2018

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Recommended Citation

Xu, S.; Tabaković, A; Liu, X and Schlangen, E., 2018, "Calcium alginate capsules encapsulating rejuvenator as healing system for asphalt mastic". Journal of Construction and Building Materials, 169, pp. 379 – 387. doi.org/10.1016/j.conbuildmat.2018.01.046

Calcium Alginate Capsules Encapsulating Rejuvenator as Healing System for Asphalt Mastic

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Abstract:

Researchers have demonstrated that the rejuvenator encapsulation method is a promising autonomic self-healing approach for asphalt pavements, where by the self-healing system improves the healing capacity of an asphalt pavement mix. However, potentially high environmental risk via leaching of hazardous chemicals such as melamine formaldehyde renders the technology unsuitable for widespread use in road design. This paper explores the potential for the use of more environmentally friendly and economically viable rejuvenator encapsulation method, where the calcium alginate is used as rejuvenator encapsulation material. The capsule morphology and microstructure were studied using the Microscopy and X-ray tomography. Capsules thermal and mechanical strength were investigated using the Thermogravimetric analysis (TGA) and micro-compressive tests. The results demonstrated that the capsules have sufficient thermal and mechanical strength to survive the asphalt production process. The healing efficiency of the system was evaluated by embedment of calcium-alginate capsules encapsulating rejuvenator in an asphalt mastic beams and subjected to monotonic three-point bend (3PB) loading and healing programme. The results illustrated that the calcium-alginate capsules encapsulating rejuvenator can significantly improve healing performance of the asphalt mastic mix.

Key Words: Self-healing, Asphalt, Rejuvenator, Calcium-alginate, Capsules

1. Introduction

Asphalt mixture have intrinsic healing potential to repair the damage (close cracks), restore its stiffness and strength when subjected to rest periods. Although the self-healing of asphalt has been proven in bitumen, asphalt mortar, asphalt mastic and asphalt concrete, this self-healing capacity is deteriorated by ageing of the bitumen and low ambient temperatures [1,2].Thus, it is a challenge for asphalt pavement engineers to improve asphalt pavement design to increase the self-healing capacity of asphalt pavement.

With the objective of increasing the self-healing capacity in asphalt, extrinsic healing methods [3] have been investigated, which can be concluded into two ways: induction healing and embedded capsules encapsulating asphalt binder rejuvenator. The concept of induction healing is to mix conductive particles inside the asphalt mixture and generate induction heating from outer alternating electromagnetic fields [4-7]. Using the induction healing, the temperature of asphalt mixture can be increased to soften the bitumen within asphalt mix allowing it to flow, close the cracks and repair the damage. Induction heating proved to be a very effective method for asphalt crack healing, but the increasing of temperature also accelerates ageing of the asphalt binder.

In order to address the issues of the asphalt ageing presented by induction healing approach, researchers studied different methods of encapsulation of the bitumen rejuvenator self-healing system. The concept of embedding the capsules which contain binder rejuvenator is to deliver healing agent (rejuvenator) to the damage site and rejuvenate the aged binder, by allowing the rejuvenator to diffuse into the aged binder and soften it, allowing it to flow and in turn close the crack and repair the damage. The healing agents used for asphalt healing includes vegetable oil, waste cooking oil and bitumen rejuvenator [8-10].

There are various rejuvenator encapsulation methods, such as:

1. Melamine-formaldehyde(MMF) modified capsules, Su [11] used MMF modified by methanol to encapsulate rejuvenator. Controlled by stirring rates, the prepared capsules have the mean size from 100.5 to 2.0 um. The microcapsules had survived in bitumen under temperature of 200℃, which indicates that these microcapsules can resist the thermal effect of bitumen in application. Microcapsules had the elastic-plastic deformation ability resisting the temperature changes and mixing stress. However, this encapsulation technology presents a potential environmental problem, where material used in production of the capsules: ‘formaldehyde’ in high concentration can be dangerous for human health.
2. Epoxy capsules, a series of capsules were successfully prepared by García et al [12]. These capsules comprise a porous sand which absorbs the rejuvenator, the sand granules are bound together and coated by a hard shell made of an epoxy-cement matrix with a volume percentage of 20.9, 13.1, 24.9 and 13.0% of rejuvenator, porous sand, cement and epoxy, respectively. The capsules obtained have a mean size of 1.60mm. The capsules are embedded into the asphalt mix by substituting a part of the aggregates in asphalt concrete by the capsules. The working principle of the system is, when the stress in capsules embedded in the asphalt reaches a certain threshold value, the capsules break and rejuvenator is released. These capsules are strong enough to survive the mixing and compaction, but the breaking mechanism is not clear and difficult to control.
3. Xue et al [13] prepared microcapsules by in-situ polymerization method with water, urea, formaldehyde, asphalt rejuvenator, emulsifier and modifier. The morphology,

particle size, coating rate, thermal stability and molecular structure of the microcapsules were investigated. The healing capacity of these microcapsules were evaluated by ductility test and asphalt fatigue test.

Results indicate that the microcapsules could survive during the asphalt melting process and showed good healing performance under conditions of low-temperature and fatigue load. While low temperature behavior and fatigue behavior on asphalt binder are not sufficient to evaluate the healing effect of these microcapsules, more evidences are needed.

4. Compartmented Alginate Fibres, Except capsules, compartmented fibres could also be used to encapsulate healing agent for self-healing purpose. This concept was first proposed to provide local healing with liquid healing agents in fibre reinforced polymer composites [14]. Followed this concept, Tabaković et al [15] used alginate as a rejuvenator encapsulating material and successfully prepared compartmented fibres to encapsulate rejuvenator. The prepared fibres were tested in both thermal and mechanical properties, and the results turned out to prove that the compartmented fibres could survive from the mixing and compaction process of asphalt. Tabakovic et al [15] also showed that the inclusion of the fibres into asphalt mastic mix increased the strength of the asphalt mastic mixture, and these alginate fibres were capable of healing local micro cracks when the asphalt mastic mixtures sustained low level of damage. However, the research showed that this self-healing system can only repair small micro-cracks and the content of rejuvenator is very limited. However, alginate proves to be a very positive material for rejuvenator encapsulation.

Alginate is a long, negatively charged molecule. Positively charged sodium ions (Na^+) dissociate from the alginate when dissolved in liquid solution. Doubly charged calcium ions (Ca^{2+}) can bind two different alginate strands simultaneously, thereby crosslinking and solidifying the solution [16]. **Fig. 1** shows the reaction between sodium alginate and calcium to encapsulate rejuvenator.

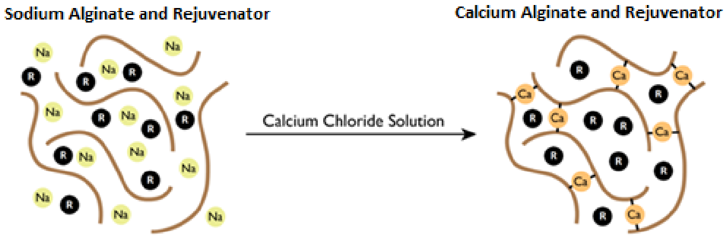

Fig. 1. Encapsulation of rejuvenator with calcium alginate crosslinking

Alginates can be found in brown algae and also in metabolic products of bacteria, e.g. pseudomonas and azotobacter. Nowadays, alginate hydrogels have been particularly attractive in wound healing, drug delivery, and tissue engineering applications to date, as these gels retain structural similarity to the extracellular matrices in tissues and can be manipulated to play several critical roles [17-19].

With the advantages of low cost and environmental friendly, alginate also has the ability of self-degrading when exposed to ambient conditions (air), this property serves as secondary self healing triggering mechanism, i.e. if capsule is not opened by the propagating crack, the self deterioration will open the capsule and release encapsulated rejuvenator. As such the key objective of this research is to investigate the potential use of calcium alginate capsule as rejuvenator encapsulating and delivery mechanism for asphalt pavement materials. In this research, the calcium alginate capsules encapsulating bitumen rejuvenator have been produced. Thermal stability and mechanical property of the capsules are investigated employing the thermogravimetric analysis (TGA) and micro compression testing. The healing performance of the calcium-alginate capsules encapsulating rejuvenator self healing concept was further tested by embedding the capsuled in asphalt mastic mix. Photography and tomography are used for the structural and volumetric study of the capsules.

2. Experimental method

2.1 Materials and preparation

2.1.1 Preparation of calcium-alginate capsules

The calcium alginate capsules, were produced from an emulsion of rejuvenator suspended solution of sodium alginate. To this aim, 6 wt.% sodium alginate in de-ionized was prepared. At the same time a 2.5 wt.% solution of poly(ethylene-alt-maleic-anhydride) (PEMA) was mixed with the rejuvenator with ratio of 40% PEMA and 60% healing agent, forming a healing agent solution. After that, the sodium alginate solution and healing agent solution was mixed by the alginate/rejuvenator ration of 30/70 for 30s at the stirring rate of 100 rpm. To remove air bubbles, the blend was processed in an vacuum environment for 30 min. Subsequently, the blend was pumped through a needle and the capsule beads were dropped into the $CaCl_2$ solution. Finally, the calcium alginate capsules can be acquired after drying in oven. **Fig. 2** shows the production process of the calcium alginate capsules. **Fig. 3a** illustrates the chemical structures of PEMA, the average molecular weight of PEMA is 100,000 to 500,000. **Fig. 3b** illustrates the schemes of the encapsulation process with alginate and PEMA.

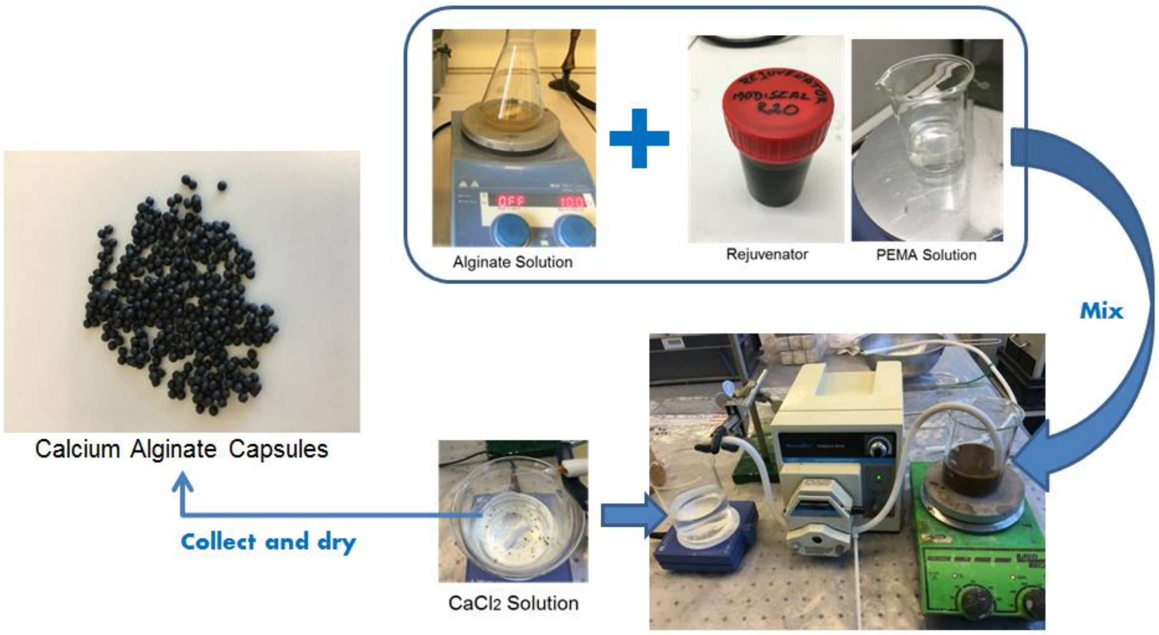

Fig. 2. Preparation process of calcium alginate capsules

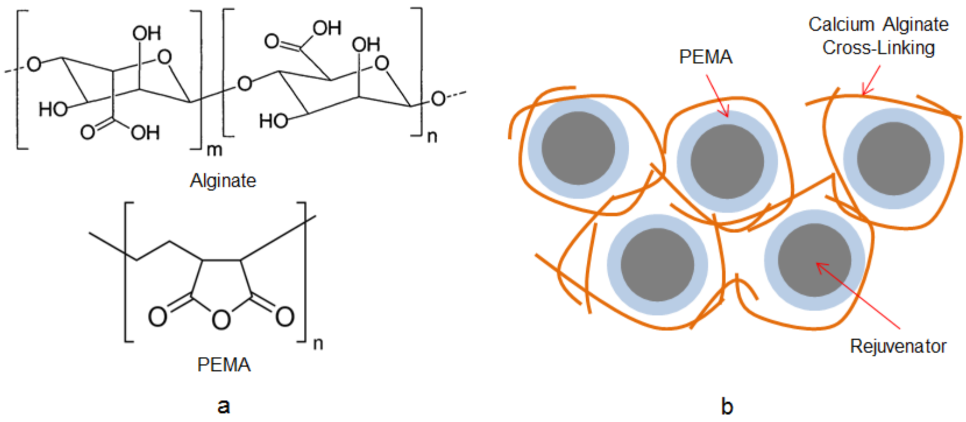

Fig. 3. (a) The chemical structures of alginate and PEMA and (b) schemes of the encapsulation process

Industrial rejuvenator R20 supplied by Latexfalt, The Netherlands was used as healing agent in this research. Other chemicals used in the process were purchased from Sigma Aldrich, The Netherlands.

2.1.2 Asphalt mastic mix design and mixing procedure

Asphalt mastic beams were prepared in order to evaluate the healing efficiency of calcium alginate capsules. These mastic beams were prepared containing three different proportions of the calcium alginate capsules (**Table 1**), including: control beams (without capsules), beams with 2 wt% capsules and beams with 4 wt% capsules. The capsules were inserted into the asphalt mastic mix design by replacing the bitumen content of the mix.

Table 1 Mix composition of asphalt mastic beams

Mix constituent	Percentage by weight		
	Without Capsules	2% Capsules	4% Capsules
Sand(0~4mm)	50	50	50
Filler(Wigro60k)	25	25	25
Bitumen(70/100)	25	23	21
Capsules	0	2	4

The asphalt mastic mix was prepared using a 5l Hobart mixer. Prior to mixing, all mix constituents were preheated to 160ºC for 2 hours. During the mixing process sand, filler and bitumen were mixed first, capsules were gradually added to the mix in order to avoid conglomeration of capsules within the mix. In order to evaluate the ageing effect on the capsules, the mastic mixture was aged following the aging programme designed by Kliewer et al [20] and used by Tabaković et al [15]. **Table 2** shows the ageing programme with three ageing protocols.

Table 2 Ageing programme

Ageing protocols	Curing time and temperature	Ageing level
No ageing	0	None
Short term ageing	135°C 4h	Simulation of 4 years ageing

Long term ageing	135°C 4h+85°C 4days	Simulation of 15 years ageing

The beam test specimens in dimensions of 125×25×15mm were compacted using a silicon mold as shown in **Fig. 4**. In order to achieve controlled crack propagation each beam specimen contained a ‘v’ notch at the center of the beam, as shown in **Fig. 4b**.

Fig. 4. (a) Mold for asphalt mastic beams and (b) the prepared sample.

2.2 Characterization of capsules

2.2.1 Microscopy

A Leica 2500P polarised light microscope was used to observe the morphology of calcium alginate capsules. **Error! Reference source not found.**shows tests sample used in the optical microscope analysis. In order to evaluate the microstructure inside the capsule, several capsules were fixed in epoxy (Fig. 5a), and then polished until the cross sections of the capsules were reached (**Fig. 5b**).

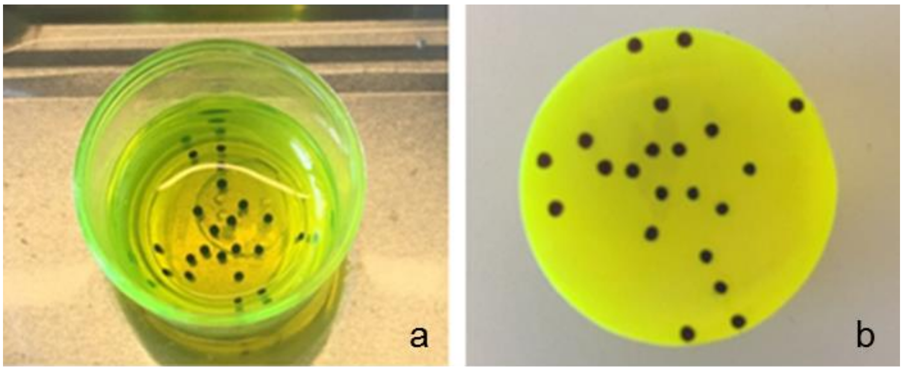

Fig. 5. (a) Capsules fixed in epoxy and (b) polished to cross section.

2.2.2 X-ray computed tomography (X-CT)

A Phoenix Nanotom CT scanner was employed in order to study structural and volumetric composition of the calcium alginate capsule. A single capsule was rotated along their longitudinal axis and three x-ray attenuation images were recorded every 0.25°. To fit the lateral dimension of the capsule volume during the scan, the resolution was set as 1.25 μm^3 per voxel [21]. After scanning, the image reconstruction was performed with Phoenix Datos|x software and images from the top slices view were analyzed to quantify the rejuvenator composition of capsules.

In an X-CT image, individual phases containing different brightness intensities can be segmented by grey level thresholding. The grey level histogram is composed of separate peaks corresponding to distinct phases with heights proportional to the relative fractions of each phase. In this research, within the area of a capsule, the grey level histogram was composed with two phases: rejuvenator and calcium-alginate.

A randomly framed area (400×400 pixels) within the capsule was selected and a feature segmentation algorithm was employed to analyze the images [21]. Two different phases in the framed area can be quantified by cumulating pixels of each phase. To increase the accuracy, ten images from top slices were included in the analysis and the average value of grey level distribution was calculated.

2.2.3 Thermogravimetric analysis

The thermal stability of calcium capsules was evaluated with NETZSCH STA 449 F3 Jupiter TGA system. The analysis was conducted using the environment of argon gas (Ar) at flow of 50 ml/min. The scanning programme started at 40°C and increased at rate of 5°C/min until 160°C; then hold on 160°C for 20min. The mass changes within this time period were recorded.

2.2.4 Compressive test on calcium alginate capsules

The micro tensile strength testing machine (TSTM) developed by Microlab, was used to investigated compressive resistance of the calcium alginate capsules. The tests were performed at loading speed of 0.01 mm/s and ambient temperature of 20±2°C. Micro tensile strength testing system is presented in the **Fig. 6**. In order to analyze the deformation of capsule during the compressive, the whole testing process was recorded by a video camera from the vertical view. In order to investigate multi-temperature effect on the mechanical performance, capsules were pre-conditioned for 15 min at 10 different temperatures (every 20°C from -20°C to 160°C). Minimum five capsules were tested for each temperature condition.

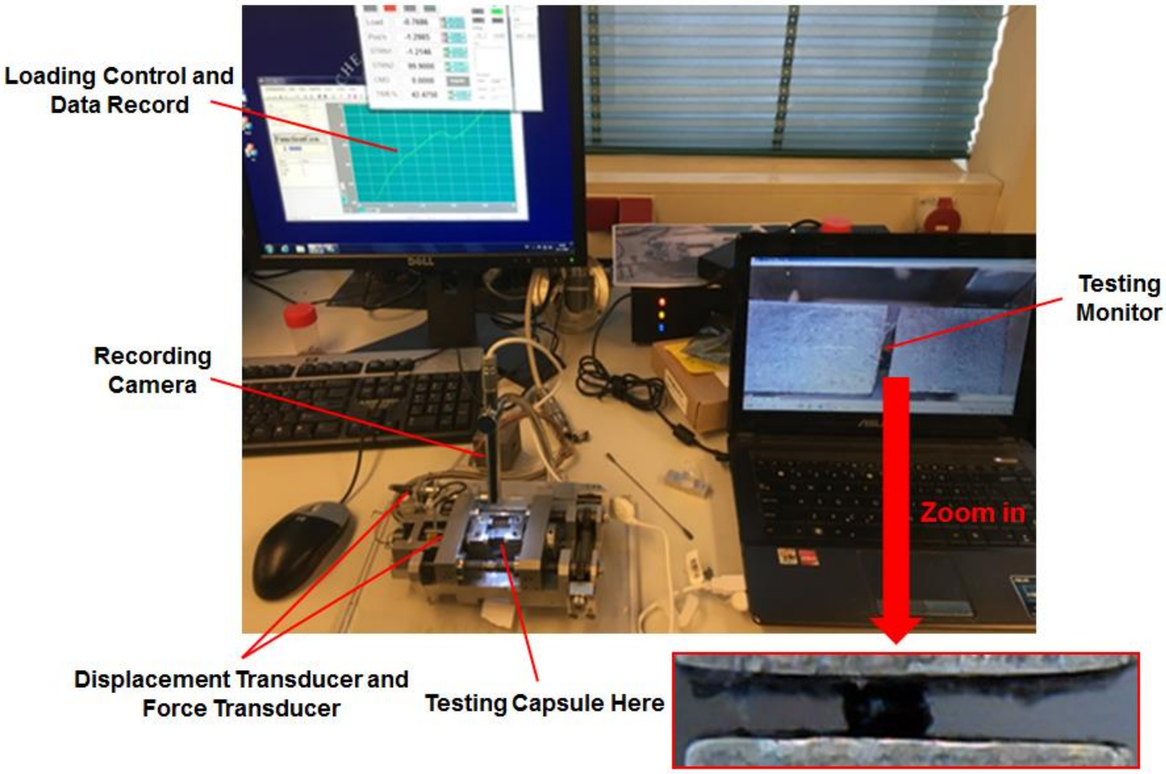

Fig. 6. Capsule compressive test setup

In this research, the yield strength was used to evaluate the compressive strength of capsules. In the stress-strain curve of a compression test, the linear region terminates at the yield point and above this point the capsule behaves plastically and the deformation will not be able to recover once the load is removed. At that moment, the compressive stress began to create permanent deformation of a capsule and accompanied with a risk of rejuvenator leaking out of capsule.

2.3 3PB test and the healing efficiency of the asphalt mastic

The 3PB test was used to determine the healing efficiency of the calcium alginate capsules. An Universal Testing Machine (UTM) with temperature chamber was employed to perform the 3PB tests. **Fig. 7** shows the experimental setup and the parameters. The 3PB tests were performed at the loading speed of 0.01mm/s under -5°C to avoid permanent deformation and to create a brittle fracture in the sample. During the 3PB tests, stress concentration on the notch allows the initiation and propagation of crack through the middle of the specimen, testing the healing function of capsules in the case of cracking.

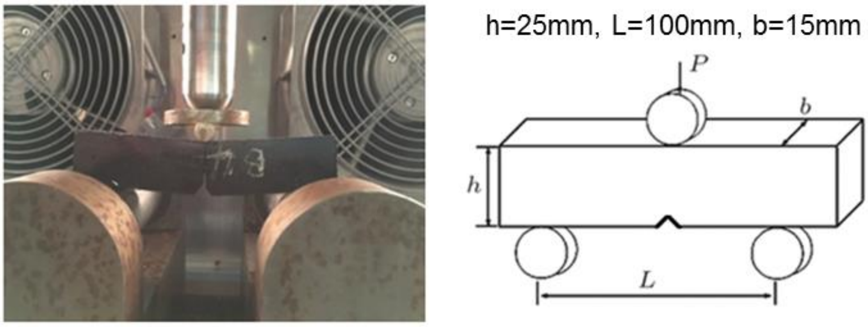

Fig. 7. 3-point-bending testing setup and parameters

To investigate the healing efficiency of the calcium-alginate capsules within the asphalt mastic mix, a testing and healing programme showed in **Fig. 8a** was designed. Firstly, a 3PB test was performed to allow crack formation in beam (**Fig. 8b**). After the test, the cracked sample was healed for 4 hours at an ambient temperature of 20±2^{o}C and followed by a second 3PB test to acquire the bending strength after first healing stage. Subsequently, the sample was healed again for 12 hours at ambient temperature of 20±2^{o}C and followed by a third 3PB test to acquire the bending strength after second healing stage. Since the confining stress on cracking surfaces plays an important role during the asphalt healing process [**Error! Bookmark not defined.**], the cracked specimens were placed in the compaction mold to achieve constant healing condition for all specimens **Fig. 8c**. The healing efficiency was characterized by the Healing Index (HI), which was calculated using the following approach:

$$\mathrm{HI} = \frac{C_x}{C_1}$$

Where:

HI=the healing index (%),

C_1=original strength of the sample;

C_x=strength after x cycles of healing.

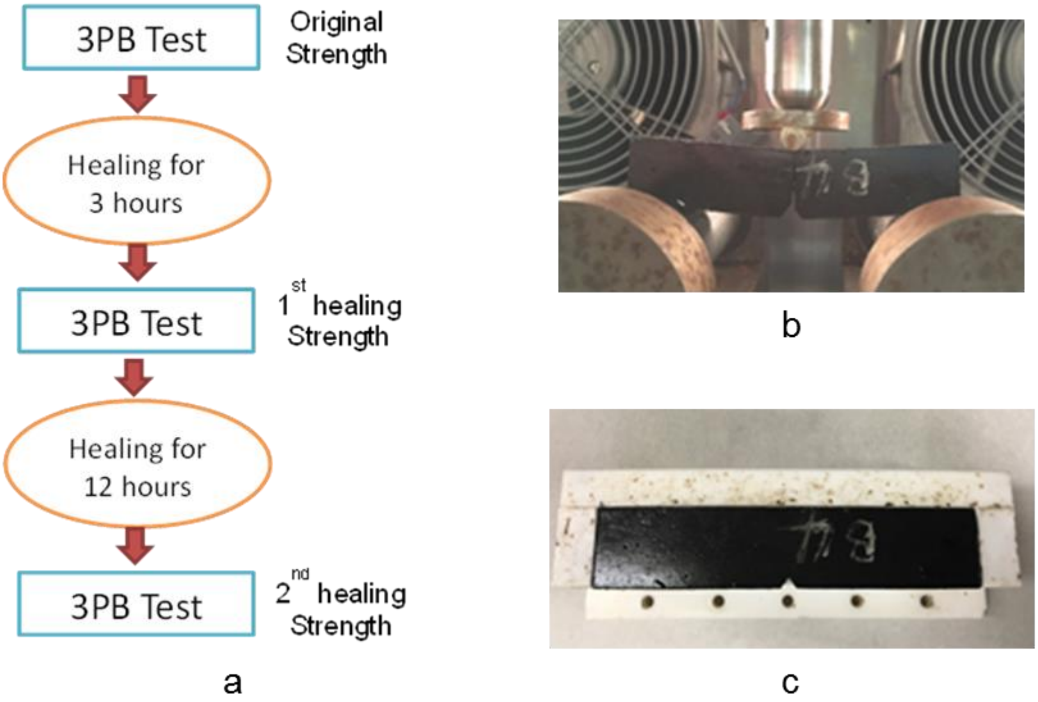

Fig. 8. Asphalt mastic beam: (a) testing and healing programme, (b) 3PB testing setup and (c) Healing in mold.

3. Results and Discussions

3.1 Capsules morphology

Fig. 9a shows the encapsulated rejuvenator makes the capsule presented in dark color, and these capsules have a uniform diameter of 1.95 mm. The cross-sectional image (**Fig. 9b**) indicates the calcium alginate capsule has a porous structure instead of a traditional core-shell structure. **Fig. 9b** shows that the capsule was surrounded by a dense layer of calcium-alginate crosslinking shell, and small rejuvenator droplets were located and encapsulated by porous media within the shell. This structure demonstrates cracking of capsules and reaching the porous media allows the leaking out of rejuvenator. A crashed capsule under microscope and rejuvenator release was shown in **Fig. 9c**.

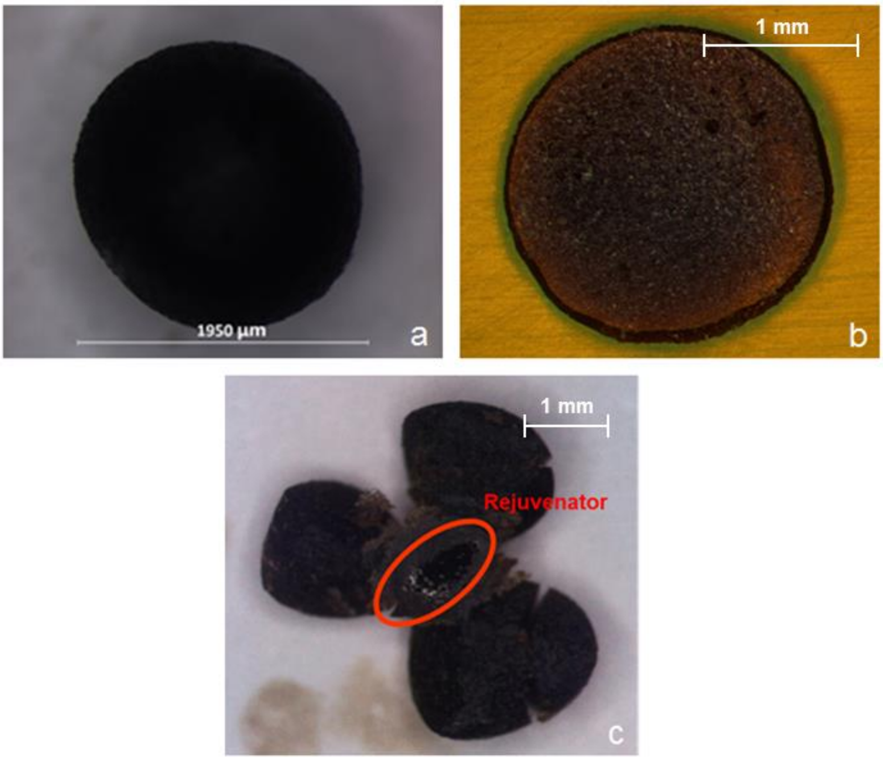

Fig. 9. Microscopic image of calcium alginate capsule: (a) general view, (b) cross sectional view and (c) crashed capsule.

An advantage of this cross-linked structure is to provide a structural reinforcement to allow capsules survive high temperature and pressure during the mixing and compaction process, also allows the capsules sustain the cyclic loading in long term service until triggered by micro-cracks. Meanwhile, random distribution of the cross-linked structure in the capsule could create compartmented rejuvenator encapsulations. In this way, when a crack reaches a capsule will not result in full rejuvenator release, indicating that capsules could provide multi-crack healing and long term healing.

3.2 X-ray tomography

The grey value distribution depending on voxel numbers and the segmented area are shown in **Fig. 10**. Only one peak can be found in the voxels grey value distribution curve, this peak is regarded as a result of superposition from two phases. In the area segmented curve, the maximum slope is located at the grey level of T=150, which indicates a dramatic change between phases. Hence, T is recognized as the boundary between the two phases.

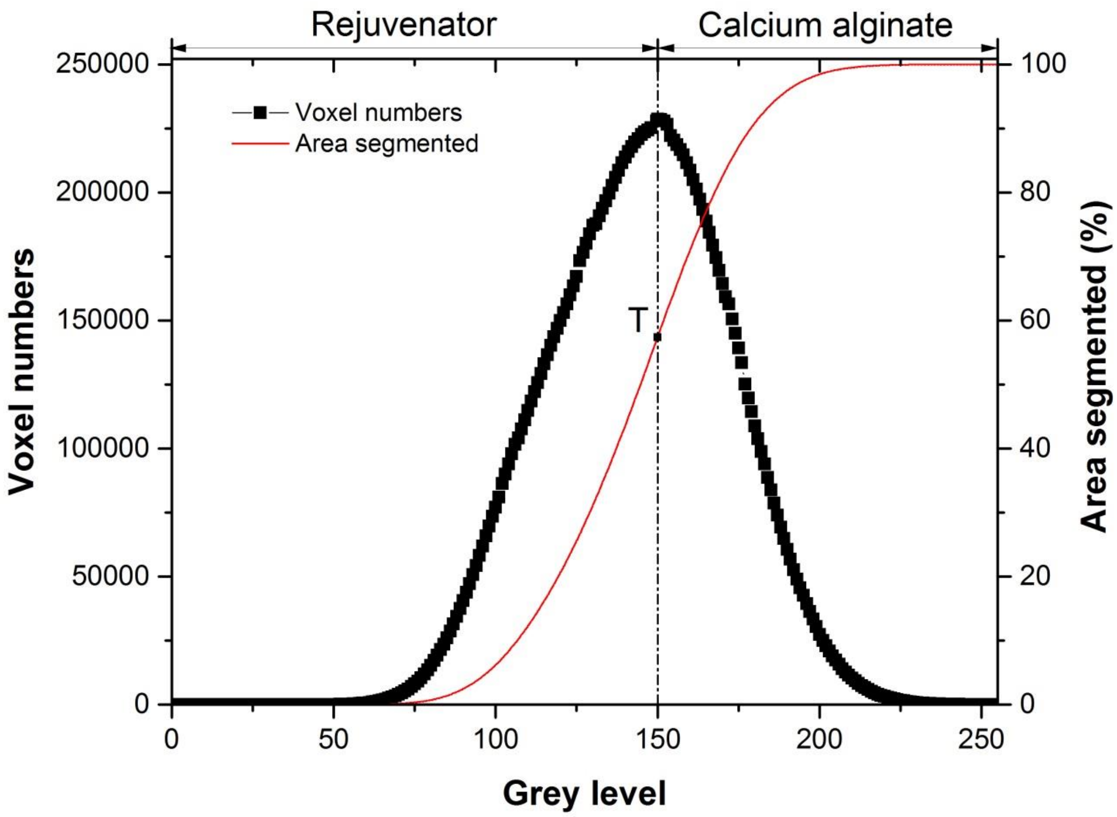

Fig. 10. Phase evaluation through grey level histogram of CT images

An x-ray tomography image is shown in **Fig. 11**. The image illustrates calcium alginate crosslinking, shown as a brighter color in the image, which means its grey level is distributed in higher values than rejuvenator. When applied with boundary T, the voxels with a grey level smaller than T is regarded as rejuvenator and the voxels with a grey level greater than T is regarded as calcium alginate. As shown in **Fig. 11**, for a better view of segmentation in phases, a framed area can be processed to an image which two different phases are highlighted: rejuvenator in black and calcium alginate in white.

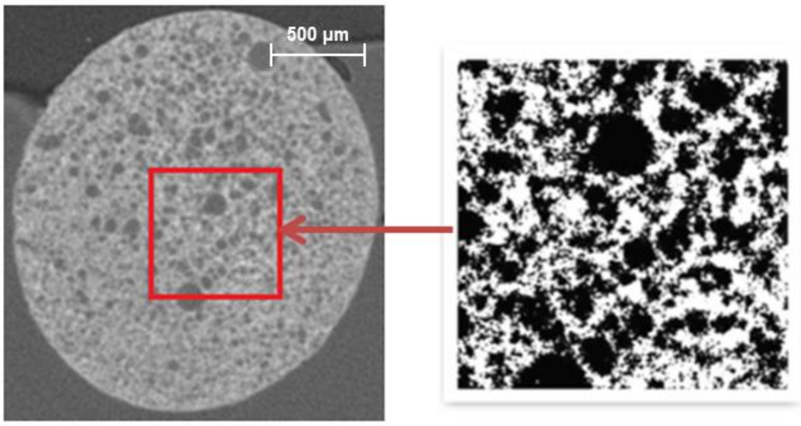

Fig. 11. (left) X-ray tomography image and (right) image of area segmentation.

Based on the grey level histogram analysis from 10 different CT images, the rejuvenator phase content of the capsule can be calculated and the result turns out to be 56% by volume. The x-ray tomography image indicates the porous structure inside a capsule, which supports the conclusion from the cross section microscopy. Further advantage of the calcium alginate capsules healing system is the 56% by volume rejuvenator content, which results in increased healing efficiency of the calcium-alginate encapsulating rejuvenator asphalt self-healing system in comparison to other rejuvenator encapsulating healing systems.

3.3 Thermogravimetric analysis

The thermogravimetric analysis results for the capsules are shown in **Fig. 12**. The results show when the temperature is below 100°C, the capsules are very stable and recorded loss is less than 1% of their weight. After 100°C, weight of capsules decreases gradually with increasing of temperature. This weight loss corresponds to the residual water evaporation from the calcium alginate. When temperature reaches at 160°C (referred as the asphalt mixing temperature), the total weight loss of capsules is 3.8%. It indicates that except dehydration, there is no further degradation on capsules under 160°C. This finding indicates that the calcium alginate capsules are capable of surviving the asphalt mixing temperature of 140^{o}C – 160^{o}C.

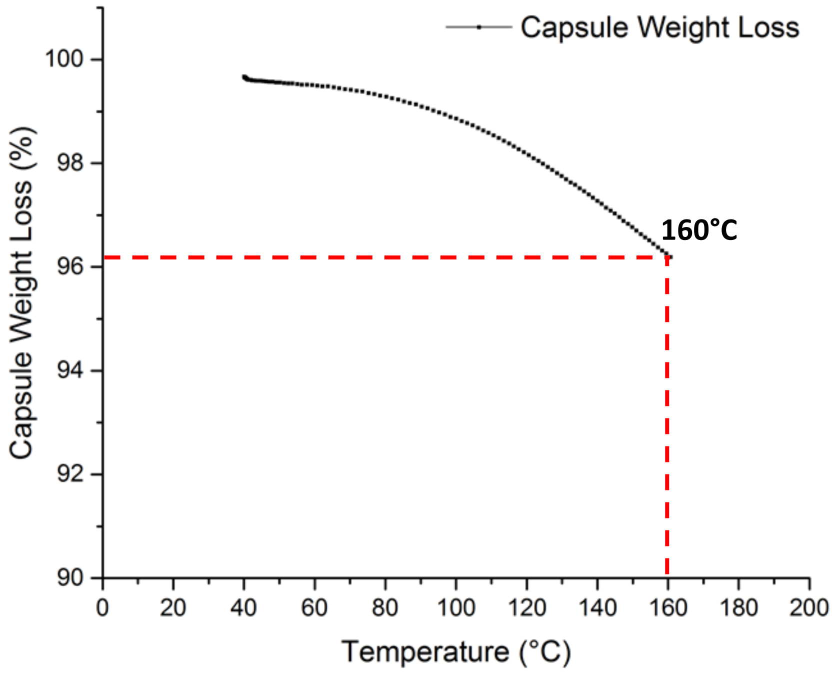

Fig. 12. Thermogravimetric analysis

3.4 Compressive tests on capsules

Fig. 13 shows the compressive testing results for capsules cured with different temperatures. Generally, with the curing temperature ranges from -20°C to 160°C, a decreasing of compressive strength can be found. When cured under temperature between -20°C and 100°C, capsules show similar compressive strength around 12 MPa and the compressive behaviors are relatively stable with different curing temperatures. However, as curing temperature exceeded 100°C, a decreasing of strength can be observed, which might because of the dehydration of the alginate gel results in degradation of some calcium alginate chains. At the curing temperature of 160°C, the lowest compressive strength in the curve remains 3.27 MPa. It is higher than the stress during both asphalt mixing and cyclic loading of service life [22]. Hence, the capsules are expected to show elastic behavior and survive the asphalt mixing process and dynamic vehicle loading during the asphalt pavement service life.

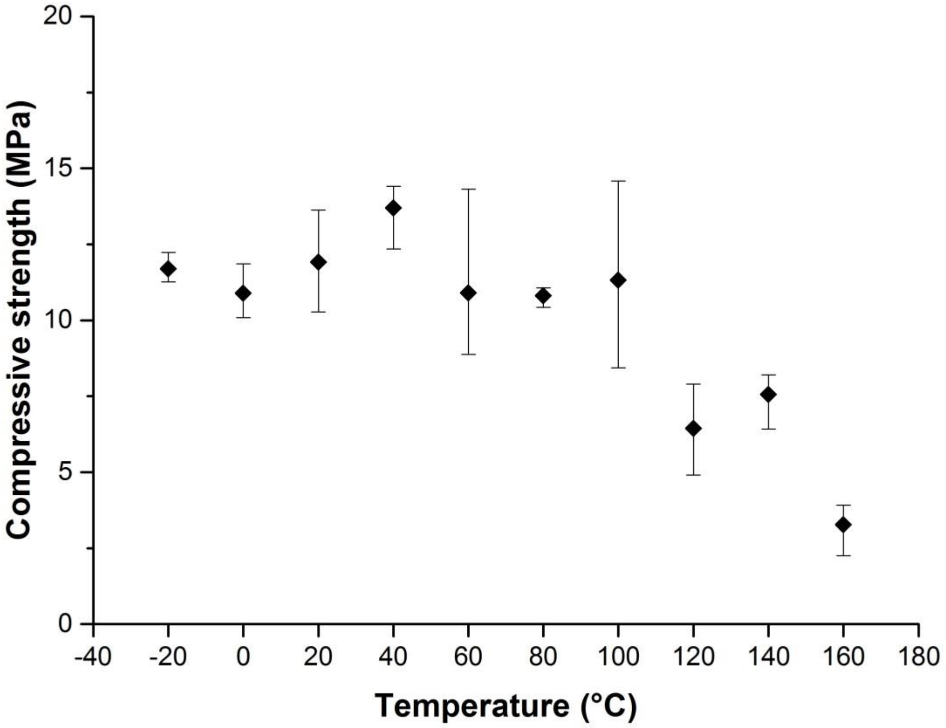

Fig. 13. Compressive strength of capsules

3.5 Asphalt mastic

The cross section profile of the crack interface from a tested asphalt mastic beam is shown in **Fig. 14. Fig. 14b** shows the cracking interface and **Fig. 14c** shows the magnified interface image. **Fig. 14b** illustrates that broken capsules throughout the depth of the beam and across the crack interface of a beam. These capsules were successfully fractured during the 3PB test. The presence of capsules throughout the crack interface demonstrates that the adhesion between capsules and asphalt binder is strong, which means that cracks are able to propagate across the depth of the capsules instead of circumventing them to trigger the release of rejuvenator as experienced with polymeric capsules [11]. **Fig. 15** summarizes the bending strength of asphalt mastic beams in 3PB tests. Beams containing capsules show higher bending strength than those without capsules, which indicates a reinforcing effect from the capsules located throughout the crack interface. This reinforcing effect is proportional to the amount of capsules in asphalt mastic beams.

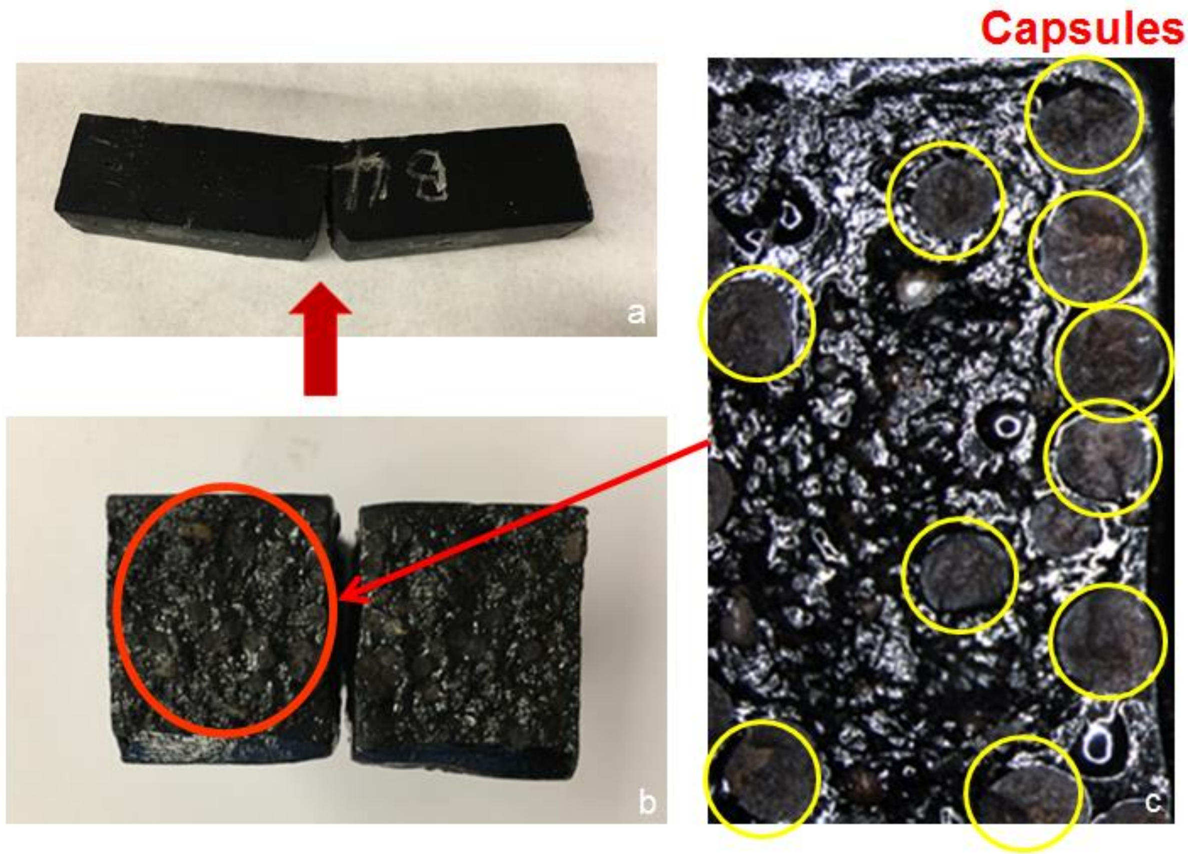

Fig. 14. Asphalt mastic beam: (a) Cracked sample, (b)cracking interface and (c) magnified interface image.

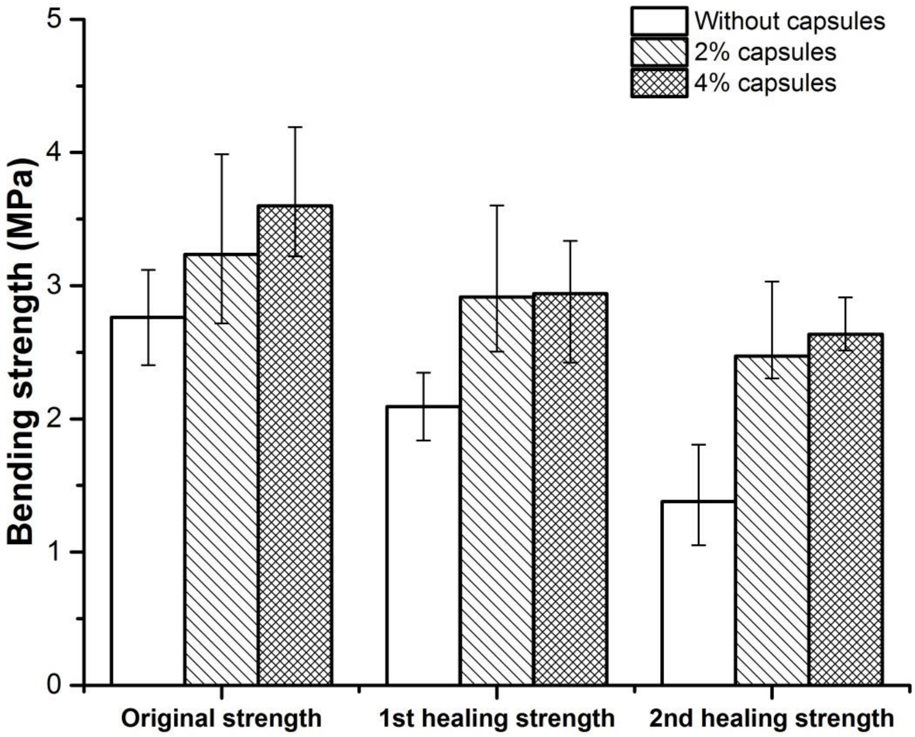

Fig. 15. Bending strength of asphalt mastic beams

Fig. 16 presents the ageing effect on capsules within the asphalt mastic mix. The mastic samples suffered ageing show no softening effect, which indicates that both short term ageing and long term ageing have little or no effect on the structural integrity of the capsule, i.e. capsules will not disintegrate and rejuvenator will not be released prematurely.

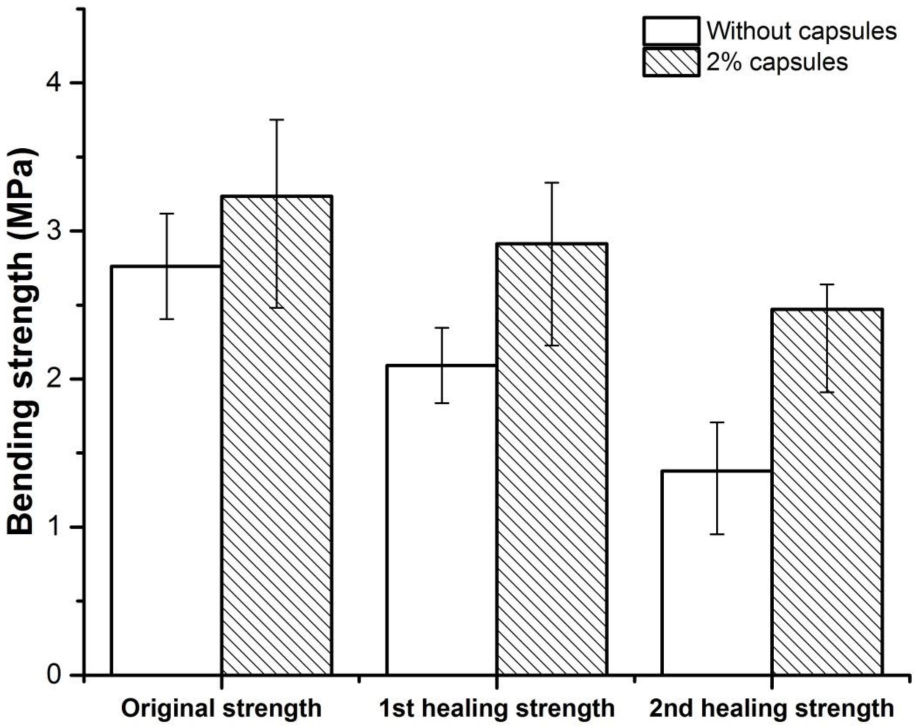

Fig. 16. Ageing effect on asphalt mastic beams with capsules

3.6 Healing efficiency

The healing efficiency of the capsules investigated with the 3PB testing and healing programme is presented in **Fig. 17**. Because of the intrinsic healing capacity, asphalt mastic beams without capsules are able to recover 75.7% of the original strength in the first healing and 50.0% in the second healing. While with capsules, this healing effect is improved significantly. Addition of 2% capsules, the healing index reaches at 90.1% after the first healing and 76.4% after second. However, addition of 4% capsules shows less healing effect than 2%, which healing index is 81.7% and 73.2% for the two healing stages.

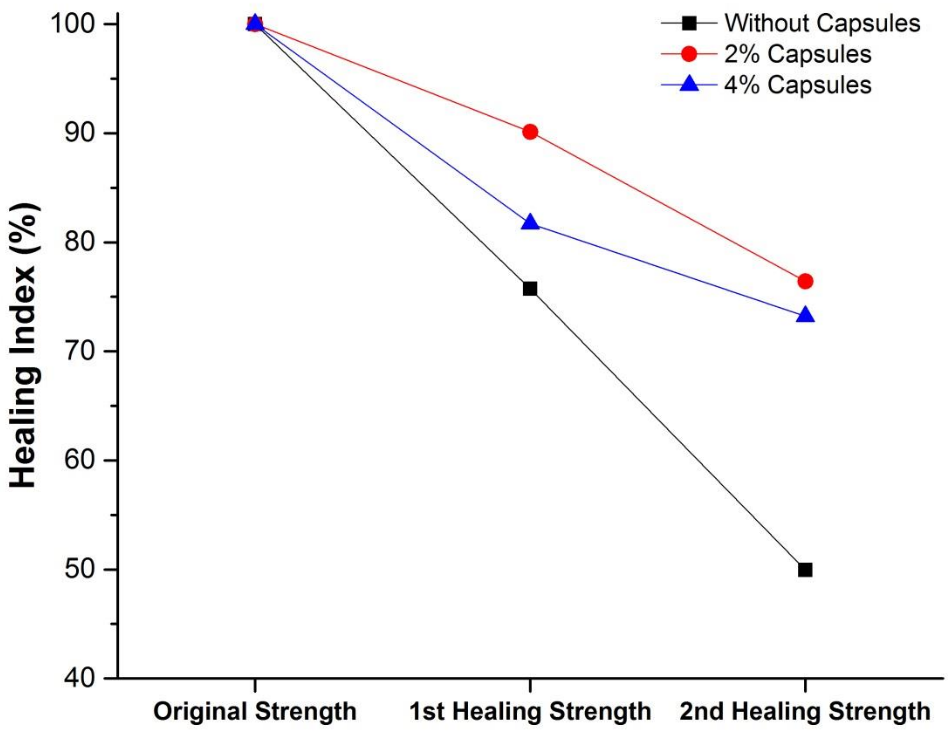

Fig. 17. Healing efficiency of asphalt mastic beams

These test results demonstrate that addition of calcium alginate capsules with encapsulated rejuvenator significantly increases the healing efficiency of asphalt mastic. However, to achieve an optimal healing rate of the asphalt mastic mix containing calcium alginate capsules encapsulating rejuvenator, the optimum volume of capsule needs to be determined.

4. Conclusions

This study illustrates the potential use of calcium alginate to encapsulate rejuvenator to improve the self-healing capacity of an asphalt mastic mix. The following conclusions were obtained based on the results in the article:

- From microscopy and x-ray tomography images, the prepared capsules have a uniform diameter of 1.95 mm and the rejuvenator content is 56% by volume. The microstructure inside a capsule is presented as a porous structure and individual rejuvenator droplets are encapsulated in the porous media.

- The results from TGA test and compressive tests on capsules indicate that these capsules have sufficient thermal and mechanical resistance to survive from the asphalt mixing and compaction period.
- The 3PB testing results show a reinforcing effect from the capsules to increase the strength of asphalt mastic by 17%, and the ageing process does not affect the capsules in asphalt mastic.
- The capsules are capable of local crack healing, and significantly increase the healing capacity of the asphalt mastic. While larger amount of capsules will not lead to higher healing efficiency. In order to achieve an optimal healing rate of the asphalt mastic mix containing calcium alginate capsules encapsulating rejuvenator, the optimum volume of capsules in the asphalt mix needs to be determined. This will form the focus of the future work of this study.

This preliminary study indicates that calcium alginate capsules have the potential mechanism for encapsulation and delivery of the rejuvenator at damage site within the asphalt mastic mix. As a result, they hold potential for the future development of self-healing asphalt technology. As a healing method in asphalt pavement, calcium alginate capsules are not expected to recover as much strength as current induction heating method [4], but the capsule healing system aims at the rejuvenation of aged binder. The advantage of this healing method is providing a more sustainable asphalt pavement.

Acknowledgement

The authors would like to acknowledge the scholarship from the China Scholarship Council (No. 201506950066). The authors also wish to thank Dr. Bert Jan Lommerts and Dr. Irina

Catiugă, Latexfalt BV, for their support to the project. In addition, the technical supports from Microlab and pavement engineering department are greatly appreciated.

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