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A Review of Expansive Agents for Autogenous Self-Healing

Jule Lushomo[1], Huang Zhuye[1*], and Fan Peiran[2]

[1]School of Civil Engineering and Architecture, Zhejiang University of Science and Technology, Hangzhou, China

[2]China United Engineering Corporation Limited, Hangzhou, China

[*]Corresponding author: Zhuye H, School of Civil Engineering and Architecture, Zhejiang University of Science and Technology, Hangzhou, China; E-mail: huangzhuye[At]zust.edu[DOT]cn

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Abstract

Self-healing is a concept that has been studied for decades. Self-healing is the ability of cementitious composites to reduce the width of cracks generated due to wear and tear of the material. Of the many self-healing mechanisms, this article reviews and outlines the progress of self-healing in past and recent decades. Primarily, the use of expansive agents for self-healing is discussed in this article. When a cementitious composite fills up the crack with hydration products, expansive or crystalline materials, the composite is said to have undergone autogenous self-healing. Autonomous self-healing refers to the intentional addition of materials to help the composite self-heal. This article delves into these two types of self-healing. At its core, this article studies research on different expansive materials used in self-healing for cementitious composites. The use of magnesium oxide (MgO), quicklime, calcium sulpho-aluminate (Type K), and Sodium Bentonite expansive materials were investigated. Combinations of expansive agents with geomaterials, crystalline additives, swelling minerals in varying proportions were also outlined. Their strength regain, crack sealing and optimum self-healing is subsequently discussed. The use of sand replacing pellets infused with expansive materials for self-healing is studied. Some of the results showed that although self-healing took place, the re-hydration products were unstable proving that a design that had a greater chemical and physical stability is required for effective self-healing. When combinations of expansive agents are used, results show that the composites have a higher crack sealing nature with most cracks being sealed in 14 days. With a new method to improve self-healing, a study was introduced that partially replaced fine aggregates with active granules. The results showed improved crack mouth healing.

Keywords: ***Autogenous; Autonomous; Crack; Expansion agent; and Self-healing.***

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1. Introduction

Like the human body, cement-based composites have the mechanism of self-healing. In general, the term self-healing, particularly in cementitious composites, refers to the reduction of cracks and the repair of damage in cement-based materials. Hyde and Smith [1], [2] studied this self-healing phenomenon at the end of the nineteenth century. Glanville [3] gave a more systematic analysis of healing phenomena, dating back to 1926. At this time, there was already a distinction made between self-healing and self-sealing.

Azarsa [4] presented a study to experimentally investigate self-healing and improvement of chloride and water permeability in concrete with the use of the of crystalline admixtures (CA). The results showed that the specimen with CA had a lower penetration depth. Different exposure conditions were used to investigate the effectiveness of self-healing in Engineered Cementitious Composites (ECC) [5]. The study showed the ECC with 10% Limestone powder replacement had the best tensile strain capacity. The use of epoxy filled microcapsules for autonomous self-healing was studied [6]. The study concluded that an addition of 5% capsules increased the self-healing and impermeability characteristics of the cementitious material. Park [7] studied the quantitative self-healing performance of cement-based materials incorporating expansive materials on research cement. The study revealed that in the specimens made with the calcium-based expansion agent underwent compete crack healing as the absorption rate between reduced to zero after two days of self-healing. This paper looks at the phenomenon of self-healing, the mechanisms responsible for self-healing as well as the two main classifications of self-healing. Expansive agents and their use in self-healing-based research is the focus of this article.

2. Mechanism of Self-Healing

In the recent decades, self-healing has piqued interest of many researchers. Self-healing leads to partial or complete crack-closing, which in turn improves the permeability, durability, and potentially mechanical properties.
This is especially important for watertight structures. It also prolongs the service life of infrastructure [8].
Several possible causes as shown in Fig 1. can be responsible for the self-healing phenomenon and that are as follows.

- Further hydration of the unreacted cement or cementitious materials.
- Formation of calcium carbonate or calcium hydroxide.
- Blocking cracks by impurities in the water and loose concrete particles resulting from crack spalling.
- Expansion of the hydrated cementitious matrix in the crack flanks.

The crystallization of calcium carbonate is believed to be the primary mechanism of self-healing [9]-[14]. This proposition is supported by the fact that precipitated calcium carbonate can often be observed at the outside surfaces of the self-healed crack as some white residue. According to the early work by Neville [15], self-healing is claimed to be caused mainly by further hydration of unhydrated cementitious components. However, later it was discovered that this only applies to very young concrete and it was concluded that the formation of calcium carbonate is the most likely cause of self-healing at later ages [16].

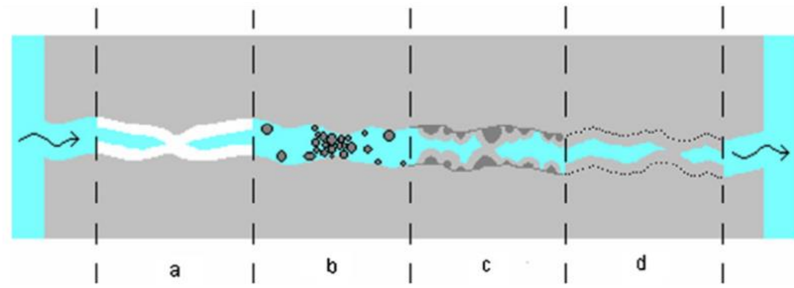

Fig. 1. Possible self-healing mechanisms (a) Formation of calcium carbonate or calcium hydroxide. (b) Sedimentation of particles. (c) Continued hydration. (d) Swelling of the cement-matrix [9].

3. Autogenous and Autonomous Self-Healing

Cement based materials have a characteristic trait: they fill the cracks with hydrated binders, expansive or crystalline products, given the proper conditions. This is known as "autogenous self-healing". Cement based materials are capable of sealing and healing the cracks without external activation, this persuaded researchers to develop "Autonomous self-healing".

Autonomous self-healing is the purposeful designing and addition of engineered materials into cement-based materials to "heal" itself in the event of cracking. They aimed at tailoring novel mechanisms that expected to be more efficient than autogenous self-healing. However, the initial cost for these techniques rises a significant concern for its wide-spread usage. To overcome this, researchers came up with a self-healing mechanism called "Improved autogenous self-healing" which follows the incorporates the mechanism of autonomous self-healing, i.e. engineered conditions, yet the entirety of its function and healing products are identical to autogenous self-healing [16].

3.1 Autogenous self-healing

The effects of autogenous self-healing have been investigated for many years. However, the reaction products of autogenous self-healing detected by various researchers are not consistent. Researchers found calcium-silicate hydrate gel (C-S-H), newly formed, in the cracks [18], [19], whereas calcium carbonate ($CaCO_3$) was found in and around the crack mouths after autogenous self-healing [12], [20]-[22]. The main mechanisms of autogenous self-healing are therefore assumed to be [23]:

- further hydration of unhydrated cement;
- formation of calcite (calcium carbonate) and portlandite (calcium hydroxide) caused by leaching from the bulk cement paste.

3.1.1 Hydration of unhydrated cement

Approximately 20–30% of cement in conventional cement composites remains unhydrated. When the concrete cracks, the unhydrated cement particles react with the ingress water. This starts hydration process again and makes hydration products fill the cracks [24,25]. Water is absorbed into the hydrated cement paste. This causes swelling of the cement matrix, which is in proximity of crack tip. More precisely, the expansion and swelling of C-S-H gel. The continuing hydration of unreacted cement particles due to lack of water during hydration process which in turn occupies crack voids. This healing mechanism is more pronounced in young concrete [26]. As the cement hydration products dissolve in water, calcium hydroxide is liberated and dissipated along the cracking surfaces. Then free calcium ions from cement hydration react with dissolved carbon dioxide, so "self-healed" crystals are formed, growing at both surfaces of the cracks and finally filling into the gaps [23], [24]. The self-healing due to the hydration of the expansive admixtures present in the cement paste is discussed in detail in section 4.

3.1.2 Formation of calcite and portlandite

Compared to other all-natural healing mechanisms, the formation of Portlandite ($Ca(OH)_2$) and Calcite ($CaCO_3$) is the most effective. Portlandite is formed as a hydration product in the cement, whereas calcite is formed as a result of carbonate reaction with free calcium ions. At later stages, the formation of calcite is the primary cause of autogenous self-healing [10]. Usually there are very few carbonate (CO_3^{2-}) ions existing in the bulk paste. In this case, calcite is hardly formed. However, CO_3^{2-}ions in cracks can come from the outside environment, although their diffusion into water is quite slow. When $CO2$ in the air dissolves in water, CO_3^{2-}ions diffuse into cracks through the crack mouth. A gradient of CO_3^{2-}ion concentration is formed within the cracks. The concentration of CO_3^{2-}ions at and around the crack mouth is more than inside the crack. Because of this, calcite is first around the crack mouth. More and more CO_3^{2-}ions reach the locations inside the cracks as self-healing continues. The portlandite that has been formed in the cracks will be gradually carbonated, leading to more and more calcite formation. The reaction products of autogenous self-healing studied by various researchers are inconsistent because of the reaction products formed in cracks dispersing heterogeneously and their components vary with time. [9], [23], [24]. Fig 2. And equations 1, 2, and 3 [9] show the formation of CaCO3.

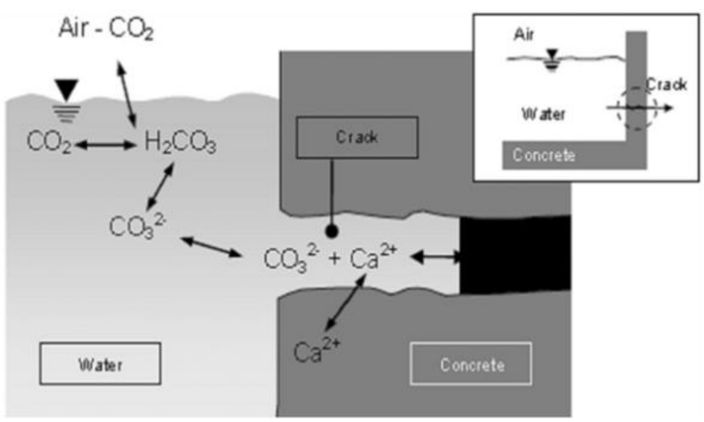

Fig. 2. Precipitation of calcium carbonate in the presence of water and dissolved CO_2 [23].

$$H_2O + CO_2 \Leftrightarrow H_2CO_3 \Leftrightarrow H^+ + HCO_3^- \Leftrightarrow 2H^+ + CO_3^{2-} \quad (1)$$

$$Ca^{2+} + CO_3^{2-} \Leftrightarrow CaCO_3 \ \ (pH_{water} > 8) \quad (2)$$

$$Ca^{2+} + HCO_3^- \Leftrightarrow CaCO_3 + H^+ \ \ (7.5 < pH_{water} < 8) \quad (3)$$

3.2 Autonomous Self-Healing

Autonomic healing is an artificial healing process. A healing agent is added to concrete, so that blocking, or "repair" of the crack can be done without any external aid. This type of healing can be achieved by chemical and biological processes. This healing follows a deliberately accelerated artificial route in material damage repair: triggering actions (actuation), transport of healing products into fractured zone and finally chemical repair and curing. Damage in a material is indeed necessary to trigger the healing process [25] [26].

This self-healing process of concrete can loosely be divided into the following main categories: self-healing by bacteria [27], [28], microcapsules [29], [30], and hollow fibers and shape memory materials [31], [32]. Bacteria, healing agents and chemical compounds can be put inside a protective casing to avoid premature reaction during the mixing of cement components. The mechanism or encapsulation is shown in Fig. 3.

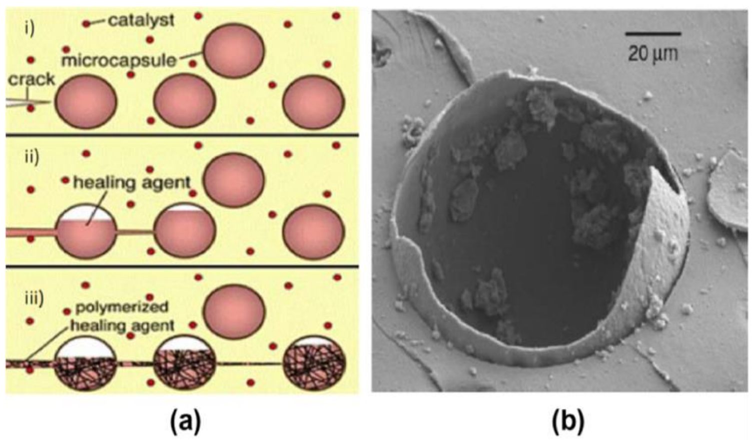

Fig 3. (a) Basic method of the microcapsule approach: (i) cracks form in the matrix; (ii) the crack ruptures the microcapsules, releasing the healing agent into the crack plane through capillary action; (iii) the healing agent contacts the catalyst, triggering polymerization thus ensuring the closure of the near-by cracks and (b) ESEM image showing a ruptured microcapsule [9].

4. Expansion Agents

Expansive agents are admixtures that increase the apparent volume of concrete, caused by specific controlled internal chemical reactions in the early stage of hardening. Shrinkage Compensating Concrete (SCC) is the term given to concrete containing an expansive agent. This concrete is used when it is necessary to reduce the number of cracks due to drying and/or autogenous shrinkage [33, [42]. The main practical application of expansive agents deals with the construction of concrete slabs on ground, large walls, and concrete repairs. Of the many types of expansive agents, the most important are based on the formation of ettringite, AFt, $(C_3A \cdot 3C\bar{S} \cdot H_{32})$ or calcium hydroxide (CH):

$$C_4A_3\bar{S} + 6C + 8CS + 96H \Longrightarrow 3C_3A \cdot 3C\bar{S} \cdot H_{32} \quad (4)$$

$$C + H = CH \quad (5)$$

Reaction (4) is based on the transformation of a mixture of sulfo-aluminate ($C_4A_3\bar{S}$), lime (C) and anhydrite ($C\bar{S}$) into ettringite, and reaction (5) on the transformation of lime into calcium hydroxide [33], [34]. The classification of expansion mechanisms is according to the chemical nature of the minerals that are responsible for the formation of these expansive hydrates [36]. The expansive agent of Type K (calcium sulpho-aluminate) results in the formation of ettringite whereas expansive agents of Type G are based on dead-burn lime, result in the formation of calcium hydroxide (portlandite) [35], [36], [43].

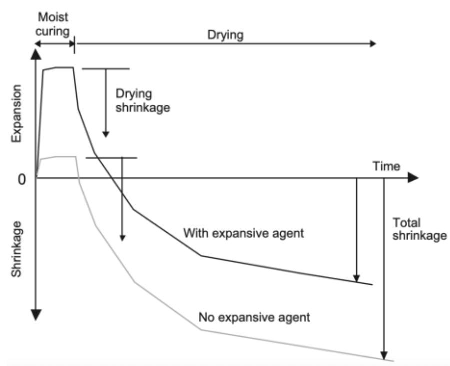

Fig. 4. Shrinkage and expansion verses time graph for concrete with and without expansive agents [35].

Fig. 4 compares two types of concrete, one with expansive mixture and one without. During the moist curing stage, the concrete without expansive admixture undergoes some expansion, most likely caused by the hydration of free lime present in the clinker. The concrete with expansive admixtures undergoes swelling much larger than the concrete without the admixture. The maximum swelling is between 2 to 7 days, depending on the type of expansive agent used. As soon as the water curing stage is finished, drying shrinkage starts. The type of expansive agent used doesn't usually, influence the extent of drying shrinkage, which is seen in the similar dimension change in both types of concrete [33]. Drying shrinkage generates internal tensile stresses that may result in the cracking of concrete. As shown in Fig. 4 the expansive agent reduces the total shrinkage, which reduces the risk of cracking in concrete [35].

Shrinkage reduction efficiency on alkali-activated coal gangue-slag (AACGS) cementitious material was investigated by H. Ma et al. [37]. Polypropylene fiber (PPF), shrinkage-reducing admixtures (SRA), sulfate-enriched materials (gypsum, high-performance concrete expansive admixture (HCSA), U-type expansive admixture (UEA)) and water retaining admixture (WRA) were analysed in order to reduce dry shrinkage. The results showed that although all the admixtures in this study reduced the drying shrinkage of AACGS mortar, the sample mixed with UEA had the best shrinkage reduction effect.

Q. Yuan et al [38] also studied the effect of the addition of UEA and aluminum powder (AP) on Cement emulsified asphalt mortar (CA mortar). The study revealed that the addition of UEA not only compensates shrinkage (in the first seven days) but also provides a reduced but more stable expansion. It should be noted that although the UEA shrinkage compensation lasted longer than AP, the joint use of both these agents works better for shrinkage compensation in CA mortar. The study was carried out to test the effect of UEA and MgO on the fracture properties of concrete [39]. Different ratios of UEA to MgO agents were tested, i.e., 4:1, 2:1, 1:1, 1:0. The study concluded that when the mass ratio was 2:1, the concrete had better fracture properties.

5. Expansive Agents in Self-Healing

Ahn [40] investigated the self-healing of cementitious composites with expansive agents, geo-materials and chemical agents. Three types of concrete were studied: plain concrete, expansive concrete and self-healing concrete. The expansive agent used in the expansive concrete was Type K. To study the early age behavior of the concrete, a temperature-stress-modulus test (TSTM) was conducted and the results showed the development of expansive concrete was faster and greater than that of the other 2 types of concrete.

To further investigate the efficiency of self-healing, 3 samples were studied. The first sample with expansive and geomaterials, only, underwent self-healing however the self-healing speed for rapid waterproofing for water leakage needed to be improved. The second sample included chemical additives to combat this shortcoming. The results showed that although self-healing took place, the re-hydration products were unstable. A third sample was designed that had a greater chemical and physical stability.

Qureshi et al. [41] studied the self-healing of efficiency of three different expansive minerals, magnesium oxide (MgO), bentonite and quicklime under continuous water immersion in cement paste. In regards to the compressive strength, the study showed that substitution of 5% bentonite and quicklime reduced the compressive strength of the cement paste whereas 5% MgO had no impact on compressive strength at 28days. The specimen with more expansive admixtures sealed showed higher sealing nature with most cracks sealed within 14 days as seen in Fig. 5. The strength recovery was calculated by equation (6). The crack sealing of individual expansive agents, as well as combinations of these expansive agents are shown in Fig. 6. The results showed that the most efficient strength recovery ranged between 5% cement replacement of each mineral mixes (Portland Cement 85%, MgO 5% Bentonite 5% and Quicklime 5%) and 5% of both MgO and bentonite mixes (PC90M5B5).

$$SR(\%) = \frac{rec,\sigma_{f,max}}{ini,\sigma_{f,max}} \times 100 \qquad (6)$$

Where, SR is the percentage of flexural strength recovery; $rec, \sigma_{f,max}$ is the maximum flexural strength recovered at 14 and 28 days (the testing time) and $ini, \sigma_{f,max}$ is the maximum flexural strength in the cracking of 1 day sample.

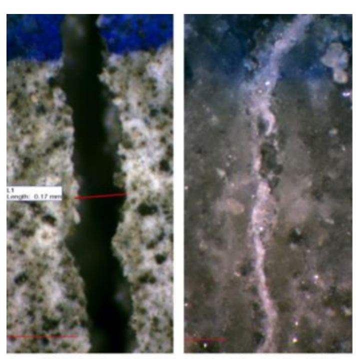

Fig. 5. Crack sealing in 28 days of cement with expansive minerals [41].

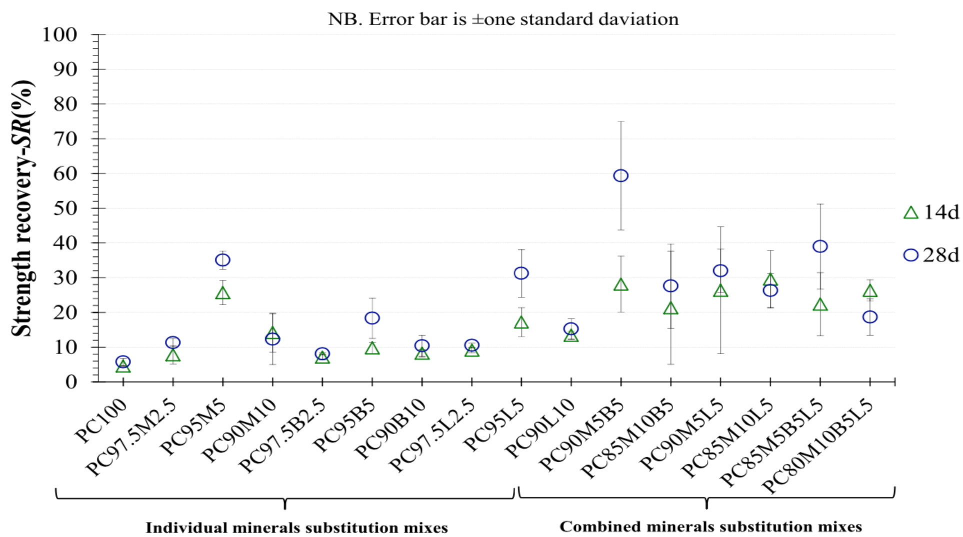

Fig. 6. Comparison of crack sealing efficiency (x-axis shows percentages of the expansive mixtures in the cement paste. PC is Portland Cement, M is MgO, B is Bentonite, L is quicklime. PC97.5M2.5 is 97.5% Portland cement and 2.5% MgO, PC97.5B5 is 97.5% Portland cement and 5% Bentonite, PC97.5L5 is 97.5% Portland cement and 2.5% Lime) [41].

In another study, porous lightweight aggregates carrying sodium carbonate solution were used to induce self-healing of cementitious composites containing CSA based expansive agents, crystalline admixture (CA), and calcium hydrogen phosphate $(CaHPO_4)_2$ [44]. The study concluded that the specimens with crack width of 0mm-50mm had the most crack healing efficincy combination of the porous aggregate in combination with the self-healing efficiency was greatly affected by the proportions of the additives. The specimen with the proportions 7% CSA, 4.5% CA, 1% CaHPO, and 12% sodium carbonate, by mass of binder, the exhibited the highest self-healing efficiency.

Similarly, Jiang [45] tested the effect of silica-based materials, chemical expansive agents, swelling minerals and crystalline components on self-healing in still and flowing water. When only a single material was used in combination with the cementitious component, the specimen with expansive agent performed best. In regard to the samples with a combination of these minerals, the specimen with silica-based, swelling and crystalline minerals had the most self-healing efficiency. The results indicated that a still water, high pH and temperature accelerate the self-healing of cementitious composites.

Alghamri [46] conducted research to replace part of the fine aggregate with active granules for self-healing. The active granules (pellets) encapsulated MgO, bentonite clay (in the ratio 1:1) and silica and MgO expansive materials (in the ration1:1). The size of the pellets was between 0.6 mm-4 mm. The pellets replaced 10% of the fine aggregate. The results showed that the healing efficiency of the composites was affected by the size of the pellets used as shown in Fig. 7. There was a relation between the pellet size, crushing strength of the pellets, the strength regain and the crack sealing. With an increase in pellet size, the pellets the with lower crushing strength had a higher crack sealing effect but a lower strength regain, whereas the pellets with an increased size and crushing strength and a higher strength regain but a lower crack sealing efficiency. Fig. 8 shows that for pellets of the same size (1-2 mm) the addition of pellets improved the crack mouth healing (CMH) of the composites.

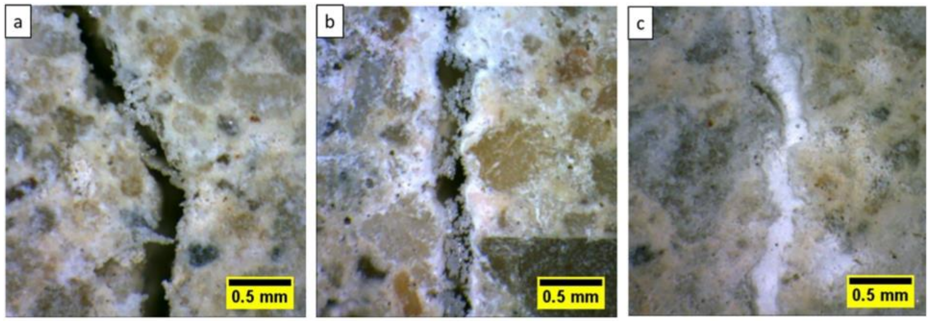

Fig. 7. Crack sealing at the mouth (a) control specimen (b) Pellet size 1-2mm with MgO and bentonite (c) Pellet size 2-4mm with MgO and bentonite [46].

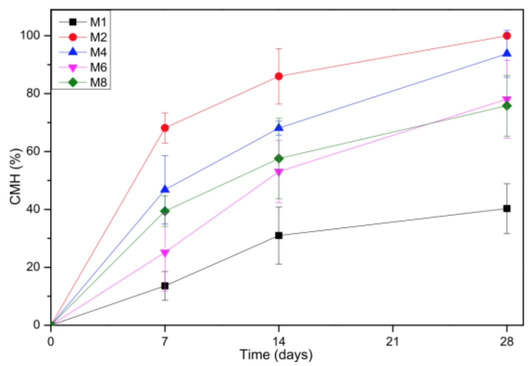

Fig. 8. Graph of crack mouth healing (CMH) over time. (M1 is the control specimen, M2, M4, M6, and M8 are the sample specimen embedded with pellets) [46].

Rajasegar [47] investigated the effect of expansive agent sodium bentonite, rice husk ash, nano silica and poly vinyl alcohol (PVA) on the self-healing of concrete. 10%, of rice husk ash, 1% Nano Silica, 6% Sodium Bentonite were added to the concrete with varying proportions of PVA (0%, 0.5%, 1%, 1.5%, 2%). The study concluded that there was a noticeable crack width closure in the sample with 1.5% and 2% PVA and was recommended as the dosage need for use in self-healing concrete.

5. Conclusion

This article reviews the role of expansive mineral additives in self-healing phenomenon of cementitious composites. Autogenous self-healing can be attributed to reactions of mineral admixtures in cementitious materials as well as the hydration of unreacted cement particles. Whereas autogenous self-healing, is the deliberate addition of materials into the cement composite to aid in self repair. Both these methods usually have the precipitation of mineral composites that are attributed to self-healing. The addition of expansive agents into cementitious composites for self-healing has been discussed in this article. The results showed that although self-healing took place, the re-hydration products were unstable and another mix design was formulated that had a greater chemical and physical stability. In other research, results indicated that the most efficient strength recovery ranged between 5% cement replacement of each expansive mineral mix. Alternatively, still water for curing, high pH and temperature accelerated the self-healing of cementitious composites.

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Jule Lushomo received the bachelor's degree in civil engineering from Xinyu University, China. She is currently working toward the post graduate in civil engineering from the School of Civil Engineering and Architecture, Zhejiang University of Science and Technology, Hangzhou, China.

Zhuye Huang received the master's degree in civil engineering from Zhejiang University, China. She is currently an associate professor in civil engineering from the School of Civil Engineering and Architecture, Zhejiang University of Science and Technology, Hangzhou, China.

Fan Peiran received the graduation from the Zhejiang College of Construction. He is currently working as an Engineering Assistant in building information modeling with China United Engineering Cooperation Limited, Hangzhou, China.

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