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Self-healing of asphalt mastic by the action of polymeric capsules containing rejuvenators

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HIGHLIGHTS

- Calcium-alginate capsules with different oil content were prepared.
- Physical, thermal and mechanical properties of the capsules are presented.
- Capsules can resist the mixing and compaction processes and break inside the mastic.
- Self-healing results of asphalt mastic by the action of capsules are presented.
- The mastic healing level depended on the oil content of the capsules and temperature during self-healing.

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ABSTRACT

This paper presents the self-healing results of asphalt mastic by the action of calcium-alginate capsules containing sunflower oil. The morphological, physical, thermal and mechanical properties of the capsules have been evaluated. Additionally, the effect of the capsule oil content and the healing temperature on the self-healing properties of asphalt mastic have been evaluated. It was proven that the capsules can resist the mixing and compaction processes and break inside the asphalt mastic due to mechanical loads, releasing the oil. Healing levels in the asphalt mastic samples with capsules were greater than samples without capsules. The healing level depended on the oil content of the capsules and temperature.

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1. Introduction

The most significant problem related to the use of bitumen in asphalt roads is the ageing [1]. This problem starts immediately after mixing the bitumen with aggregates and continues during the transport and laying of roads, as well as during the service life [2]. The ageing of bitumen is due to the oxidation and loss of volatiles from its composition, and causes stiffness and viscosity increment [3]. Zargar et al. [4] state that the ageing problem of bitumen results in pavement failure, such as surface ravelling and cracking. Bitumen is usually divided into two components: a solid one called asphaltenes and a liquid one called maltenes. Maltenes can be further divided into polar aromatics (PAs), naphthalene aromatics (NAs) and saturates (paraffin) [5]. Lu and Isacsson [6] explain that during the oxidation process, PAs convert to asphaltenes, and NAs transform to PAs, which eventually oxidise and become asphaltenes as well.

Extending rejuvenators (low viscosity oils with a very high content of maltenes) on the surface of asphalt roads is probably the only method that restores the original properties of existing pavements [7] and reconstitutes the bitumen chemical composition [8]. According to Chiu and Lee [9], extending rejuvenators over the surface of roads may affect their skid resistance, because they penetrate only for the first few centimetres, and can also lead to dangerous environmental problems, such as water contamination. Also, roads have to be closed for some time after the application of rejuvenators. For all these reasons, researcher as Li et al. [10], Su et al. [11,12], Su and Schlangen [13] and Garcia et al. [14] have proposed to solve the drawbacks of using spread rejuvenators and improve the self-healing of asphalt by replacing fractions of aggregates in asphalt mixture with capsules containing rejuvenators.

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The principle behind this approach is that when micro-cracks happen next to the capsules, they will break and release the healing agent, which will dissolve bitumen and improve its flow capacity [15]. Therefore, healing agent then mixes the bitumen to seal the crack, preventing further propagation. Until now, two types of capsules containing rejuvenators have been studied: 1) microcapsules with size under 100 μm consisting of a urea–formaldehyde resin with light weight oil of high aromatic contents [10], or an oil droplet surrounded by a hard-polymeric shell [16,17], and 2) capsules with size above 100 μm consisting of a porous sand core impregnated with oil and coated with a hard shell made of filler and epoxy-cement shell to retain the oil and resist the loads [18]. Both types were proven to resist asphalt mixing and compaction, and to break in the presence of cracks and release their oil [18–20]. Additionally, the oil released has improved the self-healing ability of bitumen [21] and rejuvenated asphalt mixture [22,23].

In this paper, five different polymeric capsule types containing sunflower oil were prepared by ionotropic gelation of alginate. The capsules have been designed with two objectives: 1) to resist the mixing and compaction process; and 2) to activate self-healing of cracked asphalt mastic. With these purposes, calcium-alginate capsules with five different oil/water contents have been manufactured in the laboratory. Thus, morphological, physical, thermal and mechanical properties of the different polymeric capsule types were characterised by experimental tests. Additionally, the effect of the capsule oil content and the healing temperature on the self-healing properties of asphalt mastic have been evaluated through three-point bending (3PB) tests on the cracked asphalt mastic samples with, and without, capsules. Finally, all the methodologies and results of these analyses are presented and discussed in the sections of this paper.

2. Materials and methods

2.1. Materials

Virgin bitumen 40/60 pen with a density of 1.030 g/cm^3 and a softening point of 49.8 °C, gap graded limestone sand (91.6 g, size 0.5/2 mm; 35.8 g, size 0.125/0.5 mm; 12.3 g, size 0.063/0.125 mm; and 51.9 g, size 0/0.063 mm) with a density of 2.700 g/cm^3 and polymeric capsules were used in this study. The capsules were made of a calcium-alginate polymer that encapsulated the healing agent. The encapsulated healing agent used in the capsules was sunflower cooking oil, which is a commercial oil supplied by East End (UK). The smoke point of this material is 227 °C, while the flash point is 315 °C [20]. This oil was selected because of the low cost and the fact that extra health and safety procedures are not required for handling. In addition, sunflower oil was proven to have a positive effect on bitumen rejuvenation [4,24]. The polymeric material in the inner core of the capsules was made from (1) sodium alginate ($C_6H_7O_6Na$), which is an anionic polysaccharide widely distributed in the cell walls of brown algae; and a calcium source, provided by Sigma-Aldrich as anhydrous, granular pellets of calcium chloride ($CaCl_2$), of 7 mm diameter, and 93% purity.

2.2. Capsule preparation by encapsulation procedure

Calcium-alginate capsules were fabricated at 20 °C by ionotropic gelation of alginate in the presence of calcium, as it is defined in Micaelo et al. [20] and shown in Fig. 1. To prepare the polymeric capsules, five different amounts of oil, 25 g, 50 g, 100 g, 200 g and 300 g, were introduced with 575 g, 550 g, 500 g, 400 g, and 300 g of water, respectively, and used to produce stable emulsions (see Fig. 1). Oil and water were mixed using a laboratory gear drive mixer for 1 min at 400 rpm. Then, the sodium alginate was added and stirred until complete solution at 400 rpm for 10 min. The amount of the sodium-alginate used to produce the capsules was 15 g, since lower amounts could not form capsules because the structure of the calcium-alginate was not strong enough to hold the oil, and higher values produced capsules that were too strong, with more polymer and less oil. The capsules produced were divided into five different types, named from C1 to C5 according to the oil to water proportion (o/w ratio), as presented in Table 1. At the same time, a calcium chloride solution was prepared by dissolving 600 ml of water with 12*g* of calcium chloride ($CaCl_2$ at solution 2%). Capsules were formed by letting the oil-in-water emulsion drop into the calcium chloride solution from a 1000 ml pressure-equalising dropping funnel with 3 mm socket size (see Fig. 1). During the capsule-formation process, the calcium chloride solution was gently agitated using a magnetic stirrer. Capsules were allowed to stay in the solution until the end of the encapsulation process. Finally, the capsules were dried for 12 h by exposing them to a constant movement of air produced by a fan. Fig. 2(a) and (b) show an example of a spherical polymeric (calcium-alginate) capsule produced in this study and SEM image of the structure of capsule core, respectively.

2.3. Asphalt mastic sample preparation

Asphalt mastic beams with, and without capsules were manufactured in this study. To do this, 24 g of bitumen 40/60 pen, 191.6 g of crushed limestone sand and capsules in different amounts were used. The capsules' content in the asphalt mastic matrix is dependent on the oil content of each type of capsules. Ji et al. [24] concluded that a cooking oil content of 6–8% could successfully recover aged binder and improve its physical and chemical properties when compared to alternative oils. According to this rule, and considering the oil percentages of the capsules presented in Table 1, the mass percentage of capsules to bitumen used in the asphalt mastic beams was: 26.6% of C1; 22.5% of C2; 19.5% of C3; 12.1% of C4; and 9.0% of C5. In this way, the estimated oil to bitumen ratio in mastic beams was 7.2%. Prior to mixing, the different aggregate fractions and the bitumen were pre-heated in an oven at 140 °C for 10 h, and at 170 °C for 2 h, respectively. The graded sand and bitumen were mixed by hand until the mixture became homogeneous, and then the polymeric capsules at 20 °C were added and mixed for 20 s. Later, the mixture was poured into prismatic shaped Teflon™ moulds with dimensions $30 \times 30 \times 100$ mm^3, and compacted by hand. The bottom inside surface of the mould was fabricated with a triangular cross section protrusion at midpoint of the length to create a notch in the asphalt mastic specimens, as the one shown in Fig. 3. Finally, 260 asphalt mastic beams were manufactured in this research, with and without, capsules.

2.4. Morphological characterisation of capsules

The average diameter of 20 individual polymeric capsules was measured by taking photographs under a stereoscopic microscope with a Nikon Digital Eclipse DXM1200 camera, and processed with the software ImageJ®. To observe the internal microstructure of the polymeric capsules, these were first cast in an epoxy resin matrix and then abraded until the inner structure/core was exposed. The oil was cleaned from the section of the capsules using absorbent paper. Finally, the capsules' morphology was studied using a Scanning Electron Microscope Philips XL30FEG SEM.

2.5. Thermal characterisation of capsules

The composition and thermal resistance of capsules and their individual components were evaluated by means of thermogravimetric

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