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Quantitative evaluation of crack self-healing in cement-based materials by absorption test

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HIGHLIGHTS

- Water absorption test was used to evaluate the self-healing performance of cement-based materials.
- Various types of cement based materials tested.
- Self-healing mechanism by further hydration and calcium ion diffusion was examined.

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ABSTRACT

This study investigates the self-healing performance of cement-based materials using the water absorption test. The experimental method was performed in compliance with ASTM C 1585 on paste specimens mixed with research cement (RC), ground granulated blast-furnace slag (GGBS), anhydrite, and calcium sulfo-aluminate (CSA) expansion agents as inorganic binders. The water flow test was also performed for comparison and the self-healing products were analyzed. The results showed that the proposed method using the water absorption test could effectively evaluate the self-healing performance and that the mixture with GGBS and CSA expansion agents displayed the best performance. The temporal variation in water absorption rates was used to analyze the effects of primary self-healing (due to the further hydration of unreacted materials) and secondary self-healing (due to ion diffusion). The analysis provided results similar to those of the water absorption test. A comparison with an experiment on self-healing products confirmed that the amount of generated calcite had a larger effect on self-healing than the total amount of self-healing products.

1. Introduction

Cracks in concrete structures are commonly due to shrinkage, heat of hydration, and external loads. Such cracks allow the passage of harmful ions like chlorine ions and sulfate, which cause rebar corrosion and cover separation, thereby degrading durability [1–4]. Therefore, crack-repair in concrete structures is crucial for increasing the service life of the structure and reducing maintenance costs. Self-healing techniques in which concrete structures are healed without external intervention have received growing interest recently in response to the exponential increase of the maintenance costs of aging concrete structures [5–23].

Autogenous healing is a unique characteristic of cement-based materials and has been addressed by many studies [12–17]. This autogenous healing is known to have less self-healing potential in comparison with autonomous healing using capsules, bacteria, etc. In recent studies, supplementary cementing materials (SCMs), crystalline admixture (CA), expansion agents, and swelling agents were utilized to enhance the autogenous healing properties of cement-based materials [18–23]. Tittleboom [18] performed water flow and isothermal calorimetry tests to evaluate the effects of a mixture of ground granulated blast-furnace slag (GGBS) and fly ash on autogenous healing. This study found that the GGBS and fly ash mixture improved the autogenous healing performance and that a higher water-to-binder ratio (W/B) decreased the self-healing efficiency. Tittleboom noticed that the $CaCO_3$ precipitation caused by further hydration on the cracked surface is the most important component of self-healing techniques. Zhang [19] evaluated the self-healing performance of engineered cementitious composite (ECC) according to the content of fly ash. The evaluation showed that ECC with fly ash increased the deformability compared with ECC with ordinary Portland cement (OPC). This larger deformability resulted in a larger number of cracks with

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reduced crack width, and in this way, improved the crack self-healing performance [19]. Termkhajornkit [20] investigated the effect of the addition of fly ash on the crack self-healing performance of cement-based materials with focus on the self-healing property after 28 days. The compressive strength, chloride ion diffusion coefficient, porosity, hydration, and hydrates were investigated with regard to the addition of fly ash. The results revealed that a higher content of fly ash increased the self-healing ability because fly ash increased the amount of un-hydrated clinker. Sahmaran [21] conducted a rapid chloride permeability test (RCPT) to evaluate the self-healing performance of ECC specimens with GGBS and fly ash. Although the specimens with fly ash had more unreacted materials, the specimens with GGBS improved self-healing performance [21]. Sisomphon [22] examined the self-healing potential of cement-based materials with calcium sulfoaluminate (CSA) and CA by performing a crack closing test using an optical microscope. A 10% replacement of CSA and 1.5% replacement of CA for the cement mass enabled the specimens to self-heal a 400-μm crack within 28 days. Sisomphon discovered that the most important factor in self-healing was the concentration of Ca^{2+} ions flowing out of the cracked surface. He also noticed that the admixture of CSA and CA increased the concentration of Ca^{2+} ions flowing out of the cracked surface, thus facilitating the precipitation of $CaCO_3$ and resulting in improved self-healing performance. Huang [23] created a model for predicting the self-healing performance resulting from further hydration of the cracked surface. The unreacted materials of a cracked surface in a paste specimen were evaluated using HYMOSTRUC3D, and the precipitation of a crack was predicted by thermodynamic modeling.

This literature review shows that self-healing concrete has been actively investigated in various studies. However, only a few studies attempted to quantitatively evaluate the self-healing performance. The water flow and crack closing tests are the most widely adopted evaluation methods [24–29]. In the water flow test, the water head difference between the upper and lower parts of a cracked specimen are utilized to generate water flow in the crack in accordance with the method proposed by RILEM TC 221. The water flow rate is then used as a measure of self-healing because the test allows the crack self-healing effects to be intuitively identified through the decrease in water flow over time. However, the self-healing properties of a crack depend greatly on its shape (i.e., its tortuosity) and the self-healing performance evaluation results may vary according to shape [30]. The crack closing test can identify the self-healing performance by measuring the changes in crack width caused by self-healing. The method is advantageous because it allows direct observation of the crack self-healing but may lead to results different from those provided by the water flow test because only crack width changes on the surface can be checked [31]. Therefore, some researchers applied other methods for evaluating the self-healing performance [32–36]. However, these attempts are limited to qualitative evaluation and it is still difficult to perform accurate evaluation according to the type of binder. Consequently, a new experimental method is required in order to overcome the limitations of previous methods and provide quantitative evaluation of the self-healing performance.

Accordingly, this study adopts an absorption experiment method to evaluate quantitatively the crack self-healing performance of cement-based materials. Similarly to ASTM C 1585, the absorption experiment measures the changes in the absorption rate for both uncracked and cracked specimens. A method is proposed to quantify the crack self-healing performance based on the difference in absorption rates between the two specimens [37]. The proposed method is verified on paste specimens fabricated by Research Cement (RC) and SCMs and the experimental results are compared with those of the water flow test. In addition, a quantitative analysis of self-healing products is carried out to examine the self-healing effects according to the type of self-healing products generated on the cracked surface. The analysis results are compared with the results of the self-healing performance evaluation obtained by the proposed method.

2. Experiment

2.1. Materials

In this study, RC, GGBS, anhydrite, and CSA expansion agent are used as raw materials. RC was fabricated to exclude the effects of SCMs (which are added during cement fabrication) on the self-healing property. RC was produced by intergrinding 96% Portland cement clinker and 4% gypsum, with a Blaine fineness of 3880 cm^2/g and a density of 3.19 g/cm^3. Fig. 1 shows the XRD patterns of the raw materials. The XRD pattern of RC in Fig. 1(a) shows that RC is composed of C_3S, C_2S, C_3A, C_4AF, and gypsum, and does not contain other mineral admixtures; according to the Rietveld method analysis, the amount for each component is 61.8 wt%, 10.6 wt%, 11.3 wt%, 8.6 wt%, and 7.7 wt% of RC, respectively.

The adopted GGBS provided by Korea steel industry presents a Blaine fineness of 4,280 cm^2/g and a density of 2.87 g/cm^3. The crystalline phase of GGBS includes anhydrite (2.9%) and quartz (2.1%). Anhydrite presents an SO_3 content of 56.9%, fineness of 4,050 cm^2/g and density of 2.89 g/cm^3.

The CSA expansion agent is composed of 53.7% of CaO 26.9% of SO_3. The fineness and density are 4,630 cm^2/g and 2.78 g/cm^3, respectively. As shown in Fig. 1(c), the CSA is in the crystalline phase with a mineral composition including mainly gypsum (48.6%), CSA (28.0%), and calcium hydroxide (16.6%). Small amounts of lime and C_3S are also included. Table 1 presents the chemical compositions of RC and GGBS used in this study.

Fig. 2 shows the particle size distribution (PSD) of the raw materials measured by laser diffraction. The mean diameters of RC, GGBS, and CSA are 11.8, 14.02, and 20.45 μm, respectively. RC and GGBS present similar particle sizes but CSA has a larger mean particle size. However, as shown in Fig. 2, RC has a wide range of particle sizes running from 0.16 to 228.85 μm, whereas GGBS shows a relatively narrow range from 1.9 to 58.96 μm.

2.2. Mixture proportions and test methods

The paste specimens were fabricated to evaluate the self-healing performance using a quantitative analysis of self-healing products with the artificial crack, water flow, and water absorption tests. Table 2 presents the adopted mixture proportions of the paste specimens.

2.2.1. Tests for quantitative analysis of self-healing products

For the quantitative analysis of the self-healing products precipitated on a cracked surface, the self-healing products must be separated from the original cement matrix. Normally, cracks in cement and concrete specimens are irregular, and the self-healing products on the cracked surfaces cannot be obtained in their original states. Therefore, this study adopted the "artificial crack method" proposed by Huang [38] to acquire the self-healing products. To that goal, 100 × 100 × 10 mm paste slice specimens were fabricated. After one day, the specimens were removed from the mold and cured in water at a temperature of 20 ± 1 °C. After 7 days, the specimens were dried and polished to create a uniform surface. The polished specimens were fixed using a compression band, and a constant space was maintained between the specimens using a 100-μm thick polyester film. Fig. 3 describes the experimental program of the self-healing products precipitation test.

The bottom of the specimen was separated by a 20-mm thick spacer, and tap water was poured up to the bottom surface of the specimen. The water level was constantly controlled. After 7 days of curing, the specimen was completely separated. A plastic putter was used to remove the self-healing product that was additionally created in the space between the artificially cracked surfaces of the specimen. For the XRD analysis, the product was dried at 40 °C for 2 days. To secure a sufficient number of samples for the analysis, a total of 32 specimens were fabricated with the same mixture proportion and size.

The XRD measurement was performed using a PANalytical X'Pert Pro MPD diffractometer with an attached X'Celerator detector. Scanning was performed from 10° to 60° 2θ, with a step size of 0.04° and 2 s counting time per step. The X'Pert HighScore Plus (PANalytical) software was used for the Rietveld quantitative phase analysis, which quantifies the crystalline and amorphous phases of the self-healing product. Corundum (Al_2O_3) 10% was added as an internal standard material.

2.2.2. Water flow test

In this study, the constant head water flow test device shown in Fig. 4(a) was used to evaluate the self-healing performance of the cracked specimens. Three circular paste specimens with the same mixture proportion and a size of ϕ100 × 50 mm were fabricated for the water flow test. The specimens were stored for one