

## **A Qualitative Study of Reactive Powder Concrete using X-Ray Diffraction Technique.**

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**Abstract**—*The development of X –ray diffraction it became possible to integrate the method into existing laboratory automation systems for process and quality control to provide a means of online monitoring. A reliable, precise and very reproducible method to quantify the relative phase abundances of cementitious product is to use X-Ray diffraction method. This analysis is a powerful method for the determination of quantitative phase amounts in multiple phase mixtures. In a present study an optimized composition of RPC in different curing regime has been studied qualitatively in XRD at regular interval of time, Better understanding hydration and microstructure changes and mineralogical changes has been studied.*

**Key words**—*RPC, XRD, ultra high strength concrete, microstructure.*

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### **I. INTRODUCTION**

X-ray powder diffraction (XRD), is an instrumental technique that is used to identify minerals, as well as other crystalline materials. In many geologic investigations, XRD complements other mineralogical methods, including optical light microscopy, electron microprobe microscopy, and scanning electron microscopy. XRD provides the researcher with a fast and reliable tool for routine mineral identification. XRD is particularly useful for identifying fine-grained minerals and mixtures or intergrowths of minerals that may not lend themselves to analysis by other techniques. XRD can provide additional information beyond basic identification. If the sample is a mixture, XRD data can be analyzed to determine the proportion of the different minerals present. Other information obtained can include the degree of crystalline of the mineral(s) present, possible deviations of the minerals from their ideal compositions (presence of element substitutions and solid solutions), the structural state of the minerals (which can be used to deduce temperatures and (or) pressures of formation), and the degree of hydration for minerals that contain water in their structure. Some mineralogical samples analyzed by XRD are too fine grained to be identified by optical light microscopy. XRD does not, however, provide the quantitative compositional data obtained by the electron microprobe or textural and qualitative compositional data obtained by the scanning electron microscope. The three-dimensional structure of no amorphous materials, such as minerals, is defined by regular, repeating planes of atoms that form a crystal lattice. When a focused X-ray beam interacts with these planes of atoms, part of the beam is transmitted, part is absorbed by the sample, part is refracted and scattered, and part is diffracted. Diffraction of an X-ray beam by a crystalline solid is analogous to diffraction of light by droplets of water, producing the familiar rainbow. X-rays are diffracted by each mineral differently, depending on what atoms make up the crystal lattice and how these atoms are arranged.

### **II. METHODOLOGY**

In X-ray powder diffractometry, X-rays are generated within a sealed tube that is under vacuum. A current is applied that heats a filament within the tube, the higher the current the greater the number of electrons emitted from the filament. This generation of electrons is analogous to the production of electrons in a television picture tube. A high voltage, typically 15-60 kilovolts, is applied within the tube. This high voltage accelerates the electrons, which then hit a target, commonly made of copper. When these electrons hit the target, X-rays are produced. The wavelength of these X-rays is characteristic of that target. These X-rays are collimated and directed onto the sample, which has been ground to a fine powder (typically to produce particle sizes of less than 10 microns). A detector detects the X-ray signal; the signal is then processed either by a microprocessor or electronically, converting the signal to a count rate. Changing the angle between the X-ray source, the sample, and the detector at a controlled rate between preset limits is an X-ray scan. When an X-ray beam hits a sample and is diffracted, we can measure the distances between the planes of the atoms that constitute the sample by applying Bragg's Law. Bragg's Law is  $n\lambda = 2d \sin \theta$ , where the integer  $n$  is the order of the diffracted beam,  $\lambda$  is the wavelength of the incident X-ray beam,  $d$  is the distance between adjacent planes of atoms (the  $d$ -spacing's),

and  $\theta$  is the angle of incidence of the X-ray beam. Since we know  $l$  and we can measure  $\theta$ , we can calculate the d-spacing. The geometry of an XRD unit is designed to accommodate this measurement (fig.).6.7 the characteristic set of d-spacing's generated in a typical X-ray scan provides a unique "fingerprint" of the mineral or minerals present in the sample. When properly interpreted, by comparison with standard reference patterns and measurements, this "fingerprint" allows for identification of the material.

X-ray diffraction is widely used to identify crystalline phases, measure crystallite sizes, lattice parameters, Orientation and provide quantitative phase analysis and atomic coordinates. This information is important for relating the production of a material to its structure and hence its properties. As well as being of academic interest, X-ray results are used in patent disputes, forensically and for quality control. it can be also use for identification of cement material.

### III. MATERIAL AND COMPOSITION

#### 3.1 SPECIFICATION OF MATERIALS

Table 1: Specifications of the materials used for RPC

Sample	Specific Gravity	Particle Size Range
Cement (IS 12269: 1987)	3.15	31 $\mu\text{m}$ – 7.5 $\mu\text{m}$
Silica fume (ASTM C1240-97B)	2.2	5.3 $\mu\text{m}$ – 1.8 $\mu\text{m}$
Quartz sand	2.7	5.3 $\mu\text{m}$ – 1.3 $\mu\text{m}$
Steel fiber - 13mm (ASTM A 820-96)	7.1	Length 13 mm and dia. 0.45 mm
Steel fiber – 25mm (ASTM A 820-96)	7.1	Length 25 mm and dia. 0.4 mm
20mm Aggregate (IS. 383:1970)	2.78	25mm – 10mm
10mm Aggregate (IS. 383:1970)	2.78	12.5mm – 4.75mm
River sand (IS 383: 1970)	2.61	0.6 mm – 0.15 mm

#### 3.2 COMPOSITION OF REACTIVE POWDER CONCRETE.

RPC is composed of very fine powders (cement, sand, quartz powder and silica fume), steel fibers (optional) and super plasticizer. The super plasticizer, used at its optimal dosage, decreases the water to cement ratio (W/C) while improving the workability of the concrete. A very dense matrix is achieved by optimizing the granular packing of the dry fine powders. These Reactive Powder Concretes have compressive strengths ranging from 160 MPa to 800 MPa.

Table 2: Optimized composition of RPC used in Experimental program (parts by mass)

Materials	Part by mass of cement
Cement	1
Sand	1.5
Silica fume or pozzolan	0.32
Powdered quartz sand	0.36
Steel fibers	0.20
Water	0.23
Super plasticizer	0.04

#### IV. RESULT AND DISCUSSION:

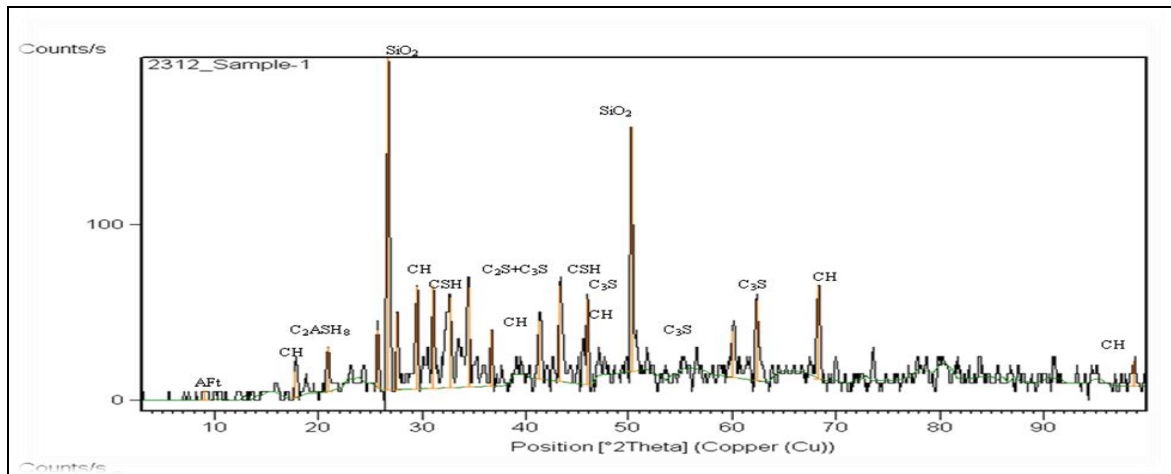


Fig. 1: X-ray diffract grams of RPC after curing for 1 day

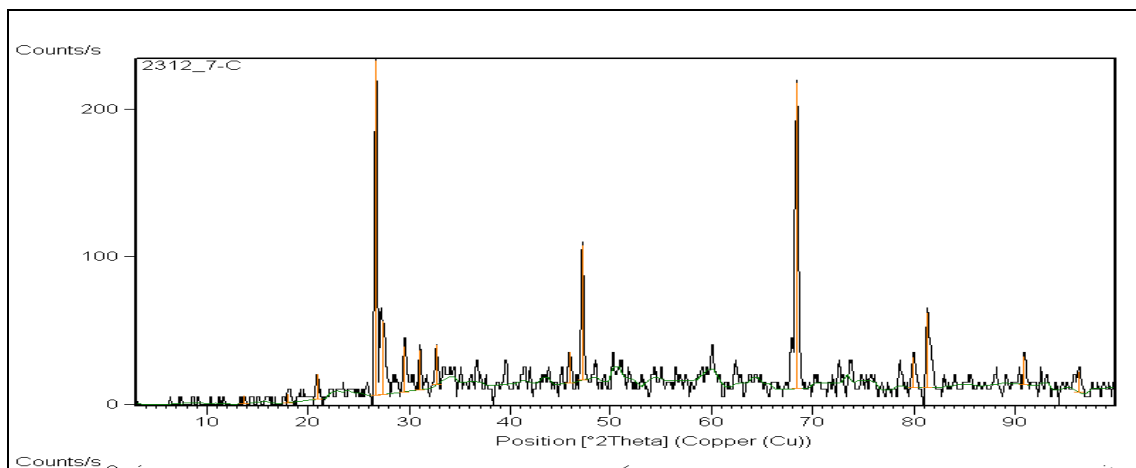


Fig.2 X-ray diffractograms of RPC after curing for 7 days normal curing

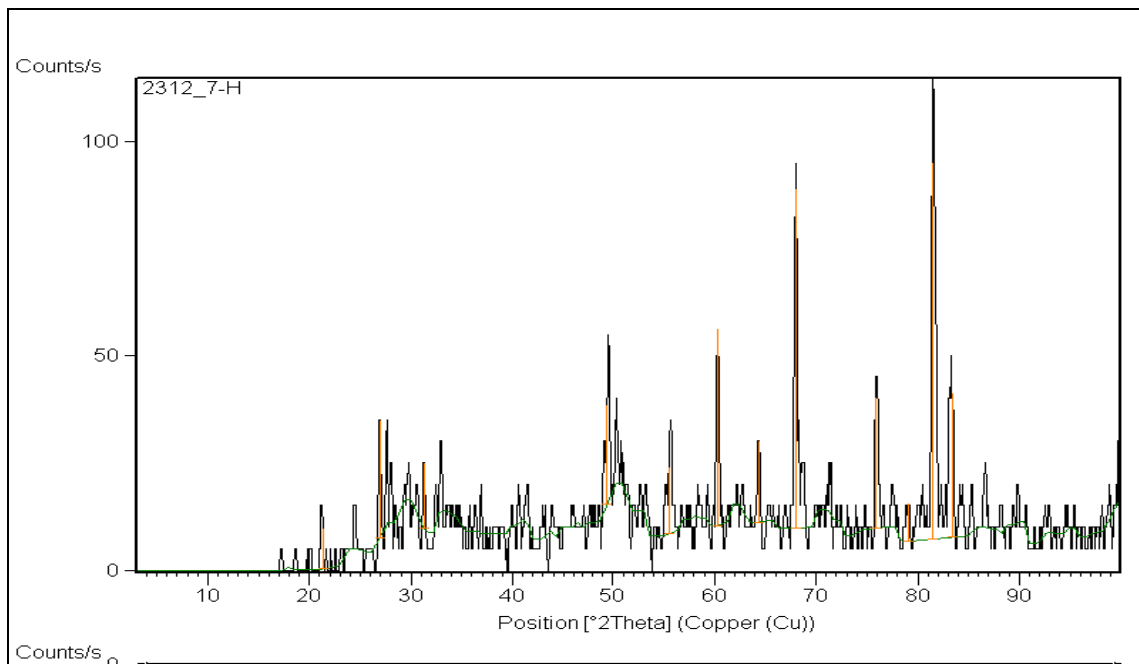


Fig 3: X-ray diffractograms of RPC after curing for 7 days hot curing

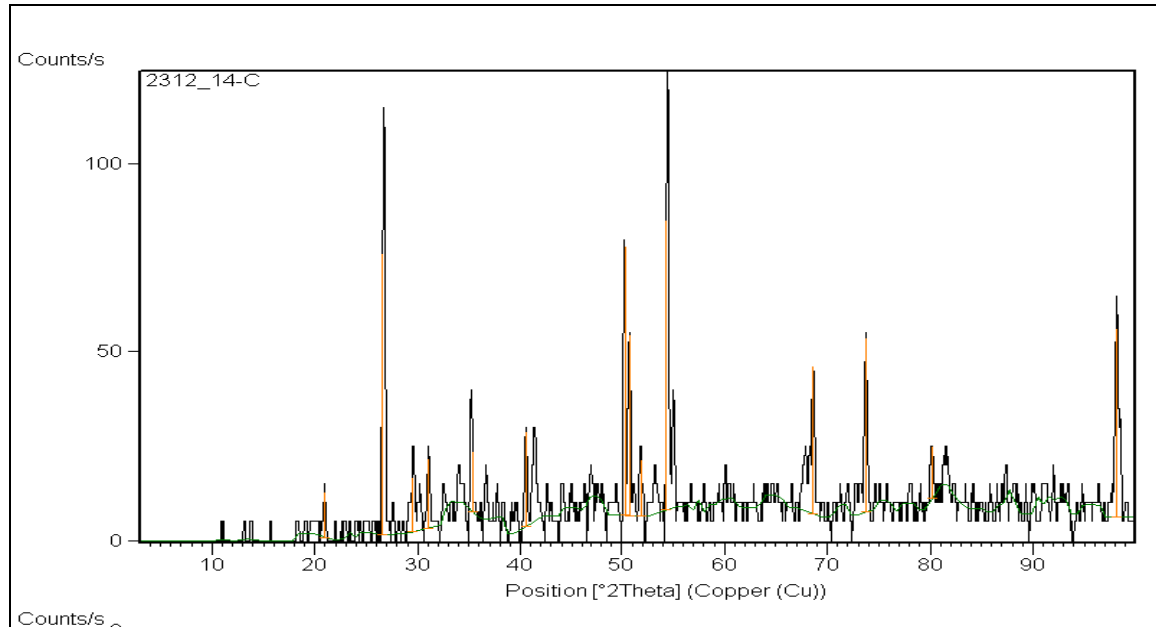


Fig.4: X-ray diffractograms of RPC after curing for 14 days normal curing

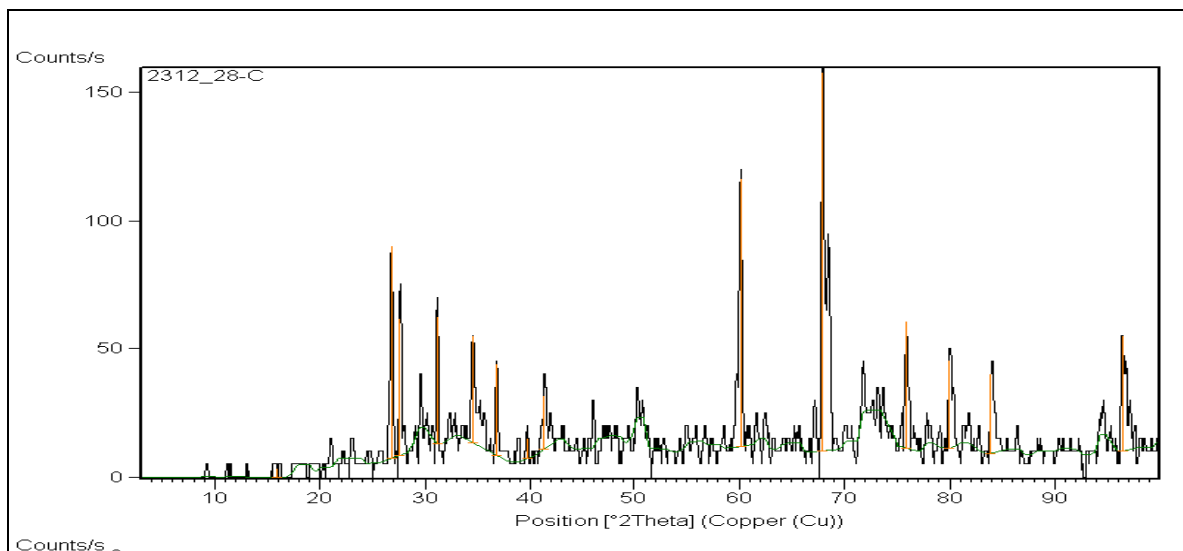


Fig.5: X-ray diffract grams of RPC after curing for 28 days normal curing

Result **Fig.1** shows the XRD patterns of bulk paste for 2.0% of steel fiber Reactive Powder Concrete, which were cured in 28-days water immersion. All of them contained Portlandite (CH) and  $\text{SiO}_2$  as a major phase. From the diffract grams patterns in sample-1, it was observed that the intensity of CH is higher and the C-S-H was lower in RPC content mix. It might be indicated that the degree of hydration in RPC with 7days hot was curing (7-H) much higher than the same sample with 28days normal water curing (28-C). The reason could be that the hot curing provided the buster or accelerating the hydration process for concrete.

**Fig.1.to Fig.5** Shows the XRD patterns of bulk pastes for sample-1. All of them contained Portlandite (CH) and  $\text{SiO}_2$  were the major phases in all samples of RPC. From the diffract gram patterns in Fig.1, it is observed that the intensity of Calcium-Silicate-Hydrate (C-S-H) in 7days (7-H) hot water carried and 28days (28-C) normal water carried was higher than in sample-1(1days normal carried). This may indicate that the degree of hydration in 7days hot water carried and 28days normal water carried concrete with high cement replacement was better than sample-1(1days normal carried) mixes under the same steel fiber contain. Further, the intensities of Portlandite (CH) in sample-1(1days normal carried) were higher than that in 7days hot water carried. The reason might be that the content of CH was prorated to the creative degree of C-S-H.

This may indicate that the degree of hydration in 7-H concrete with high cement replacement was better than Sample-1, 7-C, 14-C, and 28-C mix under the same sample. Further, the intensities of Portlandite (CH) in Sample-1, 7-C, 14-C and 28-C were higher than 7-H concrete. In this spectra  $C_3S$  and  $C_2S$  peaks characteristic of unhydrated cement, are detected, even for high temperature for curing.

## V. CONCLUSIONS

1. RPC microstructure depends on heat treatment or curried condition. XRD studies made it possible to gain better understanding of the micro structural changes induced due to curing condition. It shows that hot curried water sample led to faster change in microstructure of C-H-S hydrates. Compare to normal water curried sample.
2. It can observe change in the microstructure. The effect of mineral admixture (silica fume and quartz sand) help in developing dance microstructure of RPC.
3. As hydration increases consumption of  $CAOH_2$  also can be observed and still there is anhydrated cement is remaining in a powder of RPC.

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