracking, shrinkage, elasticity creep etc.
7.3 XRD Test Results

The XRD results are presented in Figures 21 to 24 for M25 concrete at 90 days. For M30 concrete (150, 180 and 210 days) are shown in Figures 33 to 44.

7.4 SEM Test Results

The SEM photographs are shown in Figures 25 to 32 for M25 concrete at 90 days. For M30 concrete (150, 180 and 210 days) are presented in Figures 45 to 80.

Fig. 21: X-Ray Diffractogram of Blended Cement (PPC) Concrete of Grade M25 (90 days).
Fig. 22: X-Ray Diffractogram of Blended Cement (PSC) Concrete of Grade M25 (90 days)

Fig. 23: X-Ray Diffractogram of 43 Grade OPC Concrete of Grade M25 (90 days)
Fig. 24: X-Ray Diffractogram of 53 Grade OPC Concrete of Grade M25 (90 days)

Fig. 25: SEM Photograph of 53 Grade OPC Concrete of Grade M25 (90 days)
Fig. 26: SEM Photograph of 53 Grade OPC Concrete of Grade M25 (90 Days)

Fig. 27: SEM Photograph of 53 Grade OPC Concrete of Grade M25 (90 days)
Ph. 4 BM-25
Fig. 28: SEM Photograph of Blended Cement (PSC) Concrete of Grade M25 (90 days)

Ph. 5 BM-25
Fig. 29: SEM Photograph of Blended Cement (PSC) Concrete of Grade M25 (90 days)
Fig. 30: SEM Photograph of Blended Cement (PPC) Concrete of Grade M25 (90 days)

Fig. 31: SEM Photograph of Blended Cement (PPC) Concrete of Grade M25 (90 days)
Fig. 32: SEM Photograph of 43 Grade OPC Concrete of Grade M25 (90 days)

Fig. 33: X-Ray Diffractogram of Blended Cement (PPC) Concrete of Grade M30 (150 days)
Fig. 34: X-Ray Diffractogram of Blended Cement (PPC) Concrete of Grade M30 (180 days)

Fig. 35: X-Ray Diffractogram of Blended Cement (PPC) Concrete of Grade M30 (210 days)
Fig. 36: X-Ray Diffractogram of Blended Cement (PSC) Concrete of Grade M30 (150 days)

Fig. 37: X-Ray Diffractogram of Blended Cement (PSC) Concrete of Grade M30 (180 days)

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Fig. 38: X-Ray Diffractogram of Blended Cement (PSC) Concrete of Grade M30 (210 days)

Fig. 39: X-Ray Diffractogram of 43 Grade OPC Concrete of Grade M30 (150 days)
Fig. 40: X-Ray Diffractogram of 43 Grade OPC Concrete of Grade M30 (180 days)

Fig. 41: X-Ray Diffractogram of 43 Grade OPC Concrete of Grade M30 (210 days)
Fig. 42: X-Ray Diffractogram of 53 Grade OPC Concrete of Grade M30 (150 days)

Fig. 43: X-Ray Diffractogram of 53 Grade OPC Concrete of Grade M30 (180 days)
Fig. 44: X-Ray Diffractogram of 53 Grade OPC Concrete of Grade M30 (210 days)

Fig. 45: SEM Photograph of AM30- Partly reacted fly ash grains in hydrated Cement matrix (150 days)
Fig. 46: SEM Photograph of AM30-Cement paste matrix shows platy calcium Hydroxide crystals with fly ash grain acting as a micro aggregate and partly reacted fly ash forming other hydration products (150 days)

Fig. 47: SEM Photograph of AM30-Cement paste matrix shows platy calcium hydroxide with other hydration products at the contact with aggregate (150 days)
Fig. 48: SEM of Photograph BM30-Partly reacted slag grains in hydrated cement matrix at the contact of aggregate and binder phase (150 days)

Fig. 49: SEM Photograph of BM30-Cement paste matrix shows hydration products with unreacted elongated slag grain (150 days)
Fig. 50: SEM of BM30-Cement paste matrix shows hydration products at the contact with aggregate (150 days)

Fig. 51: SEM Photograph of CM30-Ettringite fibers in a cavity in hydrated cement matrix at the contact of aggregate and binder phase. Platy calcium hydroxide with other hydration products are also seen (150 days)
Fig. 52: SEM Photograph of CM30-Cement paste matrix shows hydration products mainly C-H and C-S-H phases with less dense matrix which is somewhat porous (150 days)

Fig. 53: SEM Photograph of CM30- Cement paste matrix shows hydration products at the contact with aggregate (150 days)
Fig. 54: SEM Photograph of DM30- Ettringite fibers/crystals in a cavity in hardened cement matrix at the contact of aggregate and binder phase. Platy calcium hydroxide with other hydration products is also seen (150 days).

Fig. 55: SEM Photograph of DM30-Cement paste matrix shows hydration products mainly C-H and C-S-H phases with less dense matrix which is somewhat porous (150 days).
Fig. 56: SEM Photograph of DM30-Highly porous cement paste matrix shows hydration products C-S-H which are small acicular and cauliflower shaped (150 days)
Fig. 58: SEM Photograph of ASM30-Cement paste matrix shows platy calcium hydroxide crystals with partly reacted fly ash forming other hydration products (180 days)

Fig. 59: SEM Photograph of AM30- Cement paste matrix shows platy calcium hydroxide with other hydration products at the contact with aggregate (180 days)
Fig. 60: SEM Photograph of BM30- Cement paste matrix shows hydration products at the contact with aggregate (180 days)

Fig. 61: SEM Photograph of BM30- Cement paste matrix shows hydration products with a mesh like appearance (180 days)
Fig. 62: SEM Photograph of BM30- Partly reacted slag grains in hydrated cement matrix and binder phase (180 days)

Fig. 63: SEM Photograph of CM30- Cement paste matrix shows hydration products mainly C-H and C-S-H phases with less dense matrix which is somewhat porous (180 days)
Fig. 64: SEM Photograph of CM 30- Hydrated cement matrix at the contact of aggregate and binder phase. Platy calcium hydroxides with other hydration products are also seen (180 days)

Fig. 65: SEM Photograph of CM30- Cement paste matrix shows hydration products with cauliflower shaped C-S-H phases (180 days)
Fig. 66: SEM Photograph of DM30- Hydrated cement matrix at the contact of aggregate and binder phase. Platy calcium hydroxide with other hydration products is also seen (180 days)

Fig. 67: SEM Photograph of DM30-Cement paste matrix shows hydration products mainly C-H and C-S-H phases with less dense matrix which is somewhat porous (180 days)
Fig. 68: SEM Photograph of DM30- Highly porous cement paste matrix shows hydration products C-S-H phases which are cauliflower shaped (180 days)

Fig. 69: SEM Photograph of AM30- Partly reacted fly ash grains in hydrated cement matrix (210 Days)
Fig. 70: SEM Photograph of AM30-Cement paste matrix shows platy calcium hydroxide crystals with partly reacted fly ash forming other hydration products (210 days)

Fig. 71: SEM Photograph of AM30-Cement paste matrix shows platy calcium hydroxide with other hydration products at the contact with aggregate (210 days)
Fig. 72: SEM Photograph of BM30- Cement paste matrix shows hydration products at the contact with aggregate (210 days)

Fig. 73: SEM Photograph of BM30-Cement paste matrix shows hydration products with a mesh like appearance (210 days)
Fig. 74: SEM Photograph of BM30- Partly reacted slag grains in hydrated cement matrix and binder phase (210 Days)

Fig. 75: SEM Photograph of CM30- Cement paste matrix shows hydration products mainly C-H and C-S-H phases with less dense matrix which is somewhat porous (210 days)
Fig. 76: SEM Photograph of CM30- Hydrated cement matrix at the contact of aggregate and binder phase. Platy calcium hydroxide with other hydration products are also seen (210 days)

Fig. 77: SEM Photograph of CM30-Cement paste matrix shows hydration products with cauliflower shaped C-S-H phases (210 days)
Fig. 78: SEM Photograph of DM30- Hydrated cement matrix at the contact of aggregate and binder phase. Platy calcium hydroxide with other hydration products is also seen (210 days)

Fig. 79: SEM Photograph of DM30- Cement paste matrix shows hydration products mainly C-H and C-S-H phases with less dense matrix which is somewhat porous (210 days)
Fig. 80: SEM Photograph of DM30- Highly porous cement paste matrix shows hydration products C-S-H phases which are cauliflower shaped (210 days)