

# Microstructural Evaluation of Durability of Different Cementitious Mixtures in Microbial Induced Corrosion Environments

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**Keywords:** *Microstructure, Fluorescence Microscopy, Microbial Induced Corrosion, Cementitious Materials, Image Analysis.*

## 1 Scopes and Aims

Microbial Induced Corrosion (MIC) is a significant concern for the condition of concrete infrastructure elements in a variety of environments. This study assesses the resistance of three of the most commonly used materials for wastewater treatment plants (WWTP) facilities: ordinary portland cement (OPC), calcium aluminate cement (CAC), and alkali activated fly ash and slag (AAC). Optical and electron microscopical methods are used to characterize the response of these materials to aggressive conditions in a biogenic corrosion chamber with measurements of the evolution of the capillary porosity of the binders from fluorescence.

## 2 Materials and Methodologies

This study investigates three concrete mixtures made with OPC (w/b = 0.36), CAC (w/b = 0.42), and AAC (w/b = 0.43), respectively. All of the specimens were cured for 28 days at  $22 \pm 1$  °C. The concretes were then partially submerged in real sewage water in an enclosed chamber at 25 °C and 100% relative humidity for 12 months. The specimens were then removed from the chamber and washed to remove loose material. Petrographic examinations were performed on polished slab (Nikon<sup>®</sup> SMZ-25 stereomicroscope), thin section impregnated with fluorescence dye epoxy (Nikon<sup>®</sup> E-Pol 600 petrographic microscope), and environmental scanning electron microscopy (FEI<sup>™</sup> Quanta 250 ESEM) coupled with energy dispersive X-ray spectrometer (EDAX<sup>®</sup> Apollo X Silicon Drift). Images analysis was performed on fluorescence images (Image J) to quantify the capillary porosity of the paste.

## 3 Results

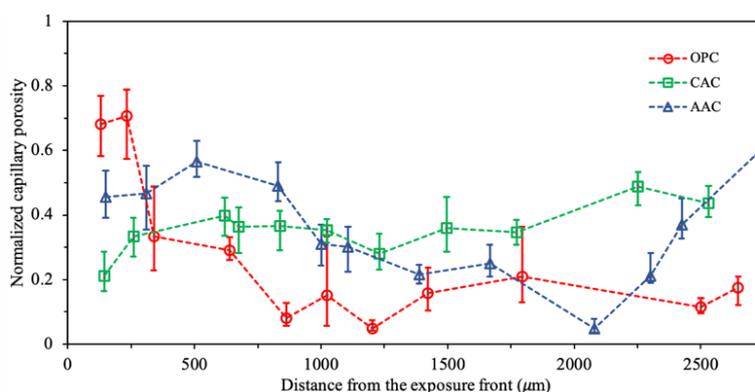
Changes in the capillary porosity of the paste were measured across alteration zones in each concrete. As a benchmark, the grey level histograms of paste and a void were analyzed and statistically fitted assuming normal distributions. A “normalized capillary porosity” of the paste is proposed by normalizing the mean grey level of the paste to that of the air void. The change in normalized capillary porosity coincide broadly with alteration zones, which are in turn dependent on the binder type, as shown in Fig. 1.

The alteration zones in OPC concrete are as follows (inward from the exposed surface): (a) coarse-grained gypsum; (b) medium-grained gypsum; (c) a transition zone with ettringite and iron oxide staining; (d) a carbonated zone; and (e) a zone of calcium leaching. The formation

of gypsum consumes calcium hydroxide and calcium silicate hydrates in the paste. The leaching of cementitious phases and deposition of gypsum produces a higher capillary porosity in direct contact with the acid in Zone (a)-(b). Ettringite forms inwards in Zone (c). The iron staining marks the oxidization of iron-bearing minerals in the aggregate. These reactions are expansive, which lowers the capillary porosity in Zone (c), compared to that of the intact paste.

The alteration zones in CAC concrete are as follows (inwards from the exposure surface): (a) a zone of cracking with deposition of gypsum; (b) a transition zone that contains gypsum, ettringite and iron oxide staining; and (c) sulfate diffusion zone. Gypsum and gibbsite form from hydrogarnet in the presence of sulfuric acid. The reaction is expansive and leads to cracking in Zone (a). Gypsum reacts with hydrogarnet to form ettringite in the inner Zone (b)-(c), which densifies the microstructure of the paste and thus a lowered capillary porosity.

The alteration zones in AAC concrete are as follows (inwards from the exposure surface): (a) a zone of cracking with deposition of gypsum; (b) a transition zone containing gypsum, ettringite and iron oxide; and (c) sulfate diffusion zone. Calcium (alkali) aluminosilicate hydrate and monosulfate (AFm), react with  $H_2SO_4$  to form gypsum, which results in cracking and thus a higher capillary porosity in Zone (a), compared to that of the intact paste. The transition zone and diffusion zones show lower capillary porosity due to the formed ettringite from AFm.



**Figure 1.** Line plot showing changes in capillary porosity of the paste in the alteration zones of three mixtures.

## 4 Conclusions

Observations from optical and electron microscopy delineate alteration zones characterized by distinct changes in mineralogy and the capillary porosity of the paste in each mixture. The OPC mixture shows deterioration where the capillary porosity of the outer alteration zones increases from the leaching of  $Ca(OH)_2$  and CSH and cracking is associated with the deposition of gypsum. The CAC and AAC mixtures show greater resistance to the MIC. The outermost alteration zones in these mixtures have markedly lower capillary porosity than the OPC mixture. As such, the alteration zones are more cohesive and remain adhered to the intact paste. The higher resistance is consistent with better resistance to leaching than the OPC mixtures, which is in turn due to the lack of  $Ca(OH)_2$  and CSH in these materials.

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