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# Quantitative solution of size and dosage of capsules for self-healing of cracks in cementitious composites

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**Abstract.** Self-healing (SH) technology of cracking is becoming a promising solution to improve the durability of cement based composites. However, little formula are available in the literature on determining the size and dosage of the self-healing capsules. Supposed that SH capsules will be broken and activated when they met cracks, a theoretical solution is developed to calculate the appropriate length of SH capsules based on Buffon's needle model. Afterwards, a method to calculate the dosage of capsules was proposed in terms of stereological theory. The reliability of the above mentioned theoretical methods was verified by computer simulation. An experiment of self-healing in mortar was performed as well, by which the theoretical models were verified.

**Keywords:** self-healing; geometrical probability; simulation verification

## 1. Introduction

It is well-known that cracks and local damage may inevitably happen in any kind of material, resulting from the attack of aggressive media in service environment and the existence of intrinsic defects in the material. The initiation and propagation of cracks in materials during service may accelerate deterioration of materials and shorten its service life. To make matter worse, the propagation of cracks may result in catastrophic collapse of the structure if the materials are used as structural element, such as a bridge. Resulting from the analysis based on the performance and cost of materials against service time, materials that have the built-in ability to sense and repair the cracks spontaneously are worth developing (van Breugel 2007). Such technology called self-healing, has been proposed (Dry 1996, Motuku *et al.* 1999) and tested in various fields relevant to materials, such as civil engineering materials (Li and Yang 2007, He and Shi 2009), advanced alloys (Buhua *et al.* 2007), biomaterials (Fratzl and Weinkamer 2007) and polymeric materials (White *et al.* 2001, Pang and Bond 2005).

Concerning self-healing of cracks in cementitious composites, several approaches are found in literatures, such as "Microbiological self-healing technology" (Jonkers and Schlangen 2009, Ramarishnan and Panchalan 2006, van Tittelboom *et al.* 2010, Wang *et al.* 2012), the "Rehydration

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self-healing technology" (Schlangen and Joseph 2008, Huang and Ye 2012, Lv and Chen 2012) and "Adhesive-filled self-healing technology" (Dry and Warner 1997, van Tittelboom *et al.* 2011). In the "Adhesive-filled self-healing" method, the cementitious matrix contains encapsulated repair agent. The adhesive-filled capsules distributed in the matrix will break easily when cracks cross over it, subsequently the adhesive is released and cures in the cracks. Compared with the "Microbiological self-healing technology", this approach has a higher self-healing speed, and is easier to practice. Thus, this paper will concentrate on the solution of some problems regarding "Adhesive-filled self-healing technology".

Although many researchers have tried out various approaches to improve the healing efficiency of cracks (Dry 1994, 2000, Brown *et al.* 2002), and some researchers analyzed the probability characteristics of spherical capsules in concrete (Zemskov *et al.* 2010), little information is available in literature about how to determine a suitable geometry size and dosage of cylindrically self-healing capsules which is more commonly used in cementitious materials and easily to be prepared (Lv and Chen 2011a, 2011b). For the specific crack pattern in the matrix subjected to compressive loading, in this contribution, a theoretical approach is first developed to determine the length of capsules. Then, a method to calculate the dosage of capsule is further derived in this paper. Finally, computer modeling as well as experiments are used to test the reliability of these two models.

## 2. Theoretical model

### 2.1 Determination of capsule length

For concrete subjected to compressive loading, the crack pattern can be simplified as a series of parallel linear cracks (Lusche 1974), which is more easily to be modeled than randomly cracks. Additionally, it is much harder to induce cracks in the concrete subjected to tensile loading while the samples remain uncrushed. In order to start with simplified modeling, this paper will only consider cracks due to compressive loading.

Obviously, the intersection probability between cracks and capsules is related to the crack spacing and the length of the capsules (Kendall and Moran 1963). The famous "Buffon's needle model" can be employed to determine the intersection probability.

Buffon's needle problem was posed by Buffon in 1773. The solution was further reproduced by Buffon in 1777 (Buffon 1777). In Buffon's needle problem (see Fig. 1(a)), a floor is supposed to be covered with a group of parallel lines with equidistance $d$. A short needle with the length $l$ ($l<d$) is randomly dropped onto the floor. The probability $P_S$ of a single needle intersecting with a line can be expressed according to Eq. (1) (Solomon 1978).

$$P_{\mathrm{S}} = \frac{l/2}{\pi d/4} = \frac{2l}{\pi d} \tag{1}$$

We suppose that $d$ is the spacing between two cracks and $l$ is the length of the self-healing capsules. Consequently, for an expected probability of a single capsule crossed by a crack, Eq. (1) can be used to calculate the corresponding length of short capsules at given crack pattern.

However, it should be noted that Eq. (1) requires $l<d$. Thus, the maximum value of $P_{\mathrm{S}}$ is 0.637 in the extreme case of $l$ infinitely close to $d$. Thus no matter how much capsules are added in

matrix, 36.3% of capsules will not break when cracks happen. As longer capsule intersect more likely with cracks, the solution for long capsules ($l \geq d$) also needs to be developed.

If $l \geq d$, single capsules have the possibility to cross over more than one crack. Let $[x]$ represent the integer part of $x$ ($x=l/d$). Then, the possible number of cracks intersecting with a single capsule could be from 0 to $[x]+1$ (Diaconis 1976, Chung 1981). Supposed that the capsule will break if any crack goes through it, the fraction of broken capsules to the total number of capsules is equal to the probability of a single capsule crossed over by at least one crack.

Thus, for the long capsules as shown in Fig. 1(b), if the distance $h$ between one of the endpoints of the capsule and the nearest crack (which is located at the same side of the other endpoint of the capsule), satisfy the inequality (2), at least one crack will cross over the capsule.

$$h < l \sin\theta \tag{2}$$

where, *l is capsule length,* $\theta$ is the orientation of capsule in the interval of [0, π], and the value range of $h$ is $[0, d]$.

Since $h$ can be varied in the range of $[0, d]$, while the range of $\theta$ is [0, π], the area ($S_2$ in Fig. 2) of the region covered by the lines of $h=0$, $h=\mathrm{d}$, $\theta=0$ and $\theta=\pi$ can be considered as the set of all the orientations of capsules. Meanwhile, we can get the intersecting area ($S_1$) between the region under the curve $h=l\sin\theta$ and the region between $h=0$, $h=d$ which indicate the set of orientations of

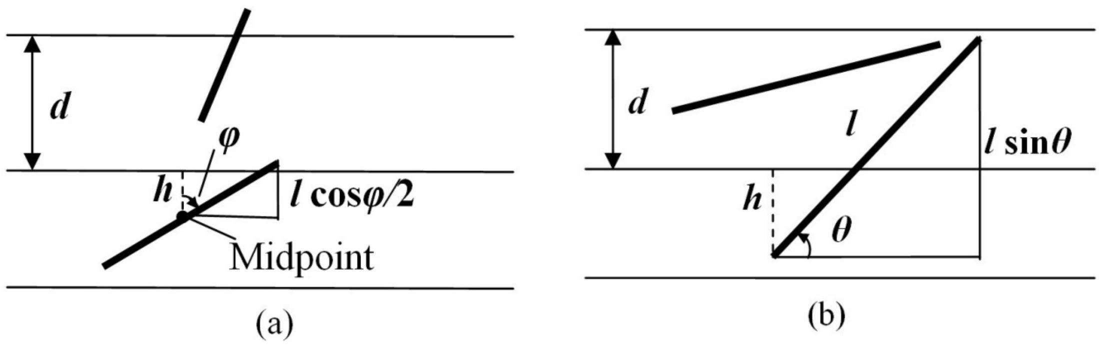

Fig. 1 Buffon's problem in 2D: (a) Short needles ($l<d$) (Solomon 1978) and (b) Long needles ($l \geq d$)

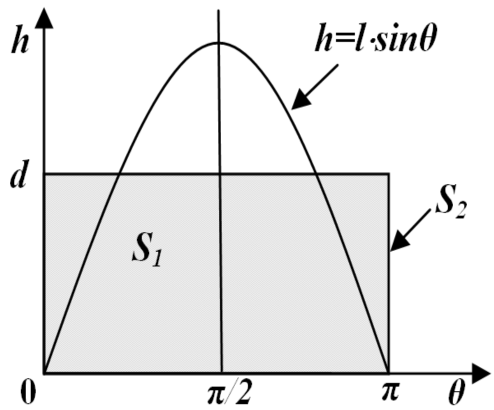

Fig. 2 Curve of ($\theta$, $h$) in Buffon`s problem with long needle

capsules when cracks intersect with capsules. Fig. 2 indicates the probability ($P_L$) of the case ($h<l\sin\theta$) is equal to the ratio of the area of $S_1$ between the area of $S_2$.

$$P_{\mathrm{L}} = \frac{S_1}{S_2} = \frac{2\int_0^d [(\pi/2) - (\arcsin(h/l))]dh}{d \cdot \pi} = \frac{2l + 2d \cdot \arccos(\frac{d}{l}) - 2\sqrt{l^2 - d^2}}{d \cdot \pi} \tag{3}$$

Now, Eqs. (1) and (3) are derived from a 2 dimensional case. Similarly, the model of the relationship between the ratio of capsule length to the crack spacing and the probability of a single capsule crossed by at least one crack can be developed in a 3 dimensional case as follows.

In 3 dimensional cases, the cracks are considered as an array of parallel planes with equidistance $d$. The orientation of the capsules is determined by the spatial angles $\varphi$ and $\theta$ as shown in Fig. 3. The length of the projection $l'$ of the capsules with length $l$ on the plane perpendicular to the cracks can be expressed as

$$l' = l\cos\varphi \tag{4}$$

Obviously, if the projection of any capsule onto the x-y plane intersects with the corresponding projection of a crack, this capsule must be crossed over by the crack in space. Thus, substitution of Eq. (4) into Eqs. (1) and (3) respectively, the probability of a single capsule intersecting with one crack in space could be derived as follows

$$P'_{\mathrm{S}} = \frac{2l\cos\varphi}{\pi d} \tag{5a}$$

$$P'_{\mathrm{L}} = \frac{2l\cos\varphi + 2d \cdot \arccos(\frac{d}{l\cos\varphi}) - 2\sqrt{(l\cos\varphi)^2 - d^2}}{\pi d} \tag{5b}$$

According to Eq. (4), when $\varphi\in$[arcos ($d/l$), $\pi/2$+arcos ($d/l$)], then $l'<d$; when $\varphi \in$[0, arcos ($d/l$)] $\cup$[$\pi/2$+arcos ($d/l$), $\pi$], then $l'\geq d$. Thus, for $l\geq d$, the probability of $l'<d$ and $l'\geq d$ is ($2\pi$-2arccos($d/l$))/$\pi$ and 2arccos ($d/l$)/$\pi$ respectively. Eqs. 5(a) and (b) should be applied in the

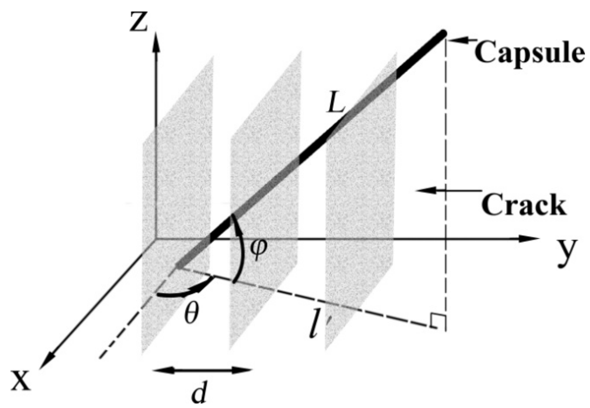

Fig. 3 Buffon`s problem in 3D

corresponding orientation cases. The probability $P$ of a single capsule intersecting with crack in 3D case should be the combination of both of the condition

$$P=\frac{\frac{\pi}{2}-\arccos(\frac{d}{l})}{\pi/2}\int_{\arccos(\frac{d}{l})}^{\frac{\pi}{2}}\cos\varphi P'_{\mathrm{S}}d\varphi+\frac{\arccos(\frac{d}{l})}{\pi/2}\int_{0}^{\arccos(\frac{d}{l})}\cos\varphi P'_{\mathrm{L}}d\varphi \tag{6a}$$

For the case of $l<d$, Eq. (6a) can be simplified to Eq. (6b) because $l'$ is always less than $d$.

$$P=\int_{0}^{\frac{\pi}{2}}\cos\varphi P'_{\mathrm{S}}d\varphi \tag{6b}$$

Combination of Eqs. 5(a) and (b) with Eqs. 6(a) and (b) may derive implicit formulae (i.e., Eqs. 7(a) and (b)) on the capsule length ($l$) for a given crack spacing as well as the expected intersecting probability.

$$\begin{aligned}P=&\frac{2\pi-4arc\cos x}{x\cdot\pi^2}\cdot\int_{arc\cos x}^{\frac{\pi}{2}}\cos^2\varphi d\varphi+\frac{4arc\cos x}{x\cdot\pi^2}\int_{0}^{arc\cos x}[\cos^2\varphi d\varphi+\\&x\cdot\cos\varphi\cdot arc\cos(\frac{x}{\cos\varphi})-\cos\varphi\cdot\sqrt{(\cos\varphi)^2-x^2}\,]d\varphi\end{aligned}\quad \text{for } l\geq d \tag{7a}$$

$$P=\frac{l}{2d}=\frac{1}{2x}\quad\text{for } l<d \tag{7b}$$

where $x=d/l$ .Via numerical analysis method, Fig. 4 was derived, giving the curve between $x$ and intersection probability $P$.

Apparently, it is possible to determine the intersecting probability for a given ratio of capsule length to crack spacing from Fig. 4 and further to derive the capsule length ($l=d/x$) for given crack spacing.

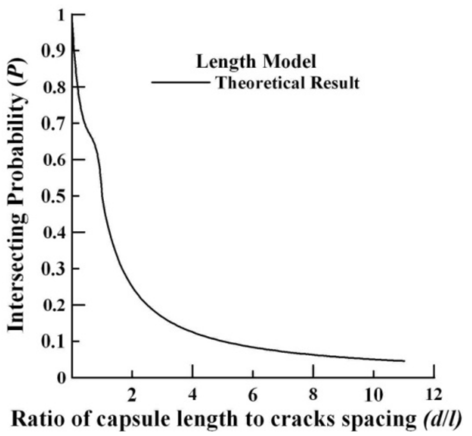


Fig. 4 Theoretical results of length model

Most of the capsules with a length determined by this model, are expected to work at the given crack pattern. Meanwhile, during the experiments indicated in section 4 we found that the capsules with such a length are not too long to be mixed into the matrix uniformly.

### *2.2 Determination of capsule dosage*

Since the strength of the capsules is normally lower than that of the matrix, incorporation of capsules will decrease the strength of the matrix. However, the self-healing efficiency of a single crack is closely related to the amount of capsules crossed by this crack. It is necessary to consider the balance between strength loss of the matrix and self-healing efficiency. In other words, the minimal dosage of capsules with optimal self-healing efficiency should be determined. This contribution will employ the stereological principle to develop a method to calculate the appropriate dosage of self-healing capsules.

According to the stereological knowledge (Underwood 1970), for a random sampling plane *T* going through a representative volume element (RVE) with side length *b* as shown in Fig. 5, the probability *P* of an arbitrary particle being crossed by plane *T* is

$$P = \frac{H}{b} \tag{8}$$

where, *H* represents the projection length of a particle onto the x-y plane.

Suppose that there are *n* particles randomly and uniformly distributed in the RVE, the amount of the particles ($n'$) intersected with an arbitrary sampling plane is

$$n' = nP = n\frac{\overline{H}}{b} \tag{9}$$

where, $\overline{H}$ is the average value of *H*.

Dividing both sides of Eq. (9) by the test plane area $b^2$ yields

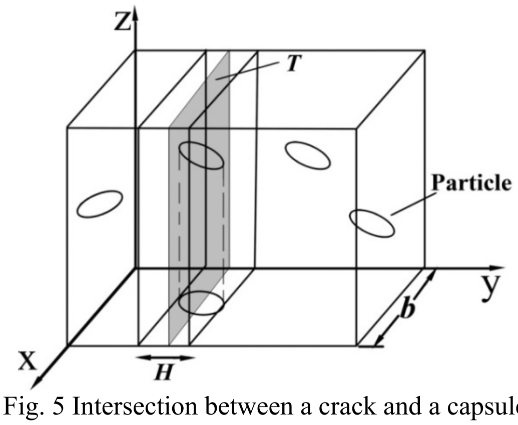


Fig. 5 Intersection between a crack and a capsule

$$\frac{n'}{b^2} = \frac{n}{b^3}\overline{H} \tag{10}$$

Suppose the sampling plane as the crack faces in the matrix, the particle as the capsules, and $H$ as the projection length of capsule $a$ onto the x-y plane, then Eq. (10) can be rewritten as

$$n_A = n_V \overline{H} \tag{11}$$

where, $n_A$ is the amount of capsules intercepted per unit area of crack face, and $n_V$ means the amount of capsules mixed into per unit volume of matrix.

The capsule is normally a cylinder-shaped tube with radius $r$ and length $l$. Suppose that the orientation angle between the capsule and the z-axis is $\varphi$ (see Fig. 6), the projection length of a single capsule onto the x-y plane ($H(\varphi)$) can be expressed as

$$H(\varphi) = l\sin\varphi + 2r\cos\varphi \tag{12}$$

The average projection length (also called caliper diameter) $\overline{H}$ of the capsules along all directions can be derived as

$$\overline{H} = \int_0^{\pi/2} \left[ l\sin\varphi + 2r\cos\varphi \right] \cos\varphi \mathrm{d}\varphi = \frac{1}{2}(l + \pi r) \tag{13}$$

Replace $n_A$ in Eq. (11) by its mathematical expectation $E(n_A)$ and substitute Eq. (13) into Eq. (11). Thus the required minimal amount of capsules to heal all the cracks in the matrix can be expressed as

$$N_V = \frac{E(n_A)}{\frac{1}{2}(l + \pi r)} \tag{14}$$

As the capsule length $l$ is determined by the expected intersecting probability $P$ and the crack spacing $d$ in Section 2.1, we write $l = l(P, d)$. Substitute $l(P, d)$ into Eq. (13) as well as $\overline{H}$ into Eq. (14) at given $E(n_A)$. Thus the appropriate volume fraction $V_V$ of capsules can be expressed as

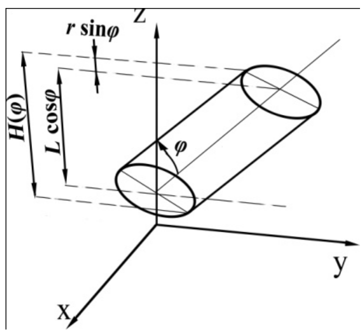


Fig. 6 The caliper diameter ($H(\varphi)$) of a capsule

$$V_V = \frac{n_V v_c}{b^3} = \frac{2E(n_A)\cdot \pi r^2 \cdot l(P,d)}{b^3 \cdot (l(P,d)+\pi r)} \tag{15}$$

where, $v_c$ is the volume of a single capsule, and $b$ is the side length of the RVE.

## 3. Model verification

Before applying the models above, it is necessary to verify their rationality. In this section, the computer modeling technology will be employed in order to validate the models.

### *3.1 Verification of length model*

The algorithm is described as follows:

(1) Generate a cubic container with side length of $b$ as matrix, and a set of crack planes penetrating the container. The crack planes are parallel with the x-z plane with spacing $d$, and the coordinate value $y_i$ along the y-axis indicates the location of the crack $c_i$ ($i$=1, 2, …$m$=[$b/d$])). Then, let $k$ be the amount of capsules crossed by cracks and the initial value of $k$ is equal to zero;
(2) Generate capsules with length $l$. A capsule $\alpha_j$ ($j$=1, 2,…$n$, with $n$ is set by the user) can be determined by the mid-point position ($x_j, y_j, z_j$) and the orientation angles ($\theta_j$, $\varphi_j$), which are randomly generated between the possible intervals. The rationality of the uniform distribution of these random variables were verified. Consequently, the endpoint coordinates of $\alpha_j$ can be calculated.
(3) Judging if capsule $\alpha_j$ intersect with cracks: Compare the coordinate value of the endpoints of capsule $\alpha_j$ with that of crack from $c_0$ to $c_m$. If the coordinate value $y_i$ of crack $c_i$ is located between the $y$ coordinates of the endpoints of capsule $\alpha_j$, it means that capsule $\alpha_j$ intersects with crack $c_i$. If so, stop the current judging process and add 1 to $k$. If no intersection is found between the crack and capsule $\alpha_j$, the value of $k$ is kept unchanged.
(4) When the judging process of capsule $\alpha_n$ is complete, the rate $P$ of all capsules intersecting with cracks can be calculate as

$$P = \frac{k}{n} \tag{16}$$

Obviously, $P$ is the probability of each capsule being crossed by cracks at the given parameters.

According to the probability theory, the value of $P$ tends to be stable with increasing of number of samples. The theoretical results are represented in Fig. 7(a) by means of a dashed line. From Fig. 7(a), it can be found that with various initial parameters, the simulated results are consistent with the theoretical results. Thus Eq. (7a) and Eq. (7b) may be used to obtain the capsule length at a given crack spacing for an arbitrary intersection probability $P$.

### *3.2 Verification of the dosage model*

Similarly, the algorithm is:

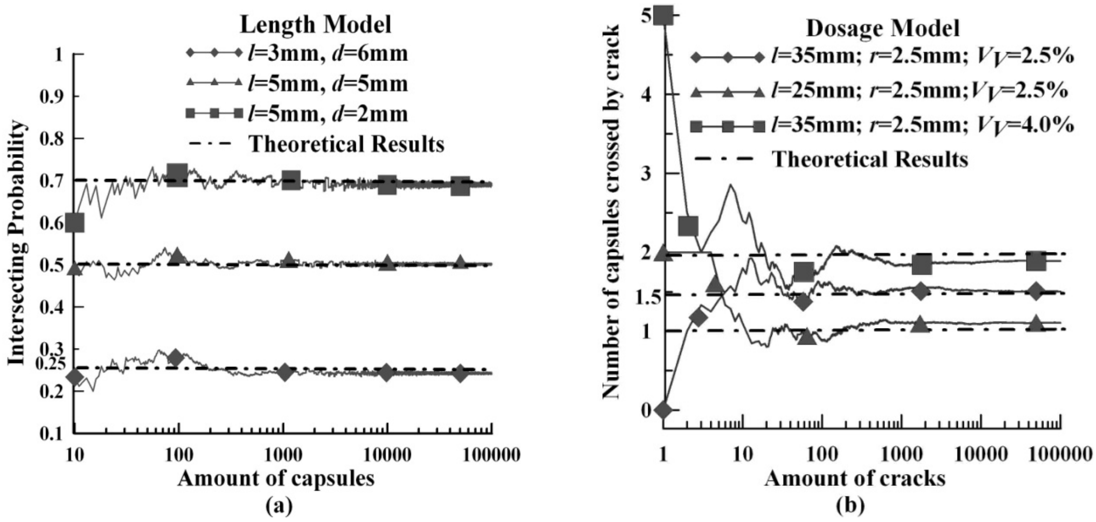


Fig. 7 Simulated results vs. theoretical results: (a) Length model and (b) Dosage model

(1) Generate a cubic container with side length $b$ containing $n$ randomly capsules, as step (1) and step (2) in Section 3.1. The amount of capsules $n$ is determined by the volume dosage of capsules and the volume of the cubic container and the capsules.

(2) Counting the number of capsules which are crossed by a random crack: Randomly generate a crack $c_i$ ($i$=1, 2, …$m$, $m$ is set by the user) which penetrates the cubic container. Then judge if any capsule (from $\alpha_0$ to $\alpha_n$) intersects with crack $c_i$ by the method introduced in Section 3.1. Let $k_i$ represent the amount of capsules crossed by crack $c_i$, and the initial value of $k_i$ is equal to zero.

(3) After generating crack $c_m$ and counting the amount of capsules which are crossed, let $\sum_{i=1}^{m} k_i$ represent the total number of capsules intersected by all the random cracks. Consequently, the mean number of capsules crossed by a single random crack penetrating the mortar matrix is $\sum_{i=1}^{m} k_i / m$.

In dosage models with various initial parameters, it can be found that the simulated results are consistent with the theoretical results indicated by dash lines in Fig. 7(b). Thus Eq. (15) may be used to obtain the capsule volume dosage for a certain number of capsules crossed by each crack.

## 4. Application

The crack pattern and crack spacing in cementitous composites depends on many factors, such as the type of raw materials used, the mixture proportion, the curing temperature and relative humidity type of external loads, the environmental condition during service, etc. In the following experiment, cracks were caused by compressive load. For convenience, the average space between cracks in the mortar specimens is supposed to be 14mm according to the literature (Ostertag and Yi 2007, Lepech and Li 2006, Sezer *et al.* 2008). Setting the expected value of intersecting probability $P$ at 0.8, and the expected number of capsules crossed over by each crack at 1.5, the corresponding length and volume fraction of the capsules are 35 mm and 2.5% based on Eq. (6a)

Table 1 Evaluation of the efficiency of self-healing of cracks in mortar specimens

| Sample | A | B | C | D |
|---|---|---|---|---|
| Length of capsules (mm) | 25 | 35 | 35 | / |
| Capsule volume fraction (%) | 2.5% | 2.5% | 3.5% | / |
| Initial ultimate compressive strength of the sample (MPa) | 33.09±1.16 | 32.83±1.00 | 30.13±1.56 | 34.67±1.01 |
| Final compressive strength of preloaded samples after Self-healing (MPa) | 35.65±0.81 | 37.41±1.22 | 34.37±1.54 | 34.07±0.96 |
| Increase (%) | 7.8 | 13.94 | 14.05 | -1.73 |

and Eq. (15), respectively. Consequently, mortar specimens containing capsules with various combinations of length and dosage will be cast to evaluate the efficiency of self-healing.

### 4.1 Materials

To heal the macro cracks, glass tubes filled with adhesive are widely chosen as self-healing capsule, because the linear expansion coefficient of glass is close to that of concrete (Ostertag and Yi 2007). Additionally, cylindrical glass tubes are easy to manufacture, thus cylindrical glass tubes filled with adhesive have been applied in this experiment. Apparently, the capsules with higher diameter and thinner wall will carry more adhesive agent. But this kind of capsules will decrease the strength of cementitous matrix since more solid portion is replaced while too thick capsules are too hardly to be broken. To meet the balance among the amount of self-healing agent, the amount of fractured capsules during loading and the loss of strength of the matrix, some preparatory tests were taken. Eventually, glass tubes with diameter 5.5 mm and a wall thickness of 0.5 mm and were adopted.

Single-component moisture-curable polyurethane, as a favorable self-healing agent, used in this experiment. It will harden when intersecting with water vapor in the air, and achieve a great strength after being fully cured (Petrie 2007). As a common building adhesive, it was applied in many experiments (Sondari *et al*. 2011, Wang *et al*. 2012) as well.

### 4.2 Methods and results

Portland cement mortar cubes containing capsules filled with healing agent, with various combinations of length as well as volume fraction of capsule were prepared. The different test series are shown in Table 1, specimens with code “D” were control samples without capsules. In order to distribute the adhesive-filled capsules in the matrix randomly and uniformly, the paste without capsules were mixed up by standard method firstly. Then the capsules were mixed into the fresh paste by hands in order to avoid the capsules being broken by blender. The mix proportion of the mortar matrix was the following: cement: sand: water: polypropylene fiber = 1:3:0.5:0.01 (by mass). The dimension of the specimens was 100 mm × 100 mm × 100 mm. The specimens were steam cured at 75℃ for 6 days to make the strength fully developed. For each test set, two groups of samples were cast and in each group 6 samples were prepared. Group 1 was used to obtain the initial ultimate compressive strength of the sample. The results are given in Table 1. Group 2 was used to evaluate the efficiency of self-healing of the capsule. For Group 2, each specimen was loaded up to 70% of its corresponding initial ultimate compressive strength to cause irreversible

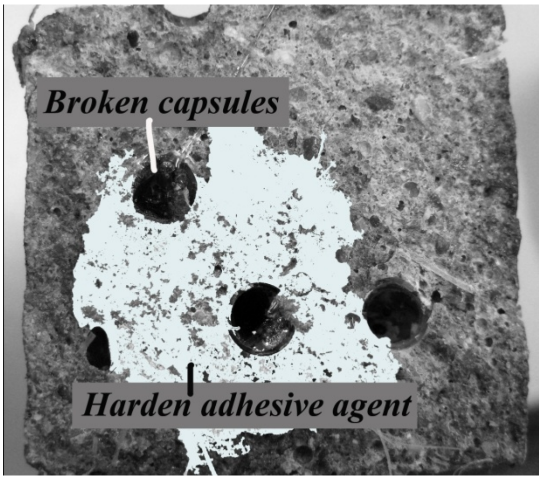

Fig. 8 Broken capsules and harden adhesive agent in the specimens after self-healing

continuous cracks in the matrix (Metha and Monteiro 2006). Then, the load was removed and specimens were allowed to rest for one day so that the healing-agent may harden. Finally, the compressive strength of these preloaded specimens was tested again and the results are given in Table 1.

### *4.3 Discussion*

After self-healing, the specimens containing the capsules were broken along the visible cracks. The broken capsules and harden adhesive agent were observed as shown in Fig. 8. A comparison of the initial ultimate compressive strength of the samples with the strength of preloaded samples after seal-healing (Table 1) revealed several important facts: (1) Since the cracks were sealed by the adhesive, the ultimate compressive strength of preloaded specimens which contained capsules increased after the healing agent released, while it was not the case for the control samples; (2) The self-healing efficiency of test series “B” cotaining longer capsules were higher than that of test series “A” containing shorter capsules, which may be attributed to the higher intersecting probability of longer capsules with cracks; (3) The self-healing efficiency of test series “C” was close to that of test series “B”, but the initial strength of the sample significantly decreased due to the negative influence of a large amount of capsules. It may be deduced that when the volume fraction of capsules in mortar reaches 2.5%, almost all of the cracks can be repaired.

## 5. Conclusions

“Adhesive-filled self-healing technology” is a method which can give materials the ability to heal cracks automatically, but there was a lack of theoretical foundation of the determination of length and dosage of self-healing capsules. To solve the problems, two theoretical models were proposed，and the reliability of these two models were validated by computer simulation as well as experiments.

Based on “Buffon`s needle model”, this paper derived a statistical model which may be employed to determine the length of capsule for a given crack pattern. And further, another model was proposed in terms of a stereological principal to calculate the proper dosage of capsules. The reliability of these two models was verified via computer simulation technology. Moreover, glass tubes filled with a single-component moisture-curable polyurethane were employed as self-healing capsules. Mortar specimens containing self-healing capsules, whose length and dosage were determined by the theoretical models, were prepared and the self-healing effect was validated by experiment based on the compressive strength recovery.

For now, the proper diameter of capsules, which should be determined by the required amount of self-healing agent, was not analyzed. This parameter cannot be calculated only if more factors, such as the width of cracks, the strength and flow properties of adhesive agent, are considered. In this article, the cementitious composites are simplified as a homogeneous material, with randomly distributed capsules and parallel crack patterns. In more complex models, such as concrete, the distribution of capsules will be affected by the aggregate and the cracks pattern will be more complex. Thus, further comprehensive investigation of these cases should be carried out. In addition, the intersection situation between capsules and cracks in matrix should be verified directly by experiment during further studies.

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