

# Preparation of biocompatible and water-proof microcapsule for self-healing concrete

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## ABSTRACT

Koch's bacillus DSM6307 was microencapsulated in polydimethylsiloxane with hydrophobic epoxy resin for concrete application. The process is anhydrous to avoid the germination of spores. All the materials used were harmless. After microencapsulation, the fraction surviving of spores is over 90%, indicating the process has mild effect on spore's viability. Soaked in water more than 10 days, there are no swollen microcapsule and no germinated spore are found.

## 1. INTRODUCTION

Some bacilli are able to biologically induce the precipitation of calcium carbonate, so recently their spores were employed as an efficient repair agent in self-healing concrete researches<sup>[1-3]</sup>. Usually, these spores are easy to germinate in the appearance of water, so microencapsulation by a waterproof and biocompatible material is necessary to help them pass the incubation period, especially in the cement-hydration stage<sup>[4]</sup>.

Generally speaking, natural polymers are biocompatible while synthetic polymers are deleterious in various degrees to the microorganisms like bacteria, so most of the existing biological microcapsules are walled by natural polymer materials<sup>[5]</sup>. However, natural polymers are usually hydrophilic, will uptake water causing the shell of microcapsule swelling, therefore not suitable for concrete application. In this paper, bacillus spores were tentatively microencapsulated with harmless and waterproof epoxy shell.

## 2. MATERIALS AND METHODS

### 2.1 Materials

Koch's bacilli DSM6307 were supplied with microcrystalline cellulose(MCC) carrier by school of life science, Shenzhen university, Shenzhen, Guangdong, China. Polydimethylsiloxane(PDMS) were purchased from Aladdin Reagent Co., Shanghai, China. Epoxy resin E-51 and its' curing agent, KH-792 [N-(amino-ethyl)-amino-propyl trimethoxy silane], were products of Tianjin Chemical Plant, Tianjin, China. All the chemicals used are analytical pure.

### 2.2 Biocompatibility test

Take the same amount of spores mixed with epoxy E-51, KH-792 and water respectively, rest for 160mins, isolate and collect the spores, cultured for 24hrs, then extract the culture medium to determine the optical density by 490nm-ultraviolet absorption value. OD<sub>490</sub> are positive correlated with the bacterial concentration.

### 2.3 Preparation of microcapsules

3g Koch's bacillus DSM6307 spores, 250g culture medium (yeast powder: inosine:  $\text{Na}_2\text{NO}_3=25:5:150$ ) and 150g MCC were mixed with 150ml water, the dough obtained was delivered into a twin-screw granulator (mini250, Xinyite Sci. and Tech. Co., Ltd, Shenzhen, China) for granulation, then cryodesiccated, dry core particles was obtained.

Weighing 10g core powders, 10g epoxy E-51, 1g KH-792, mixed in a flask at  $50^\circ\text{C}$  for 40mins. Add 200ml PDMS, agitated at 300rpm for another 60mins, the microcapsules was formed.

It must be explained, empolying PDMS as the reaction medium is due to its specific gravity ( $0.97\text{g}/\text{cm}^3$  at  $25^\circ\text{C}$ ) similar to that of core materials, so that the core pills can be suspended in the medium for a long time.

### 2.4 Characterization of the microcapsules

The morphology of microcapsules was observed under Hitachi SU-20 SEM, by which the particle diameters and shell thickness were measured.

### 2.5 Determination of viability

Weighing 1g core particles as a blank copy, ground into fine powders, then added into liquid nutrient medium, diluted  $10^8$  times with deionized water, extract  $100\mu\text{L}$  solution into  $\phi 90$  petri dish, cultured for 24hrs, counting the number of colonies and calculating the amount of spores.

Weighing another copy of 1g core particles, microencapsulated, then treated as the blank copy.

$$\text{fraction surviving} = \frac{\text{the amount of spores in blank copy}}{\text{the amount of spores in microencapsulated copy}}$$

### 2.6 Waterproofing test

Soak the microcapsules in fresh water at room temperature, observe the shape change under JPL1350 optical microscope.

## 3. RESULTS AND DISCUSSION

### 3.1 Biocompatibility of raw materials

$\text{OD}_{490}$  value of epoxy E-51, curing agent KH-792 are 49.1% and 46.1% respectively, slightly less than that of sterile water, 57.2%. it means E-51and KH-792 are micro - poisonous to the spores. It's known that, the spore's shell is mainly consist of acetylated chitin [ $\beta$ -(1,4)-2-Acetamido-2-deoxy-D-glucose]. Chemically, it's inert to E-51and KH-792.

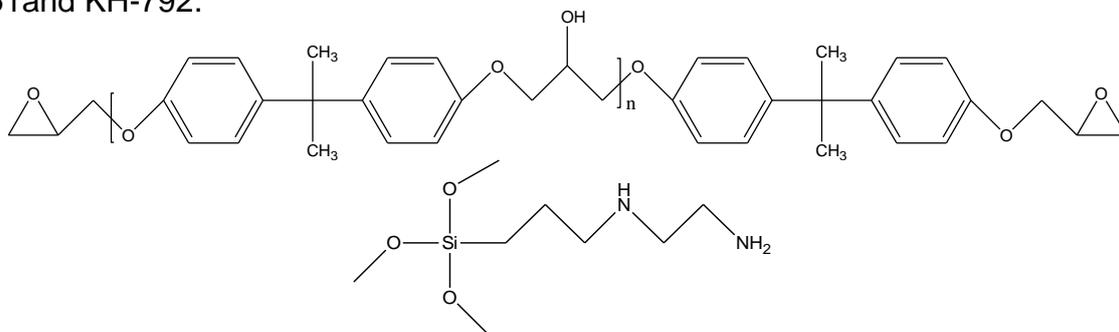


Fig. 1 molecular structure of epoxy E-51 and KH-792

### 3.2 Morphology of microcapsule and shell thickness

The microcapsule has a very good spherical shape, the size is about 600 $\mu\text{m}$  in diameter. The shell is smooth and compact, the thickness is about 75  $\mu\text{m}$ .

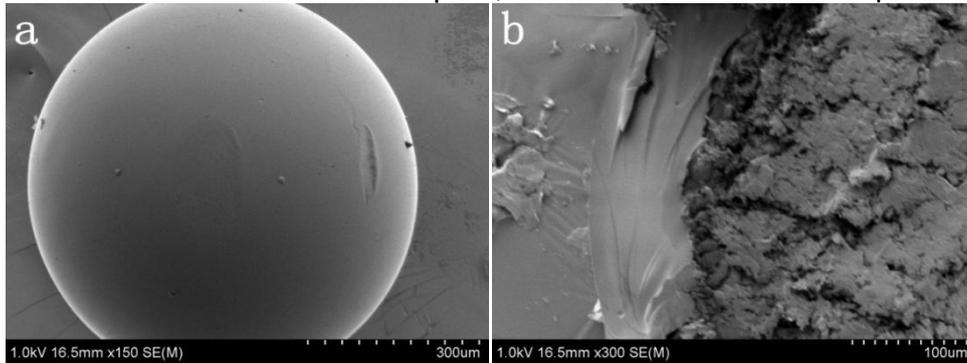


Fig.2 SEM image of (a) microcapsule, (b) shell section

### 3.3 Viability of the microencapsulated spores

The content of spores in bare core is  $7.1 \times 10^9$  cfu/ml, in microcapsule is about  $6.5 \times 10^9$  cfu/ml, it means, after microencapsulation, the fractions of surviving spores is about 91.5%.

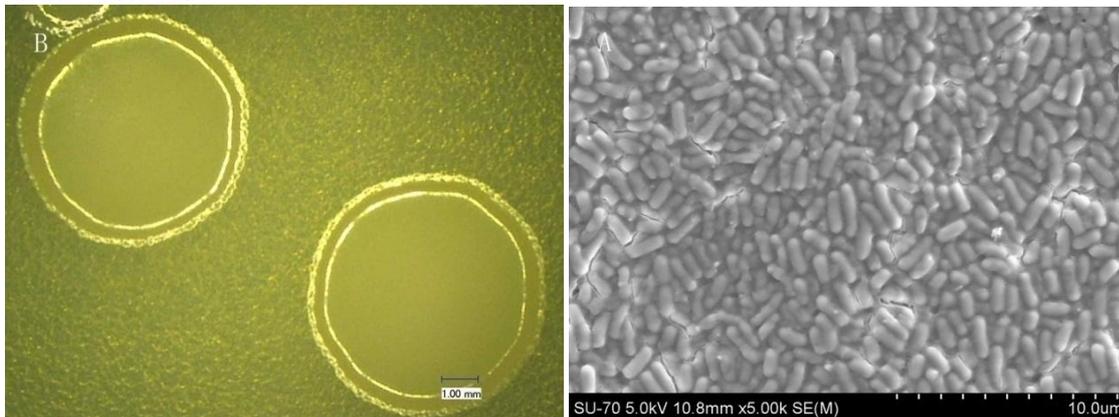


Fig.2 (a)The OM photographs of bacterial colony on the plate after 24hrs cultivation, (b) SEM image of the bacteria in colony

### 3.4 Watertightness

Saoked in water for 10days, the microcapsules does not show any change is shape, size and color. The time was prolonged to 1 month, there is still no change. So it can be concluded the microcapsules have excellent waterproof performance.

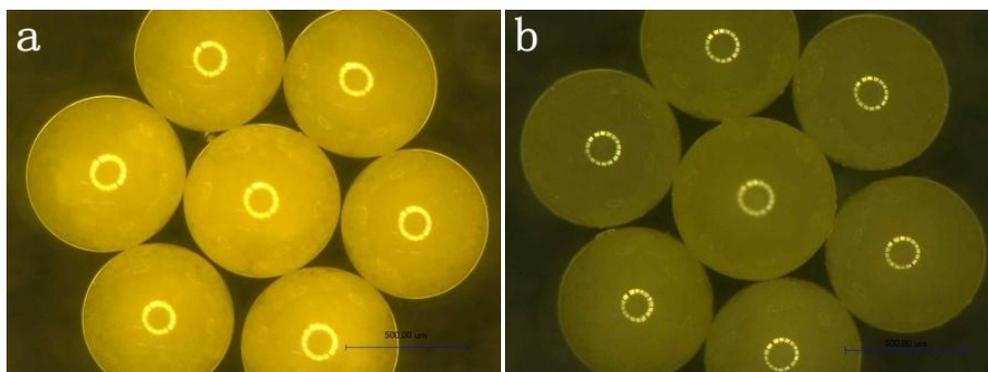


Fig.3 OM photographs of microcapsules (a) before, (b) after soaked in the water for 10days

#### **4. CONCLUSION**

The spores were confirmed to have a chemical tolerance with all the chemicals used in this process. After microencapsulation, the fraction surviving of spores is about 71%, indicating the process is bio-friendly in a general way. Water-permeability testing shows, there are no microcapsule swollen, and no microencapsulated spores germinated after the microcapsules soaked in water over 10 days.

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#### **REFERENCES**

- [1] Sookie S. Bang VR. Microbiologically - Enhanced Crack Remediation [J]. *Industrial Application of Microbial Genomes*, 2001:3-13.
- [2] C J-L, C R-N, G P. Consolidation of degraded ornamental porous limestone stone by calcium carbonate precipitation induced by the microbiota inhabiting the stone[J]. *Chemosphere*, 2007,68(10):1929-1936.
- [3] Wang JY, Soens H, Verstraete W, De Belie N. Self-healing concrete by use of microencapsulated bacterial spores[J]. *Cement and Concrete Research*, 2014,56(2):139-152.
- [4] Hui P, Jinlong Z, Bing L, Xu D, Feng X. Development of microbial self-healing technique in concrete[J]. *concrete*, 2014,298(8):38-48.