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Synthesis and characterization of a new polymeric microcapsule and feasibility investigation in self-healing cementitious materials

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HIGHLIGHTS

- PF/DCPD microcapsules were prepared via in situ polymerization.
- The mean diameter and size distribution of microcapsules could be controlled.
- The microcapsules possess good chemical stability in cementitious materials.
- XCT was used to visualize the distribution and rupture process of microcapsules.
- The microcapsules have a good dispersibility and trigger sensitivity to the cracks.

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ABSTRACT

This paper presents work towards development of a new type of polymeric microcapsule with phenol–formaldehyde (PF) resin as shell and dicyclopentadiene (DCPD) as healing agent for self-healing microcracks in cementitious materials. The PF/DCPD microcapsules were synthesized via in-situ polymerization and characterized by means of optical microscope (OM), scanning electron microscope (SEM) and thermal analysis (TGA). The chemical stability of synthesized microcapsules and the trigger performance were studied respectively in simulated concrete pore solution and hardened cement paste specimens. The results revealed that the synthesized microcapsules possessed excellent stability in both simulated pore solution and real cement environment. X-ray computed tomography (XCT) was applied to observe the status and fracture behavior of microcapsules inside cement paste matrix. Further segmentation and 3D rendering of the reconstructed data obtained from XCT showed that the microcapsules had a good dispersibility with desired trigger sensitivity. The OM investigation of a fractured surface of a cement paste incorporated with microcapsules confirmed that the self-healing function of the microcapsules could be triggered by cracking and the healing agent could be released simultaneously to heal the cracks.

1. Introduction

The Portland cement and other cementitious materials are the most widely used construction materials in the world. However, they are susceptible to many sources of damage during their entire service life. Cracking related deterioration of cementitious materials is one of the most serious threats to the safety, integrity and durability of concrete structures. Once micro-cracks formed in cementitious materials, they are difficult to detect and repair by conventional methods before they show on the surface of structure, which has long been an extremely difficult question to the maintenance of buildings and infrastructures. Although better structural design, raw material selection and proportion can help reduce the probability of cracking, further study in searching for a more efficient and automatic way to reduce the maintenance/repair frequency and improve the service life of concrete structures is essentially in need.

In recent years, inspired from biological self-healing phenomenon, the microcapsule based self-healing concept began to show its great application potential in cementitious materials [1–7]. Once the incorporated microcapsules are ruptured by the cracks or other stimuli, the self-healing is realized through the release and reaction of repairing chemicals in the region of damage. As an essential part in self-healing composite, the properties of

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microcapsule (e.g. the shell materials) are believed to have a great influence on the self-healing performance and the mechanical properties of cementitious composite [8,9]. Up to date, a wide range of materials have been explored as the shell in microencapsulation [10–13]. Inorganic compounds such as alginate, silica gel and expanded clay [1,14,15] have been described to be an effective shell material in fabricating microcapsules for self-healing cementitious materials. Owing to the similar chemical properties, microcapsules with an inorganic shell are generally considered more compatible with the cementitious matrix and may produce a stronger binding between the two phases. However, some of the microcapsules could be ruptured by mechanical mixing during the preparation of cement mixtures and/or the later curing stage due to the loose crumb structure of the inorganic shell. Additional concerns could be the leaching out of healing agent from the mesh structural shell over the long service life of concrete structures and consequently the loss of healing effectiveness and the rising risk of pollution posing on the surrounding environment. Organic compounds such as polystyrene, polyurethane and urea–formaldehyde resin [2,6,16] have also been used for encapsulating healing agents in the past decade, while the reliability over time and the trigger sensitivity of capsules have not attracted sufficient attention. Melamine-based organic compounds have been studied as a promising type of shell materials to encapsulate bacterial spores for self-healing concrete [17]. It was illustrated that the synthesized microcapsules can survive the mechanical mixing during the preparation of cement mixtures and the later curing stage. Although the microcapsules were demonstrated to rupture under certain tensile force, the probability for practical application was greatly hindered due to the rigorous requirement for the incubation condition of the bacteria healing agent.

To develop microcapsules which are practically applicable of providing long-term self-healing capacity for cementitious materials, three specific requirements need to be met: (1) the microcapsules are chemically stable in the high alkaline environment of cement mixtures, (2) the microcapsules can survive the mechanical

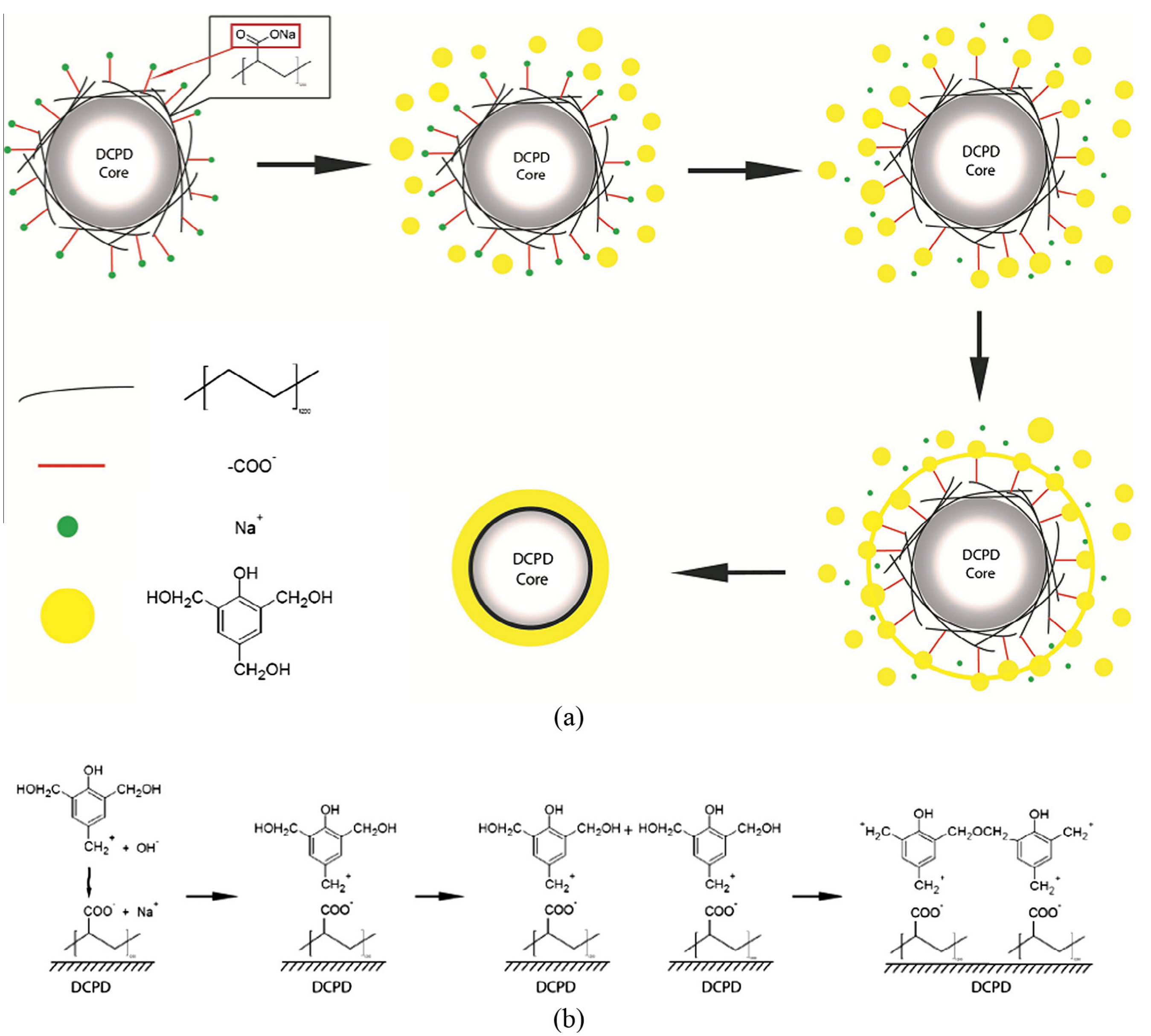

Fig. 1. Schematic illustration of (a) the synthesis route of PF shell/DCPD core microcapsules via in situ polymerization and (b) the possible mechanism of the interactions between the carboxylic group and hydroxymethyl group and the condensation process of PF resin.