Encapsulated mineral precursors for self-healing cement based composites

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Keywords: self-healing, cement-based composites, minerals encapsulation

Abstract ID No: CM-100

ABSTRACT

The resilience of buildings and civil engineering structures is typically associated with the design of individual elements such that they have sufficient capacity or potential to react in an appropriate manner to adverse events. Traditionally this has been achieved by using “robust” design procedures, described in national and international codes of practice, that focus on defining safety factors for individual adverse events and providing redundancy. As such, construction materials are designed to meet a prescribed specification; material degradation is viewed as inevitable and mitigation necessitates expensive maintenance regimes. In the UK alone this translates to ~£40 billion/year on repair and maintenance of existing, mainly concrete, structures [1]. In the United States the situation is worse; in 2006 a study reported that concrete structure owners nationwide pay ~$18 to $21 billion/year on repair, protection and strengthening whereas the associated costs for maintenance due to steel corrosion reach $125 billion/year [2].

1. MATERIALS AND PREPARATION

The selection of minerals was made on the basis of having materials that are compatible with the host matrix and concrete’s constituent materials. In concrete technology literature the effect of addition of silicon oxides in cement-based materials is well documented. Silicon oxides react with portlandite and produce surplus of calcium silicate hydrate resulting in a denser and more durable material. On this basis the minerals the materials used for encapsulation in this study were selected for their potential to act as silicon oxide precursors. More specifically the liquid mineral that encapsulated was sodium silicate (SS) and the powder mineral selected for encapsulation was magnesia (magnesium oxide or MgO). Sodium silicate had been used in the past as setting accelerator in normal concretes, as an alkali activator in geopolymer concretes and in some cases to improve the durability of concretes [3,4]. Reactive magnesium oxide (MgO) has emerged recently as a very promising addition/replacement to cement with many technical and sustainability advantages as reported in the literature [5–7]. Moreover, when used as an addition to cement based mixtures, proved to improve autogenous healing [8]. In this research, 92/200 type magnesia was used and its reactivity was found 149 seconds using the accelerated acidic test.

Soda glass capsules were used as carriers for the mineral healing compounds. The thin wall (0.45mm) glass capsules were 50mm long with 6.15mm inner diameter and

were able to carry ~1.5ml of liquid cargo. The glass capsules embedded in mortar prisms with 50x50mm cross-section and a length of 220mm. The mortar mixtures were prepared with sand to cement ratio of 1.5 and consisted of CEM-I 52.5 cement, fine sand with 2mm maximum grain size and water to cement ratio of 0.4. Two glass capsules placed next to each other and both were positioned at the middle of each specimen with a cover of 6.5mm to the bottom face. For the samples containing encapsulated MgO only one of the capsules contained the reactive powder whereas the second capsule contained water which, after rupture, expected to disperse the MgO powder in the crack. In the case of control samples the capsules contained water. All samples were initially stored in water for seven days. On the seventh day the prisms were cracked and left to cure in the predefined curing conditions. The aim of this study was to investigate the potential of the healing compounds under different exposure conditions. Therefore, different batches of prisms were cured in ambient conditions, high humidity exposure (relative humidity 90%) and immersed in water. In all three curing regimes the temperature kept constant at 21ºC and specimens cured for 28 days.

2. METHODS

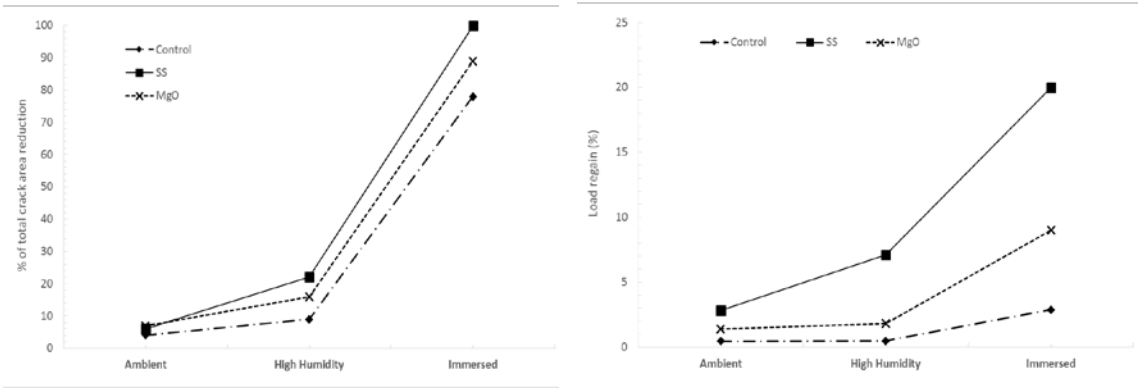
Mechanical loading of prisms performed on a 30kN static testing frame. As mentioned before prisms were initially left to cure for seven days immersed in water and following that were cracked. Prior to cracking all specimens were notched with a rotating diamond blade and their crack opening monitored with a clip gauge. Upon load removal all specimens had a remaining crack in the range of 0.20-0.25mm. During the second round of loading, after 28 days of healing, specimens loaded using the same test parameters as before. However, this time specimens brought to failure. In order to compare the healing efficiency of each mineral in each case the load recovery rate coefficient (LR%) was calculated as follows:

$$LR(\%) = \frac{P_{max,h} - P_{un}}{P_{max,i} - P_{un}} \times 100$$

Where $P_{max,i}$ is the load at which initial cracking occurred, P_{un} is the unloading point of the first test and $P_{max,h}$ is the maximum load attained by the healed sample during the second test. Bottom crack faces in all specimens were monitored over time using a stereo macroscope. Digital images were taken at three different positions of the crack face. Image analysis software was then used to analyse the acquired captions and the total crack area was calculated for each case.

3. RESULTS

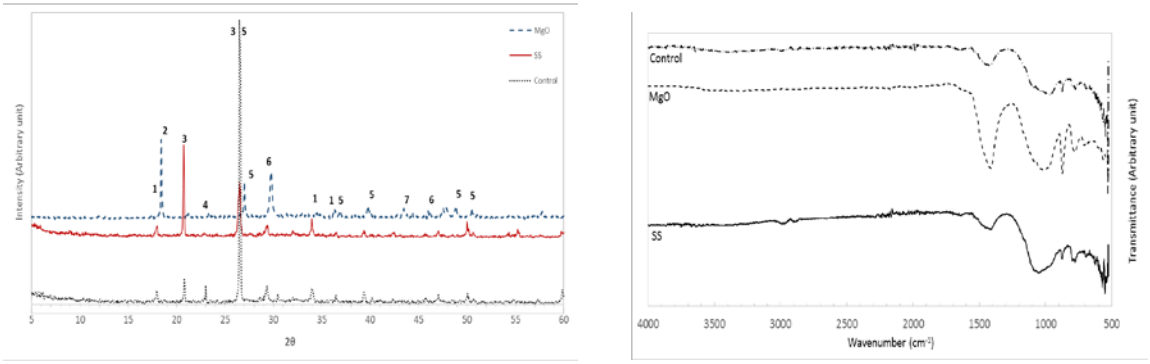
Figure 1 summarises the results for both the percentage reduction of the total crack area as obtained by image analysis and the percentage of load regain.



(a) (b)

Fig. 1. (a) % reduction of the total crack closure area for all exposure conditions and (b) % regain of load for all exposure conditions

The importance of water in the proliferation of healing products in the crack is evident when comparing the percentage reduction in the crack area. For both mineral compounds cured immersed in water the total crack area reduction found to be more than 90%. The observed crack sealing is much less for samples stored at high humidity and ambient conditions. Nonetheless, in the presence of high humidity crack area closure improves significantly compared to ambient conditions. It seems that for immersed specimens, as water penetrates into the crack improves the diffusion of mineral compounds and accelerates the reactions that yield healing products in the crack. The remarkably high values of the total crack area closure are not reflected in the load regain. Despite that, it is evident that all mineral healing compounds improve the load carrying capacity of the sections compared to the control specimens for all curing environments. Sodium silicate gives the best results in all three cases, reaching a maximum of 20% load regain for samples that healed immersed in water. Once again water is a critical factor in the development of the healing process for both liquid minerals and MgO. This is translated to an increase of at least three times in the recorded load regain values between samples cured at high humidity and immersed in water. XRD patterns and FTIR patterns of the healing products collected from the crack planes of samples cured under water are shown in Figure 3. Most of the peaks and patterns are common to all samples.



(a) (b)

Fig. 3. (a) XRD patterns for all healing products (1: $Ca(OH)_2$; 2: $Mg(OH)_2$; 3: SiO_2; 4: Ettringite; 5: C_3S/C_2S; 6: $CaCO_3$; 7: $MgO/MgCO_3$) and (b) FTIR spectra of the healing products developed in specimens stored under water.

Comparison of the XRD patterns with control shows development of different crystalline phases. In all the samples hydration products are observed including portlandite, ettringite, poorly crystallised calcium silicate hydrates and unhydrated calcium silicates. All samples indicated characteristic bands in the range 1400-1500 cm^{-1} due to stretching C-O bonds which correspond to carbonate phases. The latter also give some less strong peaks in the range between 870 and 890 cm^{-1}. The existence of calcium silicate hydrates (CSH) is confirmed by Si-O stretching vibrations that give peaks at bands between around 970 and 1000 cm^{-1}. Compared to control sample it is observed that CSH attained peaks gradually moved to larger bands (Control: 961 cm^{-1}; MgO: 966 cm^{-1}; SS: 998 cm^{-1}). This phenomenon can be attributed to increased polymerisation of silicate chains and the production of more CSH. This also explains the better defined, and stronger expressed, peaks around 550 cm^{-1} especially for CS and SS samples.

4. CONCLUSIONS

Results showed that mineral compounds (liquid and powder) have a relatively good potential to be used as encapsulated healing materials in cement based composites.

ACKNOWLEDGEMENTS

Financial support from the Engineering and Physical Sciences Research Council (EPSRC) for this study (Project Ref. EP/K026631/1) is gratefully acknowledged.

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