INVESTIGATION ON THE EFFECT OF WATER FLOW ON CEMENT PASTE MICROSTRUCTURE USING NON-DESTRUCTIVE INTEGRATED CT-XRD METHOD

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1. INTRODUCTION

Concrete is one of the most widely used construction materials in the world. Many concrete structures like hydraulic structures, water purification plants, radioactive waste containers, underground structures, tunnels etc. are contacted with water. When these structures contact with water for a long time, there will be a chance of leaching which degrades the performance of structures. Leaching changes the pore structure which has significant effects on physical characteristics (strength, permeability etc.) as well as Chemical properties of pore water (pH, element concentration etc.) In this study, the leaching behavior of hardened cement paste has been investigated by means of nondestructive integrated CT-XRD method. Water flow test was conducted, where demineralized water passes through the crack specimen of hardened ordinary Portland cement paste for different time interval. Three dimensional (3D) images was obtained from X-ray computed tomography (CT) which provides the physical information like the cracks geometry, air voids etc. Then X-ray diffraction (XRD) analysis was conducted on the specific region to know the chemical change over time and space.

2. MATERIALS AND METHODS

2.1 Non-Destructive Integrated CT-XRD Method

The nondestructive integrated CT-XRD method was conducted at BL28B2, SPring-8, Japan [1,3]. In this method white X-ray was used as the incident X-ray which covers a wide range of wave length. The energy of X-ray for CT measurement was 25 keV. Number of projection was 912 with an exposure time of 0.15 s. There were 754 pixels and each pixel size was 7.35 μm. The overall field of view was about 5 mm. The beam size was 0.05 mm in width and 0.15 mm in height. The angle of diffraction (θ) was fixed at 10°. The preset time was 300 s. Solid-state detector was used to get the energy-intensity relationship. First CT measurement was carried out to get the reconstructed cross-sectional images. Then ROI (Region of Interests) were selected on specific cross sectional image to conduct XRD. The relationship between X-ray energy and the intensity of the diffracted X-ray was obtained from XRD. Then this energy-intensity relationship was converted to diffraction angle-intensity relationship where energy was converted to diffraction angle using Bragg equation (Eq.1) and specific wavelength.

$$n\lambda = 2d\sin\theta \qquad (1)$$

Here n is a positive integer, λ is the wavelength of incident wave, d is the interplanar distance and θ is the scattering angle.

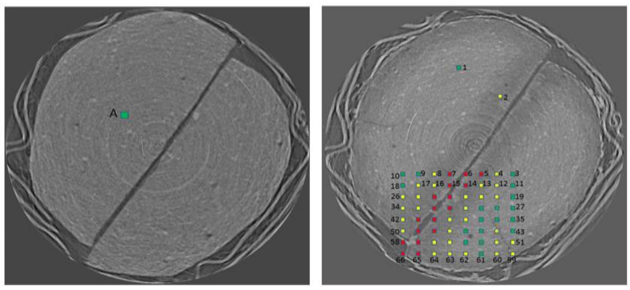
2.2 Specimen Preparation

Ordinary Portland cement (OPC) was used in this study. First hardened OPC paste (40 mm x 40 mm x 160 mm) was made with water to cement ratio of 0.3. The hardened paste was cured in water for 28 days. Then it was cut to get the cylindrical specimen of about 5 mm diameter and 5 mm height. Crack was induced on that cylindrical specimen. Aluminum tape was used to cover the crack specimen and to connect plastic tubes of 2 mm inner diameter at both ends of that specimen. Next demineralized water was passed through the tube for conducting leaching test.

2.3 Leaching Test

Tubing pump was used to circulate the water through the specimen. Demineralized water was kept at a tank from which water was circulated through the cracked specimen via the tube pump. Water flow rate was 50 cc/h and it was continued for a period of around 6 months. After certain leaching period, the specimen was moved to SPring-8 for conducting nondestructive integrated CT-XRD measurement.

3. RESULTS AND DISCUSSIONS



a) Before leaching b) After 6 months leaching

Fig. 1 Cross Sectional CT images with XRD measurement position

Fig. 1 shows the cross sectional image of the cracked specimen which are located at the central portion of the specimen (1.90 mm from the top surface of the specimen). Using gray scale value (GSV), CT image provides the physical information like location of crack, air voids, low or high density substances etc. A low density substance has low GSV whereas a high density substance has high GSV. But to know the chemical change XRD was conducted on some specific position.

Before leaching one region of interest (A) was selected for XRD measurement. After leaching to know the proper distribution of chemical change, many region of interest (1~66) were selected. Some positions situated near crack region and some are far from the crack (Fig. 1b).

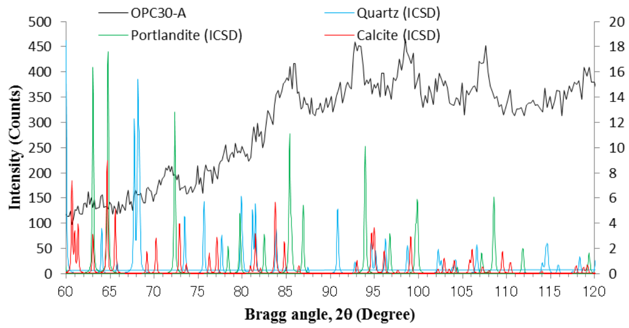


Fig. 2 XRD spectrums at position A (Before leaching)

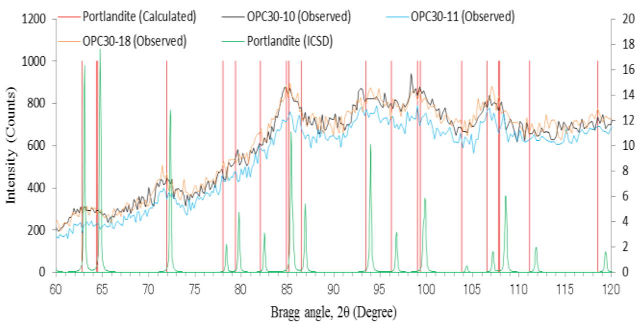


Fig. 3 XRD spectrums at position 10, 11 and 18 (After leaching)

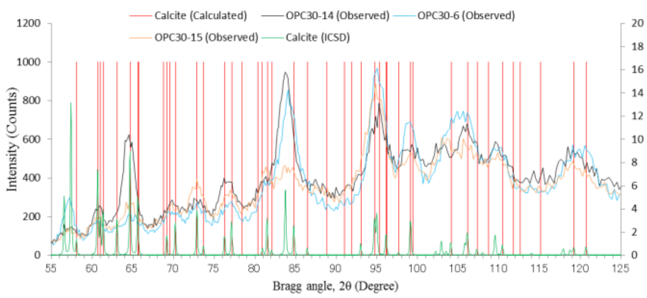


Fig. 4 XRD spectrums at position 6, 14 and 15 (After leaching)

Diffraction spectrum of location A (before leaching) is shown in Fig. 2. This spectrum is compared with ICSD (Inorganic Crystal Structure Database) spectrum for different cement hydrates like portlandite [$Ca(OH)_2$], calcite ($CaCO_3$), quartz (SiO_2) etc. From Fig. 2 it is seen that most of the significant peak position of location A is match with portlandite (ICSD) peak position. As before leaching other locations were not effected, so it can be considered that before leaching other regions were similar. For more reliability besides the ICSD observed peak position was also compared with calculated peak positon (Fig. 3 and Fig. 4). Calculated peak position was obtained using Bragg's law, different combination of possible miller indices and lattice parameter of specific mineral [2]. Diffraction spectrum of leaching sample at the location of 10, 11 and 18 which are far (more than 0.70 mm) from the crack are shown in Fig. 3. From Fig. 3 it is seen that most of the peak position match with portlandite (ICSD and calculated) peak position. Fig. 4 shows XRD spectrum of location 6, 14 and 15 which are near crack region. From Fig. 4 it is seen that almost all peak position are matched with calcite peak position.

Same diffraction spectrum comparison was conducted for all locations (1~66) of leaching sample. It was seen that near crack region (red and yellow marked location in Fig. 1) and some outer region (yellow marked) where water was passed most of the portlandite peak was diminished. But far from crack regions (green marked in Fig. 1) are portlandite riched, where most of the peak position match with portlandite peak position as like as Fig. 3. From near crack regions (red marked in Fig. 1) diffraction spectrum it was seen that most of the peak position match with calcite peak position as like as Fig. 4. Several times this sample was moved to Spring-8 for conducting CT-XRD measurement. So there was a possibility of contact with CO_2 which was optimal condition for carbonation and leads $Ca(OH)_2$ to convert calcite.

4. CONCLUSIONS

From the above result it is seen that as the water passes through the crack after a certain period Portlandite is dissolute from near crack region of the cement paste. This dissolution rate near crack is more severe. In addition, as the leaching time increase Portlandite dissolution is also increased. After a certain time interval (6 months leaching) there were the presences of Calcite near crack region. May be CO_2 from air react with $Ca(OH)_2$ and cause this carbonation.

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