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## Encapsulation of expansive powder minerals within a concentric glass capsule system for self-healing concrete



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#### HIGHLIGHTS

- Dry expansive minerals encapsulated in concentric glass capsule for self-healing.
- Dispersion and diffusion of healing agent from capsules triggered healing mechanisms.
- Crack bridging occurred by effective formation of healing compounds in mortar matrix.
- Very large cracks ( $\sim$ 400  $\mu$ m) found to be effectively healed with capsule system.

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#### ABSTRACT

This study presents the application of encapsulated expansive powder minerals (magnesium oxide, bentonite and quicklime) for self-healing of cement-based mortars. A system of concentric glass macrocapsules was used to envelope the expansive minerals (outer capsule) and water (inner capsule). Mortar samples containing concentric macrocapsules with different mineral combinations were cracked and healed under three different curing regimes; ambient conditions, high humidity exposure and immersed in water. Self-healing was assessed based on visual crack sealing, mechanical strength recovery and improvement in durability investigated by means of capillary sorption tests. Micro-structural analysis of the healing materials was investigated using FT-IR, XRD and SEM-EDX for exploring self-healing kinetics. Immersed in water have yielded the optimum healing efficiency with  $\sim\!95\%$  crack sealing and  $\sim\!25\%$  strength recovery in 28 days. Data showed an increasing trend in 56 days for both crack sealing and load recovery. The improvement in terms of capillary absorption of healed samples was also significant after 28 days of healing. Self-healing kinetics revealed that the expansive minerals were hydrated in the initial healing period and slowly carbonated over time until the peripheral crack zone became adequately water tight.

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#### 1. Introduction

Self-healing in concrete can be broadly classified into two categories: autogenic and autonomic healing [1]. Autogenic healing is an intrinsic material healing property which is triggered with the hydration of unhydrated cement remaining in the matrix. Early age concrete can heal naturally due to continued hydration of un-hydrated cement particles existing in the matrix. In addition, healing is also promoted by precipitation of carbonates and crystal growth especially near the crack tips. Autonomic healing on the other hand is the use of components which normally are not present in cement based composites. This category typically refers to different types of materials incorporated into the matrix usually

Single-component, air-curing healing agents, such as silicon based adhesive, cyanoacrylates, epoxy and alkali-silica solutions are preferred by different researchers [6–9]. Incorporation of these single component agents inside concrete systems is rather simple

in the form of encapsulated additions. Self-healing is triggered upon crack formation which results in the rupture of the encapsu-

lated system and the subsequent release of the healing com-

pounds. The autonomic concept has been developed to tackle the

limitations of autogenous healing [2–4]. These include dependence

on the age of the concrete, as older concrete structures have less

unhydrated cement particles, and the lack of healing larger cracks,

as autogenic processes can typically heal cracks up to 150 µm [5]

at best case. Although the maximum healable crack width limit

was reported widely varied between 5 to 300 μm [3], autogenous

healing crack width limit varies based on the cement content, exis-

tence of supplementary materials, water content and age.

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compared to incorporation of the multi-component methylmethacrylate [10] and two-component epoxy resin [8,9] systems. However, Dry and McMillan have argued that the short shelf life of single-component healing agents might be disadvantageous and multi-component healing agents have more stability over time [10]. In contrast, having further improved shelf-life, Mihashi et al. [9] depicted the limitation of two component epoxy resins due to insufficient mixing of the components inside the crack. This had resulted poor performance compared to a single-agent cyanoacrylates type adhesive agents. Recently, Van Tittelboom et al. [11] investigated the use of a two-component polyurethane foam as healing agent. They suggested that healing agents should have a low viscosity and that the polymerization reaction should not depend on the mix ratio of the two components.

Nevertheless polymeric resins can effectively recover the mechanical properties of concrete, they have the major drawback of limited shelf life (of single component resins), effective mixing (of two component resins) and cost when used in bulk quantities. The use of minerals compatible with the cementitious materials can resolve these limitations. Few studies reported the use of sodium silicate in microcapsules for self-healing concrete [12]. However, in these studies neither many details were given on the encapsulated system nor details on the actual self-healing processes and kinetics were provided. In a recent study, the authors reported the effectiveness of encapsulated liquid SiO2 precursors, sodium silicate and powder MgO as healing materials using a system of parallel thin walled glass capsules [4]. This study showed that sodium silicate and colloidal silica could improve dramatically the durability performance of cracked mortar samples. The results of the MgO powder were also promising; however, their limited healing performance was noted due to its poor dispersion in the crack plane while investigating the crack faces of the samples.

#### 1.1. Concentric glass macrocapsules' system

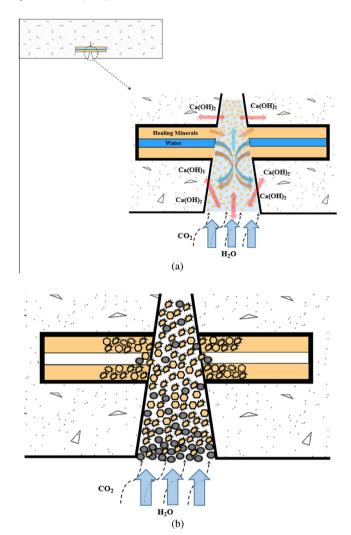
Since the major issue reported previously [4] regarding the encapsulation of mineral powder was its proper dispersion in the crack, this study focuses on an encapsulation system that will improve powder dispersion. To achieve this, a system of concentric glass macrocapsules was adopted with the inner capsule containing water and the outer capsule containing the powder minerals (Fig. 1).

Three expansive powder minerals (MgO, bentonite, and quick-lime) were used in this study. These minerals are known for their expansive properties during hydration and carbonation, and have been used previously for improving the autogenous healing capacity of a Portland cement (PC) system [13–16]. Principal products that are produced during the initial hydration of MgO and quicklime (CaO) are brucite (Mg(OH)<sub>2</sub>) and portlandite (Ca (OH)<sub>2</sub>) following the reaction in Eqs. (1) and (2) below. These hydration products have the potential to accelerate the self-healing mechanism [13,16] as they have cementing and crystal forming properties.

$$CaO + H_2O \rightarrow Ca^{2+} + 2OH^- \rightarrow Ca(OH)_2$$
 (1)

$$MgO + H_2O \rightarrow Mg^{2+} + 2OH^- \rightarrow Mg(OH)_2$$
 (2)

Bentonite is renowned for its swelling and expanding properties while bearing in contact with water. Literature further suggests that bentonite in high alkaline solutions can be modified in an aqueous silica solution and that the hydroxide solubility increases at pH > 11 [17] following the reaction refer to Eq. (3):



**Fig. 1.** Basic concept of the concentric glass capsules self-healing: (a) Release of expansive minerals within the cracks through dispersion and diffusion after crack formation and (b) consequent interaction of released healing agent with water and CO<sub>2</sub> results in the formation of different hydrated and carbonated products to seal and possibly heal the crack.

$$\begin{split} & \left(\text{Si}_{7.42}\text{Al}_{0.29}\right)^{IV} \left(\text{Al}_{2.69}\text{Fe}_{0.41}^{3+}\text{Mg}_{0.85}\right)^{VI} O_{2}(\text{OH})_{4} \\ & \times \left(\text{Ca}_{0.25}\text{Mg}_{0.20}\text{Na}_{0.8}\text{K}_{0.11}\right)_{1.29} (montmorrillonite) \\ & + 0.51\text{SiO}_{2}(silica, quartz, cristobalite, silicaglass) \\ & + 0.1\text{NaAlSi}_{3}O_{8}(albite) + 0.10\text{CaCO}_{3}(calcite) + 1.10\text{H}_{4}\text{SiO}_{4} \\ & + 0.78\text{Ca}^{2+} + 2.26\text{Na}^{+} + 4.100\text{H}^{-} \\ & \leftrightarrow 2.59\text{Na}_{1.02}\text{Al}_{1.02}\text{Si}_{2.58}\text{O}_{7.2}\text{O}.1.2\text{H}_{2}\text{O}(alancime) \\ & + 0.193\text{Ca}_{5}\text{Si}_{6}\text{O}_{16}(\text{OH})_{2}\text{O}.4.5\text{H}_{2}\text{O}(tobermorite) \\ & + 0.278(\text{Si}_{6.42}\text{Al}_{1.58})^{IV}(\text{Fe}_{1.48}^{3+}\text{Mg}_{3.78})^{VI}\text{O}_{20}(\text{OH})_{4} \\ & \times \left(\text{Ca}_{0.59}\text{K}_{0.40}\right)_{1.58}(saponiticclay) + 1.53\text{H}_{2}\text{O} + 0.10\text{CO}_{3}^{2-} \end{aligned} \tag{3}$$

Sanchez et al. [17] have characterised the hyper-alkaline reaction of Na-bentonite (0.5–0.1 M) by the dissolution of montmorillonite and the precipitation of zeolites, phillipsite, saponite, calcium silicate hydrate-gel, tobermorite and gyrolite in different temperature ranges which was driven by montmorillonite dissolution. This property of bentonite makes it a suitable choice as expansive healing agent for self-healing concrete.

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