

X-Ray Diffraction - (XRD)

"It is a technique used for investigation of structure of a material at its atomic & molecular level."

(*) It is usually applied for crystalline or partially crystalline material (that have periodic structural order).

(*) Not applied this technique for noncrystalline materials.

Basic elements of XRD:-

i. X-ray tube (ii) X-ray holder (iii) X-ray detector.

Analysis of XRD on Concrete:-

XRD analysis on ~~concrete~~ concrete is performed to analyze silica phase of powder concrete samples.

• The samples are scanned in X-ray diffractometer which is shown.

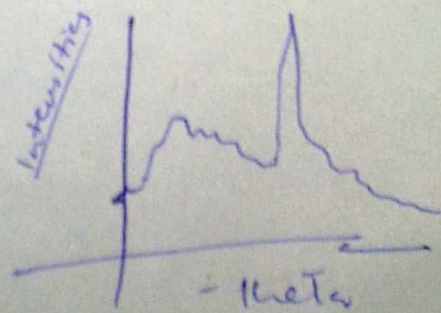
• Method to analyze structural detail of concrete using XRD:-

The sample of concrete is placed into aluminum sample holder. This holder is then placed in XRD diffractometer.

- The x-ray diffractometer used gamma rays for scanning of concrete.
- In this process, the x-ray is diffracted which indicates lattice spacing of element present in that sample.
- Once the scan is completed it ^{is} then analyze is Jade x-ray diffraction software.
- We get result in form peaks like heart beat graph.
- From these intensities at different angles are compared with intensities (Peak) of other elements and compounds.
- If they match we conclude that such material is present in sample of concrete.
- Such test can be performed on natural sand and manufacture sand. The peak intensities can be shown as;



For natural sand



(b) On Manufactured concrete.

→ The major peak of sample is due to presence of silica content i.e. from 2θ angle of 27° .

→ The peaks indicate that silica is of crystalline in nature and broad part indicate it is of amorphous nature.

→ Broadening portion of curve represent particles in diffraction pattern.

Scanning Electron Microscope SEM:-

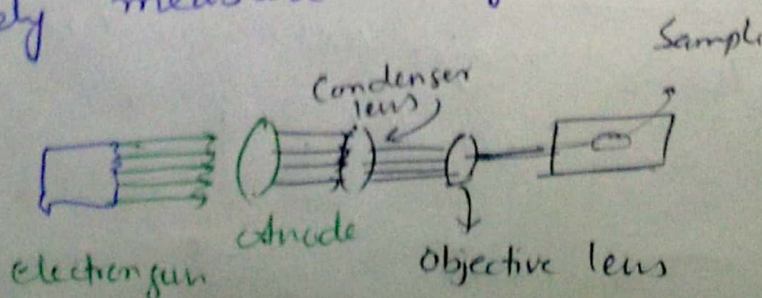
It is a technique that used electron beam to produce high magnified image of small objects.

→ This technique provide both topographic and compositional analysis of material.

→ This technique also known as SEM analysis or SEM microscopy.

(*) Importance:-

This technique used high magnification that produce high resolution image and precisely measure very small features {objects}



Analysis of SEM on Concrete

- The powder sample of concrete mixed with epoxy to stabilize them for SEM use.
- It is stirring and at same time epoxy are adding so that particles are evenly coated with epoxy.
 - It is then poured in 1 diameter sample cup and kept it in vacuum to remove bubbles.
 - It is then cured at room temperature for minimum of 24 hours.
 - (k) The sample is then remove from holder & polish with 120 grit sand paper. It is then polished with 6m, 3m, 1m & 0.25m diamond past.
 - Later provide coated over it of gold palladium to provide conductive surface for SEM.
 - The sample is then scan under electron microscope to produce its detail image.
 - It can produce image upto 20000X.

Explanation of fig (a):-

- The length of major axis is 1.55 micron & minor axis length is 1.5.

Here length of major axis = Length of minor axis.

$$\therefore \text{Elongation } E = \frac{1.55}{1.5} = 1.$$

The area of particle is;

$$= (1.55)(1.5)$$

$$= 1.88 \text{ square micron.}$$

$$\frac{E}{2} \text{ perimetry} = 4.87 \text{ micron (sum of two times of major axis + two times of minor axis).}$$

(*) The roundness of particle is calculated to be 0.99 which shows particles are spherical.

Explanation of fig(b):-

This image is manufacture sand under SEM.

Here length of major axis is 1.87 micron and length of minor axis 0.55 microns.

$$\therefore \text{elongation } E = \frac{1.87 \text{ micron}}{0.55 \text{ micron}} = 3.4.$$

→ The area of particles is measured as 1.63 square micron & perimetry = 4.84 microns.

→ The roundness of particle was calculated as 0.55 which show particles are angular in shape.

Energy dispersive X-ray analysis:-

This technique is used to analysis the composition of element from which the specimen is made.

→ Also known as EDS or EDAX analysis.

(i) This is used to determine the composition

of element after marking SEM scanning.

• Any small area can be determine at large magnification using EDS.

• When SEM scanned image is observe under EDS, the X-ray photon emitted by that element are collected by EDS and we get the detailed image of element.

Sarkar et al (2021) affirmed that;

"Amount of counts for particular element is proportional to amount of element present in specimen."

When natural sand concrete is test under EDAX:-

• The image shows that it contains silica, calcium & oxides.

→ Silica & calcium oxide react with each

Analysis of XRF on Cement

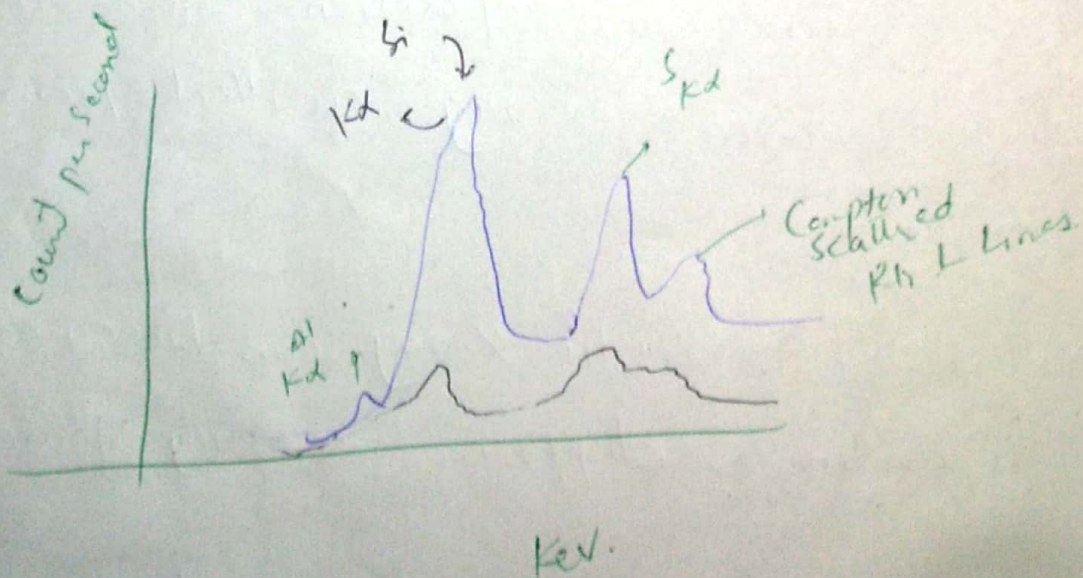
- Pressed powder pellet is used to perform XRF analysis of cement.

→ It is mixed with flux i.e. Lithium Tetraborate and melt it then mixed with platinum crucible at very high temp.

(*) It is then form a disc in mold.

(*) Elemental analysis of cement using XRF give good and reliable result.

- With fusion fluxes, reliable & repeatable preparation of cement sample for XRF analysis is made easy.



- and form hydrated calcium silicate which imparts strength at early & late period.

Analysis of Concrete with Manufacture sand

Using EDAX:-

→ Figure shows that it contains sample of silica, oxide, calcium, alumina.

→ Calcium reacts with alumina {oxide to produce calcium alumina which helps in setting of concrete.

X-Ray fluorescence:- (XRF)

(*) This technique is used to determine the element composition of sample using fluorescent x-ray emitted from that sample.

→ The characteristic x-ray can be analyzed:-

① Wavelength dispersive X-ray fluorescence.

② Energy dispersive X-ray fluorescence.