INORGANIC POWDER ENCAPSULATED IN BRITTLE POLYMER PARTICLES FOR SELF-HEALING CEMENT-BASED MATERIALS

H. Dong [1], H. Huang [1] and G. Ye [1]

[1] *Microlab, Faculty of Civil Engineering and Geosciences, Delft university of Technology, Stevinweg 1, 2628 CN Delft, The Netherlands - Email: H.Dong@tudelft.nl*

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ABSTRACT

Many types of healing agents have been investigated. These agents are processed in different ways, such as adhesive polymer in capsules or hollow fibre glasses, bacteria in porous aggregates and geo-materials directly incorporated in the cement-based materials. In this study, sodium silicate powder is encapsulated in polystyrene particles (polystyrene particle containing sodium silicate is defined as PS particle in short).

The PS particles remain intact in the cement-based matrix before cracking. If water or moisture is available, the healing agent can be released into the crack provided that the crack passes through the PS particles. The dissolved sodium silicate reacts with calcium hydroxide in the matrix, and the healing products (C-S-H gel) can form in the crack. Furthermore, compared to the reference, for the cracked specimens with polystyrene particles, the recovery of flexural stiffness can be observed.

Different sizes and mass fractions, i.e. sodium silicate / cement ratios, of PS particles used in engineered cementitious composite (ECC) mixture are studied to see their influence on mechanical properties as well as their healing efficiency. When the mass fraction of polystyrene particles is 4% of cement and the polystyrene particles have a proper slender shape, the ECC show good results in terms of flexural strength, flexural deflection capacity and recovery of mechanical properties. Therefore, encapsulation of healing agent in polystyrene could be regarded as a promising way for realising self-healing of cement-based materials.

1. INTRODUCTION

Cracks are inevitable in concrete, possible resulting in further problems such as corrosion of reinforcement, thus accelerating the deterioration of concrete structures. To reduce the cost of repair and prolong the service life of concrete structures, self-healing of concrete has been regarded a promising solution. Many types of healing agents processed in different ways have been studied in the last decade, such as adhesive polymer in capsules or hollow fibre glasses [1], bacteria in porous aggregates [2] and geo-materials directly incorporated in cement-based materials [3].

In cement chemistry, $Ca(OH)_2$ is one of the main hydration products in cementitious materials. Under certain conditions, $Ca(OH)_2$ can facilitate the self-healing process. For example, $Ca(OH)_2$ can be designed to react with sodium silicate solution that is encapsulated in polyurethane capsules [4]. However, for those capsules it is difficult to survive during casting in construction due to their weak shell. For self-healing, it is of great importance that the healing agent should be well protected before the healing process is triggered by the crack. In general, polystyrene is widely used in the industry and it is one of the most brittle polymers. This characteristic of polystyrene facilitate its rupture in cementitious materials. In this study, concerning controlled release of the healing agent, sodium silicate powder is encapsulated in polystyrene matrix. The self-healing process of cementitious materials by using polystyrene particle (PS particle) is studied, the size and amount of polystyrene particles are optimized as well.

2. MATERIALS

Na_2SiO_3 powder (diameter: 500-700 µm) and polystyrene beans (Ø2 mm × 3 mm, ultimate tensile capacity is 2%) are mixed at 170 ℃ by single screw extruder and pressed (170℃) into thin sheets (thickness: 1 mm). The amount of Na_2SiO_3 powder (36% by mass) used in the mixture is determined by the viscosity of the mixture. To improve the bond between PS particles and cementitious matrix, a coating of epoxy resin was applied on the PS sheets, and another layer of fine sand (diameter: 300-500 mm) is coated to the polystyrene sheets (After coating, mass fraction of Na_2SiO_3 is 20%).Then the sheets are cut into small particles with specified sizes, which are expected to rupture upon crack propagation in the cementitious materials (Fig 1).

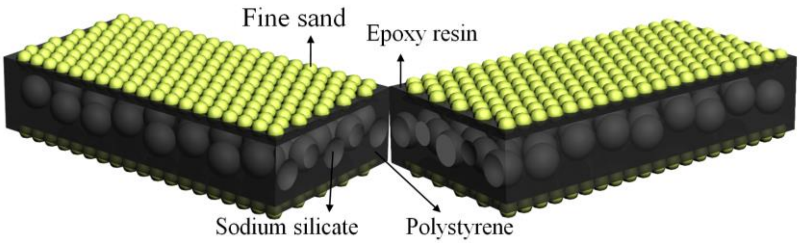

Figure 1: Rupture of PS particles (3 × 12 × 2mm^3)

Engineered cementitious composite (ECC) is adopted for the evaluation of healing efficiency of PS particles due to its good ductility with tight micro-cracks which are beneficial for self-healing process [5]. In this study, materials of thin ECC layers (size of 120 × 30 × 10 mm^3 or 120 × 30 × 5 mm^3) used for all tests consist of Portland cement CEM I 42.5 (PC), blast furnace slag (BFS), limestone powder as fine aggregate (LP), naphthalene based superplastisizer (SP) and PVA fiber (diameter: 39 µm, length: 8 mm).

2.1 Observation of self-healing process – proof of principle

A trial test to reveal the healing process is conducted by adding PS particles to a ECC mixture. The mix proportion (Kg) for 1m3 ECC is PC : PS particles : BFS : Water : SP : PVA fiber = 945 : 135 : 405 : 405 : 13.5 : 26. It is found that PS particles remain intact during mixing in the mixture. Then the mixture is poured into the mould with size of 240 × 60 × 10 mm^3 followed by sealed curing for 28 days at 20 ℃.

2.2 Optimization of PS particles and evaluation of healing efficiency

To evaluate the healing efficiency of PS particles in cementitious materials, different volume fractions and different sizes of PS particles are taken into account in the mixtures (see table 1). 4-point bending tests are conducted. The volume fraction (v%) of PS particles is 0 (for reference), 3.45%, 6.9% and 10.35%, respectively (corresponding ratio of Na_2SiO_3 to cement as 0, 2%, 4% and 6%). In each series, PS particles with a size of 3 × 12 × 2 mm^3 are used. M3 is designed for the evaluation of particle sizes, PS particle sizes of 5 × 5 × 2 mm^3, 3 × 12 × 2 mm^3, 3 × 20 × 2 mm^3 and 5 × 20 × 2 mm^3 are used in this series. After mixing, the mixture is cast into the mould (size: 240 × 60 × 5 mm^3) followed by sealed curing for 28 days at 20 ℃.

Table 1: Mix proportion of ECC (Kg/m^3)

	V% of PS	PC	LP	PS particle	BFS	Water	SP	PVA fiber
M1	0	534	427	0	640	469.6	11.7	26
M2	3.45%	534	333.8	51.1	640	469.6	9.4	26
M3	6.9%	534	240.6	102.2	640	469.6	7.0	26
M4	10.35%	534	147.3	153.3	640	469.6	4.7	26

3. METHODS

In the trial test for self-healing in ECC, after 28 days curing, the specimen is cut into 120 × 30 × 10 mm3 and cracked by 4-point bending test (setup span is 110 mm, load span is 30 mm. Loading speed is 0.02 mm/s, and vertical deflection is controlled to 1 mm), the crack width obtained is in the range between 50 - 100 μm . The cracked specimens are subjected to 3 wet-dry cycles. One wet-dry cycle consists of 24h merging in the water (20 ℃) and 24h drying in the air (20 ℃, 50% relative humidity).

For the optimization of PS particles and evaluation of the healing efficiency, the specimens are cut to 120 × 30 × 5 mm^3 for 4-point bending tests. Each series uses 8 specimens, which consist of:

- Three specimens for determination of ultimate flexural strength at 28 days.
- Two specimens (for reference) are preloaded to 3 mm deflection at 28 days. After 3 days curing in air (20 ℃, 50% relative humidity), the specimens are reloaded to failure.
- Three specimens (for self-healing) are preloaded to 3 mm deflection at 28 days. After 3 days curing in 100% RH curing room (20 ℃), the specimens are reloaded to failure.

In this test a loading speed of 0.02 mm/s is applied. For the calculation of flexural stiffness, stress from 1 MPa to 2 MPa (linear part) is specified (Fig 2). Ratio of flexural stiffness (R_S) is defined in equation (1). Higher R_S indicates a higher recovery of flexural stiffness after curing. For R_S it holds:

$$R_S = \frac{S_{Pre}}{S_{Re}} \tag{1}$$

where S_{Pre} is the flexural stiffness of specimen preloaded at 28 days, S_{Re} is the flexural stiffness of specimen reloaded at (28 + 3) days.

4. RESULTS

In the trial test for self-healing in ECC, after 3 wet-dry cycles, healing products are observed on the surface of cracks (Fig 3). From energy dispersive spectroscopy (EDS), it can be concluded that the healing products are mainly composed of C-S-H.

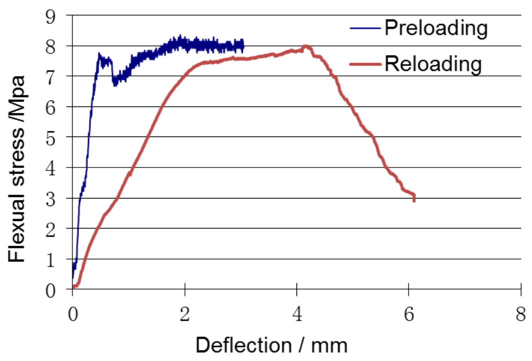

Figure 2: Stress-deflection curve of ECC

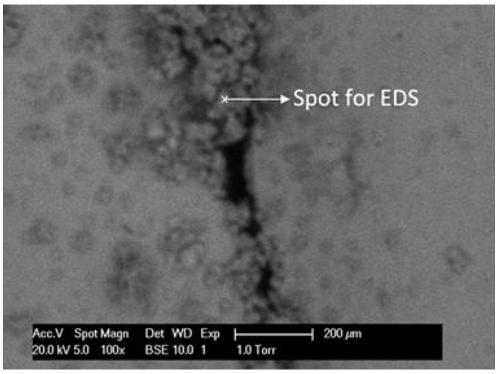

Figure 3: Micrograph of crack after healing

Optimization of PS particles is studied to see their size and volume effect on the mechanical properties of cementitious materials. In this test, specimens with different sizes and volume fractions (v%) of PS particles are bended to failure at 28 days. The results of flexural strength and deflection capacity are shown in Fig 4 and Fig 5.

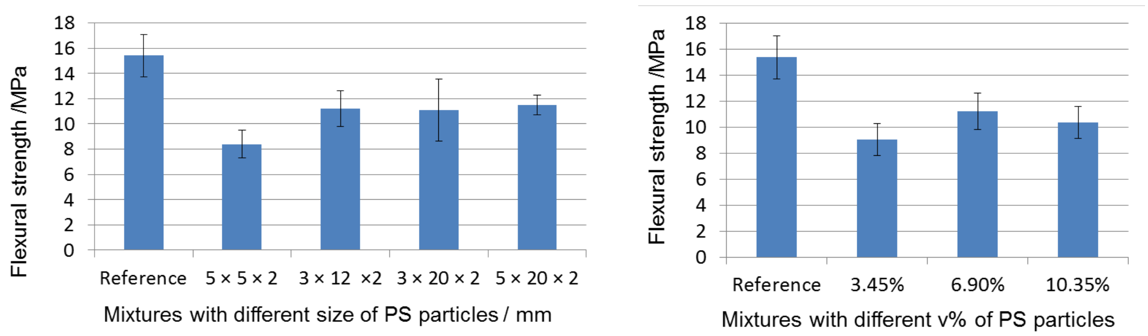

Figure 4: Flexural strength at 28 days

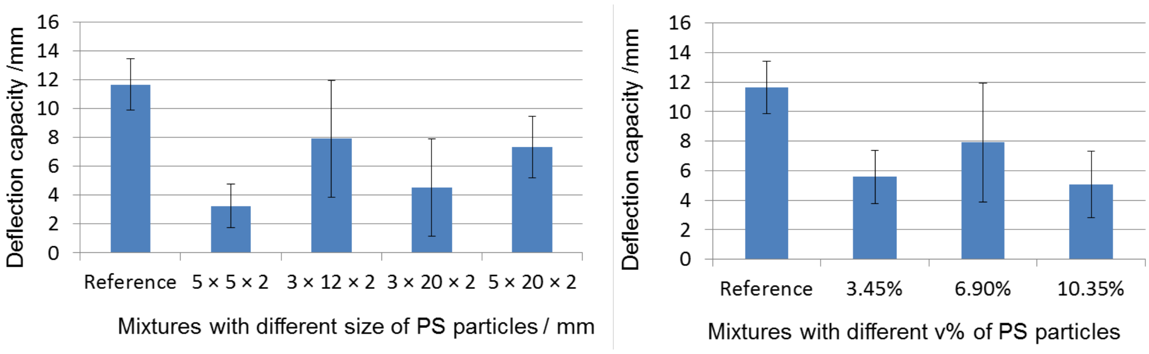

Figure 5: Flexural deflection capacity at 28 days

It can be seen that there is a lower reduction of flexural strength for the specimens with the PS particles in a slender shape, and PS particles with the size of 3 × 12 × 2 mm^3 has the lowest reduction of the deflection capacity. For the volume fraction of PS particles, it seems that 6.9% is the best one among 3 volume fractions incorporated in the specimens. In this test, PS particles are applied to replace a certain amount of limestone powder. Therefore, there is a delicate balance between the amount of PS particles and the total amount of binder.

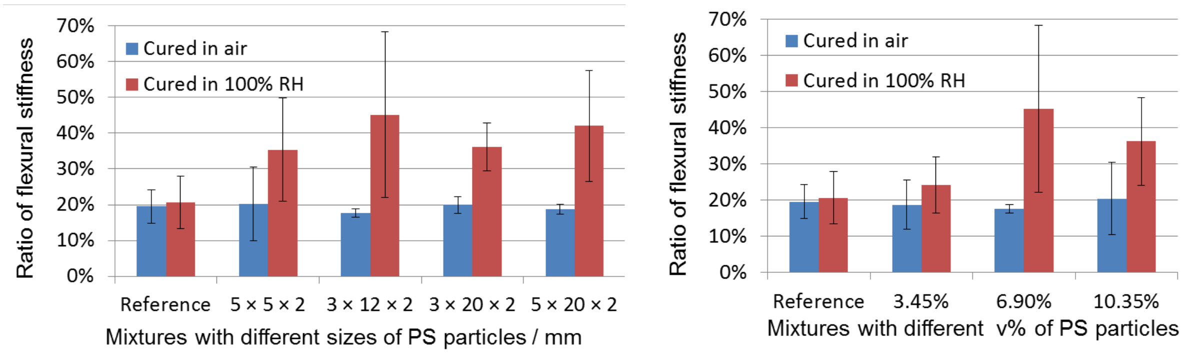

Figure 6: Ratio flexural stiffness (R_S) of specimens

To evaluate the healing efficiency of PS particles, after another 3 days curing (in air, or in 100% RH curing room) the ratio of flexural stiffness (R_S) of each specimen is calculated and plotted in Fig 6. Similar to the flexural strength and deflection capacity, PS particles with the size of 3 × 12 × 2 mm^3 and the volume fraction of 6.9% show the best results. From the results, high errors for the flexural stiffness ratio are observed which is in line with other results published in the article[6]. It is because of the presence of fibers and PS particles, and the flexural capacity and ratio of flexural stiffness (R_S) are dependent on the quantity and width of micro cracks. Nevertheless, the difference between different particle sizes and volume fractions are obvious.

5 CONCLUSIONS

A new self-healing agent, polystyrene particles containing Na_2SiO_3 is proposed. The polystyrene particles containing Na_2SiO_3 is proved to work well with cementitious materials. During mixing and casting, PS particles remain intact and can be ruptured upon the propagation of cracks. Then Na_2SiO_3 powder can be released into the crack when water or moisture is available. After moisture curing, the higher ratio of flexural stiffness (R_S) of ECC specimens with healing agent indicates a higher recovery of mechanical property than the reference. For particle dimensions, it shows that PS particles in a slender shape are preferable since they can bridge the cracks as fibers before failure of ECC.

ACKNOWLEDGEMENTS

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