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#### **Construction and Building Materials**

journal homepage: www.elsevier.com/locate/conbuildmat



## Self healing behavior for crack closing of expansive agent via granulation/film coating method



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#### HIGHLIGHTS

- Granulation method was used to control response time of expansive agent.
- Granulated expansive agent was coated with PVA film to control time of self healing.
- Complete crack closing was observed after immersion via microscopy investigation.
- Expansive agent via granulation/coating method can control time of autonomic healing.

#### ARTICLE INFO

# Article history: Received 6 June 2014 Received in revised form 1 August 2014 Accepted 23 August 2014 Available online 16 September 2014

Keywords: Autonomic healing CSA-based expansive agent Granulation PVA film coating Crack closing

#### ABSTRACT

Although there are many self-healing approaches, these approaches have merits and demerits. The utilization of expansive agents and mineral admixtures in various approaches may be appropriate due to their good healing efficiency, compatibility with the cement matrix, and low cost. In this study, after the granulation of the CSA (Calcium Sulpho Aluminate)-based expansive agent, it was coated with a PVA film to control the time of autonomic healing. It was verified through various tests that the granulated CSA-based expansive agent with PVA film coating can control the time of autonomic healing and can prevent water migration via crack closing.

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#### 1. Introduction

Self-healing is a common phenomenon in the human body; that is, injuries are automatically healed, even without any major operation. Self-healing is also known to occur in conventional concrete. The self-healing function in concrete usually results from chemical reactions such as further hydration and carbonation [1].

Self-healing in concrete is a combination of complicated chemical and physical processes. Based on most of the literatures studied, the phenomenon of self-healing is reported to involve the following processes: the formation of calcium carbonate and of loose concrete particles resulting from crack spalling, the further hydration of the unreacted cementitious materials, and the swelling of C–S–H. Of these, the primary mechanism is believed to be the crystallization of calcium carbonate [2,3].

To fully understand self-healing in concrete, the terminologies related to self-healing were classified in JCI Technical Committee TC-075B. Natural healing is a natural phenomenon involving the sealing of cracks resulting from some chemical reactions. Autonomic healing is the healing of cracks by admixtures. Consequently, admixtures such as fly ash and expansive agents are intentionally incorporated into concrete in advance. Finally, activated repair involves automatic repair using some artificial devices, which usually consist of sensors and actuators [4].

Various self-healing methods have been suggested until recently, as shown in Table 1, but these methods have merits and demerits. The utilization of expansive agents and mineral admixtures in various methods may be appropriate due to their good healing efficiency, compatibility with the cement matrix, and low cost, but the efficiency of healing products generated by necessity is not guaranteed [5–7].

Powder can be fabricated into a solid preparation to control the medicine release time inside the body, through the methods used in the pharmaceutical field. Tablets, granules, microcapsules, etc. are typical. The tablets and microcapsules in these methods are

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**Table 1** Self-healing methods.

Terms on healing	Methods
Natural healing	<ul><li> Hydration of unreacted cement</li><li> Carbonation/precipitation of reaction products</li></ul>
Autonomic healing	<ul> <li>Mineral admixtures such as fly ash, slag, and slica fume</li> <li>Expansive agent, geo-materials</li> </ul>
Activated repair	<ul><li> Microcapsules, hollow fibers</li><li> Shape memory materials, bacteria</li><li> Heat evolution from devices, monitoring techniques</li></ul>

inferior to granules due to their low effects and complex processes [8]. The granulated powder materials may be gradually released depending on the environmental condition and elapsed time. In addition, they can be coated with a water-soluble film. Coating, which is done to protect the contents, to improve the exterior, and to control the medicine release time, among others, is mainly used [9].

Therefore, if granulation/coating methods are applied to expansive agents or mineral admixtures, the release time of healing materials can be controlled until cracks occur, and healing products will be formed because healing materials react with moisture via the crack faces. Release time control can be divided into two terms: control of granule hardness and control of the surface coating thickness by a water-soluble film.

In this study, the CSA-based expansive agent was granulated and was then coated with a water-soluble polymer substance. A length change test was conducted to determine the proper coating thickness. Also, the autonomic healing efficiency was evaluated via microscopy investigation, measurement of the relative dynamic modulus of elasticity, and the water passing test.

#### 2. Experimental procedure

#### 2.1. Granulated expansive agent with film coating

Granulation is the process of gathering particles together by creating bonds between them. Bonds are formed by compression or by using a binding agent. One of the traditional wet granulation methods is forming granules through a process involving wetted powder or a powder mixture pass through a predetermined. One of the other wet methods is fluid bed granulation, a process that involves suspending particles in an air stream and spraying a liquid from the top of the system down onto the fluidized bed sieve. The particles in the path of the spray get slightly wet and become sticky sieve [10,11]. The main coating methods are pan-coating, fluidized-bed coating, press-coating, and others. Of these, the pan-coating method shown in Fig. 1 is the most common [12].

The CSA-based expansive agent consists of hauyne (3CaO·3Al $_2$ O $_3$ ·CaSO $_4$ ), free lime (CaO), and anhydrite (CaSO $_4$ ). They mainly provide ettringite (3CaO, Al $_2$ O $_3$ , 3CaSO $_4$ , 32H $_2$ O), which is a micrometer-sized acicular crystal, due to hydration. In particular, ettringite fills the micropores of concrete and is induced by the expansion of concrete [13].

In this study, the surface of the granulated CSA-based expansive agent (granule) was coated with a water-soluble polymer substance, which used polyvinyl alcohol (PVA). PVA as a coating material can withstand the friction between the aggregates and the mixer blade in early mixing due to its stiffness. The water solubility can be adjusted to any of multiple grades (degree of deacetylation) [14,15].

The pan-coating method was used in this study. A blending solution of PVA and purified water was sprayed onto the pan-inside granule, which simultaneously blew hot air. The surface of the granule was then bonded with PVA through the evaporation of purified water. By repeating this process, the coating thickness was increased. The PVA coating was 0.012–0.0073 mm thick when the process was repeated at least twice.

The material volume theory was used as the basis of the calculation of the coating thickness in this study because it is hard to measure the thickness of the PVA coating. Therefore, the thickness was measured using the following equation, as suggested by Zhao et al. [16]:

$$t = \frac{m}{S_0} \times \rho_p \tag{1}$$

where t is the calculated thickness of the PVA coating according to the above equation, m is the mass of the PVA coating,  $S_0$  is the specific surface area of the granule, and  $\rho_p$  is the density of the PVA coating. And  $S_0 = S'/V$ , S' is the surface area of the granule, V is the volume of granule.

In this study, CSA-based expansive agent was granulated through a process involving kneading, wet granulation, and drying. The granules were fabricated using the granulator machine. The granules were cylindrical in shape, with 0.5 mm diameters and 0.5 mm lengths. The features of the granule and coating are shown in Table 2. Fig. 2 shows a close-up image of the granule.

#### 2.2. Preparation of specimens

Mortar specimens ( $40 \times 40 \times 160$  mm prism) were used for all the analyses in this study. Mortar mixtures for length change test were designed based on W/C=0.5 and with a cement-to-sand ratio of 1:3. As can be seen in Fig. 3, specimens were not sealed. To determine the appropriate coating thickness for the control of the release time of the granules with PVA coating, the length change test was conducted according to KS F 2562. Fig. 3 indicates a view of the length change test after strain gages are attached to specimens.

Mortar mixtures for autonomic healing efficiency were designed based on W/C = 0.5 and with a cement-to-sand ratio of 1:3, according to ISO 679. Also, the replacement of the granules with PVA coating was limited to 10% by mass cement because some researches noted the occurrence of unexpected expansion [6].

All the mortar mixtures were placed into the  $40\times40\times160$  mm moulds in different layers. First, a 10 mm mortar layer was brought into moulds and was compacted via vibration. Two  $\Phi2$  mm reinforcement bars were placed onto this layer. Afterwards, the moulds were further filled with mortar. After casting, mortar prisms were cured at  $20\pm2$  °C and in a  $95\pm5\%$  humidity chamber until the testing time. At the age of 28 days, cracks (range: 0.01-0.35 mm) were created in the mortar prisms by means of a three-point bending test. Then the crack widths and images were obtained via microscopy investigation. Table 3 indicates detail of the experimental procedure.

#### 2.3. Evaluation of self-healing efficiency

The parameters for evaluating self-healing efficiency are divided into two groups, one concerned with mechanical properties such as strength or stiffness, and the other related to a transport substance such as ion or water. The improvement of self-healing is expected to be more pronounced in the latter than in the former [4,17]. As such, in this study, self-healing efficiency was mainly verified with regard to water migration rather than the mechanical properties. All the specimens were immersed in water after the introduction of cracks. Afterwards, the crack widths and images were obtained via microscopy investigation depending on the elapsed time. At the same time, a water passing test was conducted, and the dynamic modulus of elasticity was measured.

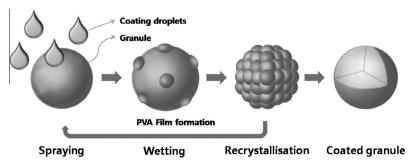


Fig. 1. Pan-coating method.

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