



Durability of blast-furnace slag mortars subjected to sodium monofluorophosphate application

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ABSTRACT

The aim of this study is to investigate the effect of sodium monofluorophosphate (Na-MFP) on frost scaling durability and transport properties of carbonated blast-furnace slag cement (BFSC) mortar. The application of Na-MFP solution as curing solution and also surface treatment compound for BFSC mortar was evaluated. Three different Na-MFP solutions with concentrations of 10%, 20% and 30% by weight, in water were used as curing solution and surface treatment compound. The experimental results reveal that both techniques significantly improve the frost salt scaling durability and microstructure of carbonated blast-furnace slag mortar. Moreover, the carbonation rate and capillary water uptake of the treated mortars were substantially decreased compared with the untreated specimen. The mortars cured in Na-MFP solution show higher compressive strength than the untreated control after carbonation exposure. In general, the durability performances were improved when increasing the concentration of the Na-MFP solution applied.

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1. Introduction

Blast-furnace slag (BFS) is a by-product in the production of iron and steel manufacturing. It is classified as a latently hydraulic material which can exhibit a hydraulic activity only with the presence of appropriate activators. It is widely accepted that BFS concrete produces a low heat of hydration, and have a superior durability against aggressive environment. The superior durability of BFS concrete against chemical attacks allows this mineral admixture a suitable binder for the concrete exposed to chloride, acid and sulfate attacks. However, it has been reported that concretes bearing high amount of BFS have a poor resistance against carbonation. The carbonation on BFSC mixture transforms the C–S–H gel to porous silicate hydrates, and the strength reduction of the matrix might be expected. Consequently, the carbonated BFS concrete is vulnerable to scaling under the combined load of freezing–thawing and de-icing salt. Due to concrete surface disintegration, also other chemical attacks on the structure have more chance, which can result in a dramatic decrease in durability.

Sodium monofluorophosphate (Na-MFP) is widely used in concrete industry as a surface applied corrosion inhibitor. A number of researches [1–4] investigated the interaction of Na-MFP and cement hydrated products and its effect on inhibiting corrosion of

the embedded rebar. The basic mechanism of Na-MFP treatment is to protect the passive layer of the reinforcement against disruption due to carbonation. However, it has been recently found that Na-MFP has another benefit to improve the durability of blast-furnace slag cement (BFSC) mixtures. In previous studies [5–7], it has been reported that the application of Na-MFP treatment increases the frost salt scaling resistance of carbonated BFSC pastes and mortars. The 10% Na-MFP solution was used as a surface treatment compound on carbonated BFSC specimens. In general, the results show that the treatment modifies the mineralogical structure and improves the resistance of carbonated BFSC paste against frost salt attack. The carbonation rate and capillary water uptake of the treated mortars were substantially decreased compared with the untreated specimens. The quality of the interfacial transition zone (ITZ) between sand and paste has been significantly improved. The technique is effective particularly for highly carbonated surface since a thick resistant zone can be created by the application.

In this study, apart from surface treatment, the application of Na-MFP solution as a curing solution for the BFS mortars was also investigated. The effect of Na-MFP application on microstructure, transport properties, strength development and frost salt scaling durability of carbonated specimens was evaluated. The solubility limit of Na-MFP in pure water is about 40–42% by weight. To optimize the healing process, several concentrations of the Na-MFP solution (up to 30% by weight) have been investigated in this study.

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2. Experimental programs

2.1. Materials and preparation

A BFSC with a slag content of 67% (CEM III/B 42.5 N HSR LH) from ENCI cement manufacturer was used. The typical chemical composition and physical properties of the cement is given in Table 1. The mortar specimens were prepared by controlling a w/c ratio of 0.45 and sand-to-cement ratio of 2.5 by weight. De-ionized water was used in mixtures throughout the experiment. The mixing sequence was 2 min low speed followed by 2 min high speed mixing with a commercial Hobart mixer. For most analyses, the mortars were cast in plastic moulds with 55 mm diameter to a height of about 50 mm. For compressive strength test, however, the 20 mm cube specimens were used for investigation.

The specimens were conditioned and treated according to the schedule shown in Table 2. After 24 h in moulds, the specimens were cured with controlling temperature of 25 ± 2 °C for 3 days period. For the control (*Con*) and surface treatment (*ST*) series, the specimens were cured in ordinary tap water, while specimens from the Na-MFP curing series (*CR*) were cured in Na-MFP solutions. Three different Na-MFP curing solutions with concentrations of 10%, 20% and 30% by weight in water were used, where the specimens were indicated as *CR-10*, *CR-20* and *CR-30*, respectively.

Thereafter, all specimens were conditioned in a CO₂ free desiccator at 50–55% relative humidity. After 10 days of the conditioning process, the Na-MFP treatment was performed on the surface treated specimens (*ST*) as the schedule shown in Table 2. Three different Na-MFP treatment compounds with concentrations of 10%, 20% and 30% by weight were applied. The specimens were designated as *ST-10*, *ST-20* and *ST-30*, respectively. De-ionized water was sprayed on treated surface after each Na-MFP treatment to promote the penetration of the compound into the specimen. The treatment was applied for six times a day providing 24 times treatments in total. During the same period, the specimens from *CR* series and the control (*Con*) were received de-ionized water instead of Na-MFP solution. This process was essential to ensure the identical pore saturation of treated and untreated specimens. Eventually, all specimens were placed in the laboratory air for another 14-days period.

All specimens were subjected to accelerated carbonation environment which provides the carbon dioxide concentration of 3% by volume, with 65% relative humidity for a period of 28 days. Before that, the circumference surface and the bottom area of cylinder specimens were coated with epoxy. The top trowel surface was only the area which was exposed to the environment. For cube specimens, no epoxy coating was required. After the carbonation period, all specimens were sprayed with de-ionized water for 3 days (six times a day). The purpose of this process is to imitate a real condition where the outdoor concrete has to be exposed to rain from time to time. The chemical reaction between Na-MFP and the products of carbonation would be enhanced with the presence of moisture in pore of specimens [7]. Eventually, specimens were bench-dried in laboratory (~40–50% relative humidity, 20–25 °C) for 28 days.

2.2. Analysis and testing methods

The frost salt scaling test by conforming the ASTM C672 freezing–thawing cycle which is consisted of 17 h in –20 °C freezing and then 7 h at 25 °C thawing was conducted. There were two methods of frost scaling experiment performed in this study as following:

2.2.1. Method A

The un-coated surface of cylindrical mortar was facing downward submerged into the 3% NaCl solution which was maintained a level of about 5 mm above the specimen surface during the cycles of freezing and thawing. The specimens were placed on plastic rack to allow free circulation of the salt solution. The total depth of salt solution was controlled at about 25 mm. The scaled material was collected by using filter paper after 1, 2 and 4 cycles. Eventually, it was oven-dried at 105 °C and weighted after 24 h drying. The scaling weight result was obtained from an average of two specimens.

2.2.2. Method B

The prepared mortar cylinder was placed in a plastic mould where the un-coated surface was upward. Beforehand, a thin rubber sheet was wrapped along the side surface of specimen for sealing-fit purpose. Approximately 20 mm of 3%

NaCl solution was maintained on top of the specimens during freezing and thawing cycles. The scaled material was collected by using filter paper after 1, 2, 4 and 7 cycles. Thereafter, it was dried at 105 °C and weighted after 24 h drying. The scaling result was obtained from an average of two specimens.

To evaluate transport properties, a mortar cylinder was placed in a tray filled with water to observe the capillary water uptake. The initial weight of specimen has been recorded before immersion. The un-coated surface was facing downward immersed into the water pond which was kept a constant level at about 5 mm above the specimen surface. The specimen was placed on glass rods to allow free access of water. The specimen had to be weighted and returned to the pond within 30 s in each investigation. Before weighing, the excess water on the surface has wiped off with a clean damp tissue paper. As a fact that the water penetration is mainly due to the capillary suction force, the clock has not been paused during the measurement. The weight change of specimens was monitored and recorded with respect to immersion time. After 8 h of immersion, the specimen was half-broken to observe the depth of water penetration. The depth was examined and reported to the nearest millimeter.

Another mortar cylinder was for carbonation depth test. The specimen was split, and the carbonation depth can be observed by spraying 1% phenolphthalein solution on the fresh broken surface. The depth was measured and reported to the nearest millimeter. It would be remarked that, after following the program in Table 2, there was still another cylinder further carbonated for another 28 days period. Eventually, the depth of carbonation was measured which reflexes the propagation of 56 days exposure in total.

The effect of Na-MFP curing on the development of compressive strength of BFSC mortar was also studied. The Na-MFP cured specimens (*CR series*) were crushed to investigate the compressive strength at 4 and 28 days. The compressive strength result was obtained from the average of three specimens. It is known that carbonation attack damages microstructure, and decreases the strength of BFS mixture. In this study, the compressive strength was also investigated on the mortars after carbon dioxide attack. The specimens were crushed at the carbonation periods of 14 and 28 days. Before testing, all carbonated specimens were sprinkled with tap water for 3 days. The mortar cubes were crushed in wet condition. It would be remarked that the size of cube specimens used for compressive strength test were rather small (20 × 20 × 20 mm). The reaction between hydration/carbonation products and Na-MFP takes place at the near surface area where the penetration of the compound and carbon dioxide would reach. If there is any negative influence of Na-MFP on the compressive strength, it would be pronounced on these small specimens. Moreover, the effect of carbonation attack on the compressive strength would be also obvious.

The microstructure of mortars was studied by using environmental scanning electron microscope (ESEM): *Philips XL30*. The cylindrical specimens were vertically cut to a thickness of about 10 mm by a machine saw and dried in the oven at 35 °C until no further significant weight change. Low viscosity epoxy was used to maintain the integrity of the samples in the photomicrographs. A DBT Diamond Roller and Grinder/86 thin sectioning unit were used for preparing the perfectly smoother ESEM specimens. Eventually, the specimens were polished with 6 m, 3 m, 1 m and 0.25 m diamond paste, respectively, on a lap wheel. Thereafter, the prepared specimens were oven-dried at 35 °C for a period of 3 days.

3. Results and discussions

Fig. 1a and b presents the conditions of mortars after four cycles of frost scaling investigated by *Method A* for the *CR-series* and *ST-series*, respectively. Before testing, two reference lines were drawn on each specimen. The first line was on the edge of sample, and another line was drawn 5 mm away from the exposed surface. The area between two lines represented the section of specimen which was immersed into the salt solution during testing.

It was clearly seen that the control mortar were severely attacked. The surface of specimen was completely disintegrated. However, for the mortars with Na-MFP application, the specimens show excellent resistant against the frost salt attack. After four cycles, there was no visible scaling on *CR-20*, *CR-30* and *ST-30*. The edge scaling with some local pop-outs can be observed on *CR-10*, *ST-10* and *ST-20*. However, the damage on *CR-10* was less severe compared to those *ST-specimens*. In comparison, the specimens from *CR-series* perform better frost scaling durability than those *ST-series*.

Fig. 2 presents the results of scaling weight due to frost salt attack per unit surface area of specimen from *Method A* testing. The scaling weights were measured after 1, 2 and 4 freezing–thawing cycles. The results indicated that both Na-MFP curing and Na-MFP surface treatment methods reduced the scaling significantly.

Table 1
Properties of BFSC used in the experiment.

| Chemical | (wt.%) | Physical | |
|--------------------------------|--------|------------------------------|------------------------|
| | | Strength of standard mortars | |
| CaO | 45.00 | 2 days | 12.1 N/mm ² |
| SiO ₂ | 27.60 | 7 days | 32.7 N/mm ² |
| Al ₂ O ₃ | 12.20 | 28 days | 53.2 N/mm ² |
| Fe ₂ O ₃ | 1.27 | | |
| SO ₃ | 3.25 | Fineness | 376 m ² /kg |

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