Microstructure Evolution from X-CT Measurements for Concrete/mortar under Multi-actions of Composite Salts Dry-wet Cycles and Loading

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Abstract: In this research, microstructure evolution for concrete/mortar under multi-actions of composite salts dry-wet cycles and loading was investigated through X-CT measurements. The evolution process of pores and micro-cracking with the erosion time were tracked. Compared the different erosion actions, it was found that dry-wet cycles promoted the pores become connected gradually. Besides, the dry-wet cycles accelerated the damage seriously on interface area between concrete and aggregate, whistle, loading contributes to the cracking propagation toward the internal. Moreover, fly ash played a positive role in the increasing of the number of harmless holes again and contributed to the durability of concrete.

1. Introduction
Concrete is a kind of typical porous material, and which performance mainly depends on the pore structure. As porous system composed of different scale and size of pores, concrete included coarse pores (100μm-10mm), capillary pores (0.05μm-100μm) and gel pores(<50nm). Numerous researchers put different views and methods with regard to the classification of the pores[1,5]. For instance, the most popular classification in China about pores in concrete was put forward by Wu[6] based on the influence of performance on concrete, namely harmless pores (<20nm), less harmful pores (20-100nm), harmful pores (100-200nm), more harmful pores(>200nm), whilst suggested that it is a good method on improve the durability of concrete by reducing harmful pores above 100nm and increasing the harmless or less harmful pores below 50nm.

X-CT testing method could be used to detect the internal micro-cracking and pores or holes distribution on concrete, in other words, estimating the interior damage process on concrete as a kind of non-destructive and simple testing technology. Only simple samples directly getting from the corrosion concrete could satisfy the requirement of X-CT testing. Since the resolution of X-CT is micro-scale, the sub-micro-scale and nano-scale pore information couldn’t be obtained. Therefore, the pore volume obtained from this experiment is more than 1.5 × 10⁻³ mm³, namely, the pore diameter is more than 140nm. Compared with the MIP test, X-CT detect the larger size of the pore distribution, which belong to harmful pores according to Wu’s classification, in others words, which is harmful
holes or cracking. In this research, Precision S type X-CT scanner from German company YXLON was adopted in Figure 1, and its working principle shown in Figure 2.

![Fig. 1 X-CT for Precision S](image1)

![Fig. 2 X-CT Working Principle](image2)

### 2. Materials and mix proportions

Materials used in this study consisted of ordinary Portland cement (P.II.52.5 according to Chinese standard), Fly ash (FA) and grounded blast furnace slag (GBFS). The chemical and mineral compositions are summarized in Table 1. River sand with fineness modulus of 2.6 and coarse aggregate of crushed limestone with a maximum size of 20mm were used. A polycarboxylate-type super-plasticizer with a water-reducing rate of 25% by weight was used. The mix proportion is given in Table 2, where the mix code C50F30 means mortar specimen with the binder replaced by FA at the dosage of 30% by weight, while the C50K50 means mortar specimen with the binder replaced by GBFS at the dosage of 50%.

Concrete specimens were cast in steel molds of 70×70×280 mm, removed from the molds 24 h after casting, and cured in the condition of 20±3°C and 95% relative humidity for a certain days. Except for the two opposite surfaces (70×280 mm), other four surfaces of the specimen were covered with epoxy resin before the exposure experiments.

To examine the micro-evolution process of concrete/mortar under the multi-action of chloride and sodium sulfate, drying-wetting cycles, and loading, three different mortar mixtures were prepared with the same binder types as used in the concrete mixes and cured in the condition of 20±3°C and 95% relative humidity until the age of testing. The paste samples were also produced for X-CT testing.

| Material | Chemical compositions (wt. %) | Mineral compositions (wt. %) |
|----------|-----------------------------|-----------------------------|
|          | CaO | SiO₂ | Al₂O₃ | Fe₂O₃ | MgO | SO₃ | K₂O | Na₂O | LOI |
| Cement   | 62.53 | 20.75 | 4.78 | 3.45 | 1.11 | 2.64 | 0.58 | 0.07 | 3.26 |
| FA       | 5.75  | 51.07 | 30.86 | 5.26 | 2.72 | 1.48 | 1.13 | 0.79 | 2.80 |
| GBFS     | 36.35 | 33.48 | 12.21 | 1.40 | 10.60 | 0.66 | 0.56 | 1.27 | 0.36 |
| Cement   | C₃S | C₂S  | C₃A  | C₄AF |
|          | 53.07 | 19.47 | 9.46  | 10.49 |

Table 1 Chemical and mineral compositions of raw materials (wt%)
Table 2 Mix proportion of concrete and mortar specimens

| Mix   | Binder (kg/m³) | Fine aggregate (kg/m³) | Coarse aggregate (kg/m³) | Water (kg/m³) | Super plasticizer (kg/m³) | w/b |
|-------|---------------|------------------------|--------------------------|--------------|---------------------------|-----|
| C50   | 448           | -                      | 673                      | 1121         | 157                       | 4.03| 0.35 |
| C50F30| 314           | 13                     | 673                      | 1121         | 157                       | 4.03| 0.35 |
| C50K50| 224           | 13                     | 673                      | 1121         | 157                       | 4.03| 0.35 |
| P35   | 1494          | -                      | -                        | 523          | -                         | 0.35|
| P35F30| 1046          | 44                     | -                        | 523          | -                         | 0.35|
| P35K50| 747           | 747                    | 523                      | -            | -                         | 0.35|

3. X-CT testing results and discussion
3.1 Results from the corrosion concrete/paste with 0.35 w/c under the different actions of composite salts solution and dry-wet cycles

(a-1) Before erosion
(a-2) 120 days after dry-wet cycles
(a-3) 210 days after dry-wet cycles
(a-4) 210 days after immersion cycles
(b-1) Before erosion
Fig. 3 X-CT patterns from different hardened cement paste/concrete (w/c=0.35) exposed to composite solutions of chloride and sodium sulfate (5%NaCl+5%Na₂SO₄) under 1d-2w cycles or immersion.

**Note:** (a)cement paste(b)concrete(a)

Fig. 4 Pores defect from X-CT patterns
Fig. 5 Porosities from X-CT patterns

The color scale in the X-CT tomography represents the order of the pore volume, where blue is the smallest and the red is the largest. Figure 3 shows an apparent views of the hole defects in the past or concrete that were subjected to different erosion actions. For example, for hardened cement paste under dry-wet cycles, with the increase of the age of erosion, round holes reduced while the long columnar pores increased gradually, which indicated that the pores in the paste are gradually become connected. Whistle compared to immersion action, the pore size of the round hole gradually becomes smaller than that before the erosion, but the connected hole is not found. Figure 3 (b) shows that the number of observable pores decreases with the increase of erosion age, and long columnar holes increase with decreases of the round hole; compared with that immersion, the long columnar (the red hole) at the boundary (concrete and aggregate interface) is longer much, indicating that the dry-wet cycles accelerated the damage seriously on interface area between concrete and aggregate.

The statistics to porosity defects in Figure 3 are shown in Figure 4. The results presented that whatever paste or concrete, the volume of holes below 0.1mm$^3$ accounted for about 90% of the sum of all the holes. For both samples under dry-wet cycles, with the increase of the age of erosion, the amount of 0.01~0.1 mm$^3$ holes reduced compared to that before erosion due to the formation of the erosion products from the salt solution gradually filling the pores. In addition, for the paste, the number of 0.01~0.1 mm$^3$ holes after 210 days dry-wet cycles is more than that after immersion for 210 days, which could be explained that the dry-wet cycles promoted the pores to be gradually connected; but for the concrete after 150 immersion, the number of holes in this scales have no different.

Figure 5 shows porosity testing results from X-CT under different erosion conditions corresponds to Figure 4. For paste samples under dry-wet cycles, with the increase of the age of erosion, the porosity reduced, namely, 1.29% for before erosion, 0.89% for after dry-wet cycles 120d, and 0.58% for after dry-wet cycles 210d; for paste samples under immersion, the porosity also reduced with the erosion age, compared to that before erosion, but reduced less than that under dry-wet cycles. Besides, The concrete samples has the similar change trend with paste.

3.2 Results from the different types of corrosion concrete/paste with 0.35 w/c under the action of composite salts solution and dry-wet cycles
Fig. 6 X-CT patterns from different cement paste (w/c=0.35) immersed in composite solutions of chloride and sodium sulfate (5%NaCl+5%Na₂SO₄)

Fig. 7 Pores defect from X-CT patterns

As can be seen from Figure 7 (a) (porosity statistic from Figure 6), the common paste has the similar amount of >0.05mm³ holes with the paste samples adding mineral admixtures, which scale belongs to harmful holes; but the difference is the scale <0.01, which belongs to harmless holes, and the paste with fly ash mixture is higher than the latter two, indicating that fly ash played a positive role in the increasing of the number of harmless holes. Figure 7 (b) shows the concrete with mineral mixture has less amount of 0.01~0.1mm³ (harmful holes) than the common concrete, especially the fly ash addition concrete, which verified fly ash played a positive role in the increase of the number of harmless holes again and contributed to the durability of concrete.
In Figure 8 (a), different types of pastes porosity from X-CT test follows that P35>P35F30>P35K50, due to the addition of mineral admixtures does refine the pore structure, and combined Figure 6, in addition to the red holes (belonging to the hole), the paste presents more green holes (harmful holes). Whistle in Figure 7(a), all the three have the similar numbers of above 0.05 mm$^3$, even if the P50F30 has a large number of pores of about 0.01 mm$^3$, but the contribution of the total porosity is less because the pore volume is too small. On the whole, the porosities in Figure 8(b) could be explained by Figure (b).

3.3 Results from the corrosion paste with 0.35 w/c under the action of composite salts solution, dry-wet cycles and loading

Figure 9 shows the X-CT defect analysis of mortar specimens after 105 days under the actions of composite salts solution, dry-wet cycles and different loading (load stress ratio of 0, 30% and 50%, respectively). The red color represents micro-cracking, the bright red is surface cracking and dark red is internal cracking. As can be seen, compared to that under loading action, all the micro-cracking scattered on the surface of the specimen without loading action, without extended to the internal, which revealed that loading could promoted the cracking propagated toward the internal. There are three characters to be concluded from Fig.9 (b),(c): (1)The crack is concentrated on nearby of the two fulcrums in the tension zone of the specimen and extends to both sides of the fulcrum. (2)The crack starts from the non-molding surface of the tension zone and extends to the inside of the specimen under the action of tensile stress. (3) with the stress ratio increases, the effects from (1) and (2) are more and more obviously. Excepted the crack the deformation also show the similar trends.
Figure 10 shows three-dimensional reconstruction from the X-CT defect analysis of mortar specimens after 105 days under the actions of composite salts solution, dry-wet cycles and different loading (load stress ratio of 0, 30% and 50%, respectively). The test specimen size of about 38mm * 38mm * 38mm ≈ 1/4 standard mortar specimens, and nearby bending load zone. It can be observed that the overall defect increases with the stress ratio increases, and the defects are mainly concentrated on tensile zone. The defects exist only in the tension zone for the specimen under 30% loading, and the compressive zone is difficult to find defects. While for specimen under 30% loading, the defects almost cover the whole tension zone, and which significantly extended to the compressive zone. It could conclude that the increasing on the stress ratio leads to a significant expansion of the distribution of defects in both horizontal and vertical directions.

![Figure 10](image)

Fig.10 Three-dimensional reconstruction from X-CT defect patterns from different cement paste (w/c=0.35) immersed in composite solutions of chloride and sodium sulfate (5%NaCl+5%Na$_2$SO$_4$) under the action dry-wet cycles and loading.

Table 3 shows computer calculated porosity results from the three-dimensional reconstruction patterns, and verified the results from three-dimensional reconstruction quantitatively. The porosity are 5.14, 7.62 and 11.41% corresponding to 0, 30%, 50% loading. It could be found that the loading not only improved the porosity of the tensile zone and the compressive zone but also widened the gap between the tension zone and the pressure zone.

| Loading | Porosity of the whole | Porosity of the tensile zone | Porosity of the compressive zone |
|---------|-----------------------|-----------------------------|---------------------------------|
| 0       | 5.14%                 | -                           | -                               |
| 30%     | 7.62%                 | 9.00%                       | 7.08%                           |
| 50%     | 11.41%                | 15.68%                      | 8.27%                           |
4. Conclusions
Based on the results from this study, the following conclusions may be drawn:

For hardened cement paste and concrete, dry-wet cycles promoted the pores become connected gradually. Besides, the dry-wet cycles accelerated the damage seriously on interface area between concrete and aggregate.

It is verified that fly ash played a positive role in the increasing of the number of harmless holes again and contributed to the durability of concrete.

It could conclude that loading contributes to the cracking propagation toward the internal, moreover, the increasing on the stress ratio leads to a significant expansion of the distribution of defects in both horizontal and vertical directions.

It could be found that the loading not only improved the porosity of the tension zone and the compressive zone but also widened the gap between the tension zone and the pressure zone.

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