



A self-healing cementitious composite using oil core/silica gel shell microcapsules

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ARTICLE INFO

Article history:

Received 6 December 2009

Received in revised form 3 December 2010

Accepted 29 January 2011

Available online 4 February 2011

Keywords:

Self-healing concrete

Microcapsules

Carbon microfibers

Cementitious composite

ABSTRACT

This paper presents work toward the development of a new family of self-healing materials that hold promise for “crack-free” concrete or other cementitious composites. This innovative system features the design of microcapsules with oil core and silica gel shell. Methylmethacrylate monomer and triethylborane were selected as the healing agent and the catalyst, i.e. oil core phases in the system. They were microencapsulated, respectively, through an interfacial self-assembly process and sol–gel reaction. Then the microcapsules were dispersed in fresh cement mortar along with carbon microfibers. The self-healing effect was evaluated using permeability measurements along with a fatigue test under uniaxial compression cyclic loading and further confirmed by surface analytical tools including optical microscope and field emission scanning electron microscope coupled with energy-dispersive X-ray analyzer (FESEM/EDX).

Published by Elsevier Ltd.

1. Introduction

With a smaller fracture energy relative to that of mild steel (about 0.1 kJ/m² vs. 100 kJ/m²), cementitious materials are known to be inherently brittle and tend to crack under stress. Once microcracks form in concrete, they are difficult to detect and repair by conventional methods before they develop, coalesce and grow into macrocracks, which can pose a significant risk for the performance and service life of concrete. While better structural design, material selection and proportioning, and construction practices can help control concrete cracking, continued research in the domain of materials science (e.g., application of various fibers [1]) is crucial in improving the service life of concrete structures.

In recent years, the biomimetic concept of self-healing has been demonstrated for cementitious materials by incorporating healing agents in hollow porous fibers, or in hollow glass tubes with a brittle breakable sealer [2–6]. This concept has also been demonstrated for polymer composites [7,8], by dispersing the microencapsulated repairing agent and catalyst within the composite matrix phase, or by a bleeding action from filled hollow fibers [9]. As a response to applied heat or vacuum (active mode) or to mechanical loading (passive mode), the repairing chemicals

(monomers) are released and repair the cracks or fill the voids often through polymerization [10].

The use of short fibers to reinforce cementitious composites seems to be a promising strategy in mitigating cracking at the microscale. Some composite systems with fiber reinforcement feature the strain-hardening behavior associated with the enhancement of tensile/flexural strength and toughness due to the combined actions of the fibers, cementitious matrix and their interface [11–14]. Owing to their small diameter and high aspect ratio, short fibers have a large numerical density in the matrix even when admixed at relatively low volume fractions and thus have the potential to interact with microcracks formed in cementitious composites [15]. As such, short fibers have been proven to effectively flank and bridge microcracks before they become coalescent and form macrocracks [16]. The admixing of short fibers in cementitious composites has shown many prospective benefits, especially in enhancing the self-healing capability of cementitious materials [17,18], but the effectiveness hinges on the uniform dispersion of fibers in the matrix.

This paper presents work towards the development of a new family of self-healing materials that are expected to offer potential synergy with microfibers in arresting cracks formed in concrete or other cementitious materials. The microfibers are expected to control the width of the cracks at the microscale in this work. This system we proposed herein features the design of microcapsules (PSMs) with oil core and silica gel shell, consisting of methylmethacrylate (MMA) as the healing agent and an initiator (catalyst) containing triethylborane (TEB). The healing agent and catalyst are microencapsulated respectively through an interfacial self-assembly process and sol–gel reaction [19,20]. More technical

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details related to this process can be found in the following parts of this paper. The healing is designed to occur through a passive mode, i.e., as a response to crack propagation caused by cement hydration or external mechanical stimuli. The microcapsules are dispersed in fresh cement mortar along with carbon microfibers. For the hardened mortar, self-healing can be triggered by crack propagation through the microcapsules, which then releases the healing agent and the catalyst from their respective stress-ruptured reservoir upon crack intrusion. The monomer and its initiator have similar viscosity with water (i.e., dynamic viscosity is around 1.0 cP), so they can easily migrate into the microcracks through capillary action. Polymerization of the healing agent is then initiated by contact with the catalyst, bonding the crack faces.

2. Experimental

2.1. Materials

Commercially available monomers: styrene (St) and methylmethacrylate (MMA), poly(vinylpyrrolidone) (PVP), tetraethyl orthosilicate (TEOS), and concentrated sulfuric acid (98%) were obtained from Fisher Scientific Inc. Triethylborane solution (1.0M in hexanes) was obtained from Sigma–Aldrich, Inc. All the solvents were of analytical grade and were used as received. Deionized water was used for all experiments. The carbon microfibers were KRECA chop C-103T, 3 mm in length with a filament diameter of 18 μm , as obtained from Kureha Co. (Tokyo, Japan). In light of published work by other researchers [1,15], ASTM C1240 silica fume obtained from BASF/MB admixtures, Inc. (Henderson, NV), was used as a dispersant for the fibers in the mixes. ASTM C150-07 Type I/II low-alkali Portland cement was used in this study. The chemical composition and physical properties of the cement were presented in a previous reference [21]. The fine aggregate used was river sand sifted to allow a maximum aggregate size of 1.18 mm. Before proportioning and admixing, the aggregate was pretreated and taken to a saturated surface dry (SSD) condition.

2.2. Preparation of the PSMs

The overall preparation procedure of the PSMs is illustrated in Fig. 1. The first step was to self-assemble surface-sulfonated poly-

styrene particles at the water–oil droplet interface. The oil phase, either methylmethacrylate (MMA) monomer or triethylborane (TEB) solution (1.0M in hexane), was microencapsulated following the same procedure. The surface-sulfonated polystyrene particles of $0.7 \pm 0.5 \mu\text{m}$ in diameter were prepared as reported previously [20] and the sulfonation time of polystyrene particles was 60 h for this study. After some preliminary trials, 0.15 g of the sulfonated polystyrene particles were dispersed into 45 ml deionized water using a high-intensity ultrasonic vibracell processor operated at 20 kHz and up to 10 W for 2 min to form a homogeneous system. Then 3 g of oil phase (oil/TEOS = 17/3, mol/mol) was introduced and stirred continuously using a magnetic stirrer operated at 500 rpm under the nitrogen atmosphere. The turbid mixture gradually evolved into a creamy-white emulsion in appearance after 20 min. Then the sample was set aside for 24 h.

During the reserving period, the TEOS tended to diffuse into the interface of oil phase and water, leading to hydrolysis and condensation reactions of alkoxy silane precursors that were catalyzed by surface sulfogroup of the sulfonated polystyrene particles. After 24 h, microcapsules with oil core and silica gel shell were formed and at the same time the adsorbed polystyrene particles disengaged from the shell. The microcapsules with MMA oil core were used as reservoirs for healing agent, while the microcapsules with TEB oil core were used as reservoirs for chemical initiator (catalyst) in this system.

2.3. Preparation of mortar specimens

Three types of carbon microfiber-reinforced mortar specimens including the control (with no particles admixed), the sulfonated polystyrene modified mortars (SPSM, with sulfonated polystyrene particles admixed), and the self-healing mortars (SHM, with microcapsules admixed) were investigated. For each type of mortars, three or more specimens were fabricated for each planned test to ensure the statistical reliability of the test results. The carbon microfiber content in the mortar mix was 2.0% by volume. The silica fume content was 15% by weight of binder. All specimens were prepared in an ordinary mortar mixer at a constant water-to-binder mass ratio of 0.52 and a constant sand-to-binder mass ratio of 2. The mixing sequence was the following: the cement, silica

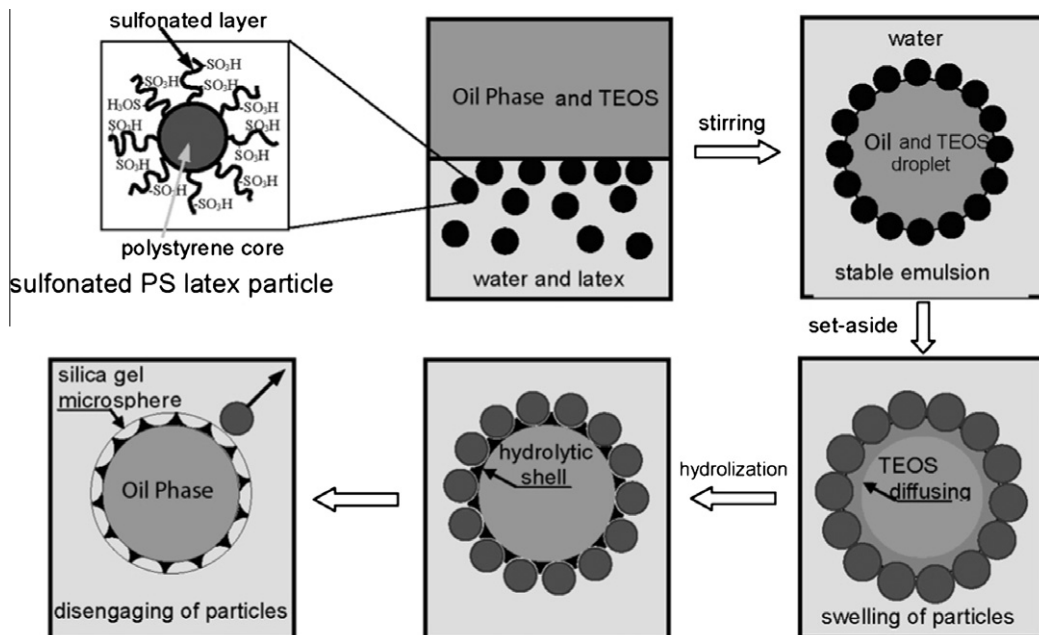


Fig. 1. Schematic illustration of the formation of the passive smart microcapsules.

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