CHAPTER 7

MICRO STRUCTURAL PROPERTIES OF CONCRETE
WITH MANUFACTURED SAND

7.1 GENERAL

Characterizing the mineralogy of the samples can be done in several ways. The SEM identifies the morphology of the structure. Energy Dispersive Spectroscopy (EDS) allows high resolution identification of elements and compounds present in prepared 2-D cross-sections of aggregate samples. X - Ray Diffraction (XRD) is also used to identify the compounds and minerals present in powdered specimens.

7.2 MICRO STRUCTURAL ANALYSIS

7.2.1 Scanning Electron Microscopy (SEM) Analysis

SEM coupled with EDS is an effective tool for visually examining the particles that are too small to be seen under an optical microscope. The SEM works by aiming an electron beam at the surface of the specimen. When the electron beam strikes a solid object, the electrons are either scattered or absorbed. The collection of these responses is what forms the SEM image. Sarkar et al (2001) stated that any electrically conductive object can be microscopically examined in this manner.

The powdered samples of concrete specimens with natural sand and manufactured sand are suspended in a two-part Epotek 301 epoxy to stabilize
them for SEM use. First, the epoxy is mixed according to the manufacturer’s
directions. Then powdered samples are stirred into the mixture by adding
small amounts at a time in order to ensure that particles are evenly coated
with epoxy. The mixture is then poured into a one inch diameter sample cup,
put under vacuum to remove air bubbles. Then the sample is cured at room
temperature for a minimum of 24 hours under normal pressure. Next, the
samples are removed from the sample holders and polished first with 120 grit
sandpaper and progressed with levels of higher grit sandpaper until reaching
1200 grit size. Further polishing is continued with 6µm, 3µm, 1µm and
0.25µm diamond paste. Later on they are coated with gold palladium in order
to provide a conductive surface for the SEM. The images of concrete sample
with natural sand and manufactured sand are taken at various magnifications
to identify the shape and texture of the particles.

7.2.2 Energy Dispersive Spectroscopy (EDS) Analysis

Energy dispersive spectroscopy is performed to determine the
composition of elements present in the sample. Any smaller size particles can
be analyzed at large magnification using EDS. Depending upon the samples,
several points are selected on the SEM image and analyzed through EDS.
EDS detects the elements present in a specimen based on the detection of
X-Rays emitted by that specimen. The X-Ray photons emitted by the
specimen are collected by EDS and converted into a number of “counts” at
each emission voltage. Sarkar et al (2001) affirmed that “The total number of
counts for a particular element is proportional to the amount of that element
present in the object”.

7.2.3 X-Ray Diffraction (XRD) Analysis

In the next step X-Ray Diffraction (XRD) analysis is performed to
determine the silica phase of the powder concrete samples. The samples are
scanned by an X-Ray diffractometer which is shown in Figure 7.1.
A small amount of powder sample is put into an aluminum sample holder and the surface is finished smoothly. The holder is then placed into the X-Ray diffractometer. The samples are scanned by an X-Ray diffractometer using CuKα radiation at 40 kV / 20 mA, CPS = 1k, width 2.5, speed 2° / min and scanned with an angle of 2θ from 3 – 70°. The analysis is stepped at 0.04 degree increments and continued for a period of 3 seconds. In X-Ray diffraction, X-Rays are scattered by atoms in a pattern that indicates lattice spacing of elements present in the material analyzed. Once the X-Ray analysis is completed, the scans are analyzed using Jade 7 – X-Ray Diffraction (XRD) software. Using Jade, peak intensities at different angles are compared with a database of different minerals and compounds. Compounds with peak intensities matching those of the scans are identified and the compounds present in the samples are also determined.

7.3 DISCUSSION OF TEST RESULTS

7.3.1 Scanning Electron Microscopy (SEM) Analysis

Figure 7.2 shows the SEM image of concrete sample with natural sand and manufactured sand at magnifications of 20000x.
(a) Concrete with NS  
(b) Concrete with MS

**Figure 7.2 SEM images of concrete sample with NS and MS**

From the Figure 7.2(a) the major axis length was measured as 1.55 micrometer and the minor axis length was calculated as 1.5 micrometer. Here, the major axis length was almost equal to the minor axis length and the elongation was calculated as the ratio of the major axis length to the minor axis length, which was 1.0. This indicates that the particles are circular in shape. The area of the particle was measured as 1.88 square microns and the perimeter was calculated as 4.87 microns. The roundness of the particle was calculated as 0.99, which represents that the particles are almost spherical in shape. This improves the workability of the concrete.

Figures 7.2 (b) exhibits the SEM image of the sample with manufactured sand at the same magnification of 20000x. From the Figure, the major axis length was measured as 1.87 micrometer and the minor axis length was 0.55 micrometer. The elongation was calculated as 3.4. This indicates that the particles are elongated one. The area of the particle was measured as 1.03 square microns and the perimeter was calculated as 4.84 microns. The roundness of the particles was calculated as 0.55, which represents that the particles are angular in shape. They create better packing between the
particles and reduce the porosity. Because of this, the strength and durability characteristics are improved.

7.3.2 Energy Dispersive Spectroscopy (EDS) Analysis

Figure 7.3 conveys the EDS test results of concrete sample with natural sand and manufactured sand.

(a) Concrete with NS  (b) Concrete with MS

Figure 7.3 EDS analysis of concrete sample with NS and MS

From the Figure 7.3(a), it is found that the minerals present in the concrete sample are silica, calcium and oxides. Here the calcium reacts with silica and oxides, and produces the hydrated calcium silicates, which impart strength to the concrete at early and later periods.

Figure 7.3 (b) provides the test results of concrete sample with manufactured sand. From the Figure, it becomes evident that the concrete sample contains silica, calcium, alumina and oxides. The calcium reacts with alumina and oxides and produces tri calcium aluminate, which is the reason for early setting.
7.3.3 X-Ray Diffraction (XRD) Analysis

The X-Ray diffraction pattern of sample with natural sand and manufactured sand are shown in Figures 7.4 (a) and (b).

![XRD pattern of concrete with NS and MS]

(a) Concrete with NS  (b) Concrete with MS

**Figure 7.4 XRD pattern of concrete with NS and MS**

From the Figures 7.4 (a) and (b), it is seen that the major component present in the sample is silica content due to the peak on 2θ angle of 27°. The peak indicates that the silica content is of crystalline nature and the broad parts indicate that the elements are amorphous in nature. The broadening portion of the curve represents the particles in diffraction pattern.

7.4 CONCLUDING REMARKS

The micro structural properties of concrete with natural sand and manufactured sand are investigated by SEM coupled with EDS and XRD analysis. SEM images confirmed that the natural sand particles are spherical in shape while the manufactured sand particles are elongated and angular in shape. EDS analysis confirmed that the manufactured sand contains the minerals of silica, calcium, alumina and oxides and from XRD results, it is confirmed that the major component is silica content and it is in crystalline form.