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Review Article

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Self-healing Microencapsulation Technology for Asphalt Pavements: A Review

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Abstract

Self-healing bituminous materials are at the cutting edge of asphalt pavements technologies. To enable self-healing capability, various techniques "based on extrinsic self-healing" have been developed: externally triggered heating using induction heating or microwave radiation, and, more recently, the use of embedded microcapsules. These technologies typically involve adding rejuvenators, special materials, or additives, to the asphalt mix that can react and repair microcracks at the beginning of the cracking process. One particularly promising approach is microencapsulation healing technology, which involves encapsulating rejuvenating agents within microcapsules. This revolutionary technology holds immense potential for the autonomous healing of aged bituminous materials. When microcapsules are incorporated into asphalt pavements, they can achieve impressive recovery rates, with healing efficiencies ranging from 30% to 90%, bringing the pavement closer to its initial state. There are many techniques used to encapsulate materials which can be classified into three categories depending on the capsule manufacturing. These categories include physico-mechanical methods, which rely on mechanical actions or physical processes; chemical methods, resulting from chemical interactions between the materials; and physico-chemical methods, which involve the formation of the capsule wall using natural or synthetic preformed polymers. The purpose of this paper is to provide an overview of microencapsulation technologies to promote asphalt self-healing from a sustainable perspective.

Keywords

Self-healing, Microencapsulation technologies, Bituminous materials, Microcapsules, Microcracks

Introduction

Asphalt pavement has been widely used in roads construction since its installation is simple, inexpensive, easy to maintain and recyclable [1]. As known, asphalt mixtures are composed of air voids, aggregate particles, and asphalt binder [2]. Bitumen serves as binder for asphalt, which is a combination of asphaltenes and maltenes (Resins and oils) [3]. Furthermore, durability of asphaltic mixtures is strongly dependent on the bonding characteristics between the asphalt binder and mineral aggregate [4]. However, during the oxidative process and with the exposure of the asphalt pavement throughout the years to mechanical, thermal, and environmental effects (Such as air oxidation, ultraviolet radiation, and moisture), the detachment between bitumen and mineral aggregates occurs at the interface [5, 6]. Subsequently, the asphaltene content increases, while the maltene content decreases [7]. All these changes at the molecular level led to an increase in pavement stiffness, which causes cracking of the asphalt materials [8, 9]. This cracking is one of the main types of problems faced by asphalt pavements. It eventually leads to irreversible damage that results in significant maintenance and repair costs for the road agencies in every country [10, 11].

Asphalt mixtures are considered as self-healing materials, since bitumen is a viscoelastic material displaying the Newtonian fluid behavior at temperatures from 20 to 30 °C and exhibits changes in its viscosity during rest periods [12,13]. However, this process may require several days for complete healing, which can be complicated due to continual traffic flow [14]**.** In order to accelerate the healing process of asphalt mixtures and extend the service life of asphalt pavements by providing the crack closure at an early stage, this subject has become a new area of study. Researchers have proposed and developed some technologies which are based on self-healing bituminous materials. The main approaches practiced for self-healing of asphalt pavements are: Incorporation of nanomaterials in asphalt pavement, heating and encapsulate rejuvenators [15].

The first approach is based on incorporating nanoparticles which are added to the bitumen mix to improve the physical and mechanical properties of the binders [16]. Consequently, improving the *in situ* performance of the asphalt pavement [17]. Based on the literature, many researchers used to improve the asphalt mixture resistance to fatigue, permanent deformations, and oxidative ageing, modifying asphalt binder by adding nanomaterials. Hamedi et al. [18] evaluated the modified binder with nano-Al_2O_3 and nano-Fe_2O_3. Concluded that nano-additive of Fe_2O_3 improved asphalt mixture resistance to moisture compared to Al_2O_3. Golestani et al. [19] studied nano-clay addition which enhanced viscosity and mechanical proprieties to asphalt binder as results. Zhang et al. [20] used nano-ZnO that improved stability in high-temperature and resistance to ageing of bitumen. Qiu et al. [21] modified binder with nano-rubber which upgrades self-healing capability of bituminous binders. Therefore, increase lifespan of asphalt pavements.

The second one is based on reducing the viscosity of bitumen which has self-healing characteristics by increasing its temperature through externally triggered heating via induction and microwave radiation [15]. Nanoparticles incorporation approach improves material properties and reduces cracking susceptibility. However, it's proved that the effectiveness of crack healing depends on the external stimulus based on heating which triggers the process of healing [22]. Induction heating works based on the principles of electromagnetic induction in order to heal damage appeared to asphalt pavements by adding conductive fillers and fibers [23, 24]. Liu et al. [25] evaluated the healing capability of steel wool (1.27% mass content of the total mixture) which has increased the healing rate of the porous asphalt. Additionally, Liu et al. [26] used steel fibers which revealed 60% of mechanical resistance recovered. While microwave radiation based on an electromagnetic radiation, producing a change in the orientation of polar molecules, which results in internal friction and an increase of the mixture temperature, adding ferrous materials which accelerate heat increase [27]. Zhao et al. [28] tested filler additives in asphalt mixture, the NiZn ferrite content resulted in a significant increase of the heating speed of asphalt. Franesqui et al. [29] investigated healing efficiency of up-graded asphalt containing steel wool, steel filling, and metallic powder using microwave heating.

The final technology is the microencapsulation which is an autonomic approach by the action of encapsulated light component oil (Such as asphalt rejuvenator agent, waste vegetable oil, waste cooking oil, etc.) with certain wall materials to form self-healing microcapsules with certain sustained release ability [30, 31]. Furthermore, the self-healing process based on microcapsules has been successfully proved in some self-healing inclosing rejuvenators [32, 33]. They are sufficiently thermally and mechanically stable to survive the asphalt production process as shown in table 1 [34]. During heating, the high temperatures used may change the flow behavior of the asphalt binder, which provides the aging of this last [35]. Nevertheless, microencapsulation restores the maltenes ratio, regulating the chemical composition of asphalt. Thus, the released rejuvenator not only restores the asphalt stiffness and strength by closing the micro-cracks but also promotes the self-healing properties of asphalt, extending the

Table 1: Microcapsules characteristics used for self-healing in asphalt pavement.

Materials used	Size	Temperature	Mechanical strength	Ref.
Melamine urea formaldehyde/Soybean oil	90.19 μm	Stable below 200 °C	Young modulus 2.5 GPa Hardness 0.28 GPa	[44]
Urea-formaldehyde/Rejuvenator	35 μm	Stable below 250 °C	Young modulus 5.024 GPa Hardness 155.45 MPa	[45]
Calcium alginate/Sunflower oil	1.8 mm	Stable below 200 °C	Strength higher than 10 N	[46]
Melamine-formaldehyde/Cooking oil	55 μm	Stable below 180 °C	Young modulus 2.10 GPa	[40]
Alginate/Rejuvenator	4.6 mm	Stable below 200 °C	Strength higher 12 N	[47]
Alginate/Sunflower oil	1.05 mm	Stable below 180 °C	11.9 N	[48]
Alginate/Rejuvenator	1.95 mm	Stable below 160 °C	Compressive strength stable at 12 MPa	[49]

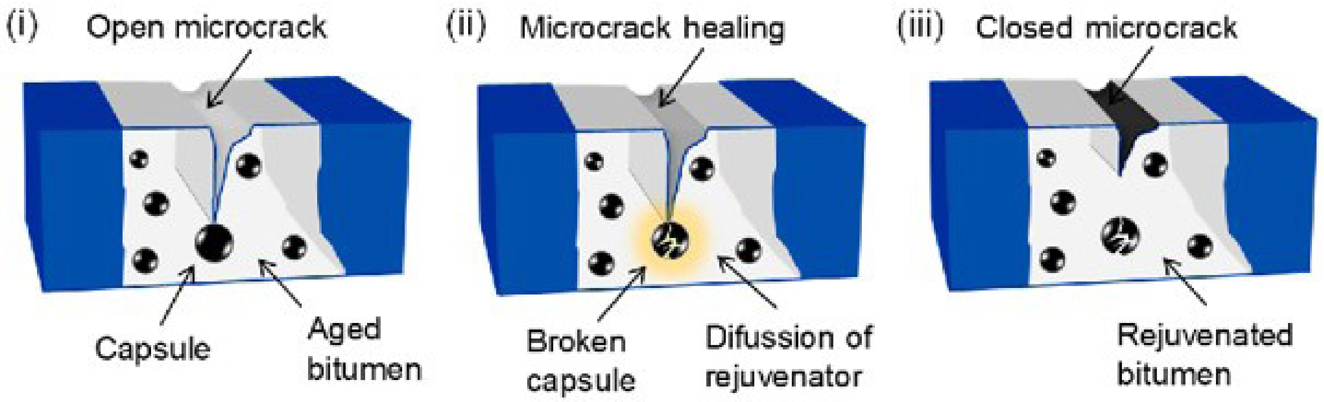

Figure 1: Microcapsules mechanism in asphalt pavement to heal cracks [8].

serviceability time of asphalt pavement [36]. The self-healing microcapsules are incorporated into asphalt materials, the light component oil was slowly released from the microcapsules, softening the aging asphalt around the cracks, so as to achieve the purpose of self-healing of asphalt performance [37]. Figure 1 illustrates self-healing the cracking in asphalt pavement by microcapsules. Considering Self-healing microencapsulation is an innovative emerging technique to enhance the lifetime and quality of asphalt pavements [38]. Researchers are nowadays carrying out intense research to promote the healing of bituminous materials in asphalt pavement by the action of microencapsulating the rejuvenators agents [39, 40]. Chung et al. [41] prepared microcapsules using SBS/DMP coated by urea-formaldehyde as shell material, providing that asphalt containing microcapsules possesses excellent self-healing potential. Tabakovic et al. [42] used alginate fiber for the encapsulation of rejuvenator, and they found that these microcapsules could increase the strength of the asphalt mastic by 36%. Sun et al. [43] made microcapsules which proved high thermal stability and strength mechanical resistance.

The main objective of the present paper is to give an overview about microencapsulation methods used to reach microcapsules, which are summarized into the three categories physical-mechanical, chemical, and physico-chemical, as well as the materials used in each microcapsule component.

Materials Used for Microencapsulation

As described previously microencapsulation technology is based on the formation of microcapsules in which a solid, liquid, or gaseous substance (i.e., core materials) are surrounded by a uniform and stable coating wall (i.e., shell materials) by physical and chemical methods [50]. Figure 2 illustrates the composition of microcapsules in a simplified form [51]. These microparticles have been widely used in several fields, including roads construction. There are many materials that have been used in microcapsules synthesis applied in self-healing asphalt pavement with several microencapsulation techniques as summarized in table 2. Microencapsulation process is based on building a functional barrier between the core and the wall material to avoid chemical and physical reactions and to maintain the functional, and physicochemical properties of the core materials [51, 52]. This last is referred to as the internal phase or fill which has the ability to repair the cracking appeared by recovering the asphaltene/ maltene ratio [30]. The wall materials referred to the coating, shell, or membrane, that determines the stability of microparticles, the process efficiency, and the degree of protection for the core. Wall materials used commonly for the microencapsulation of oils include synthetic or biopolymers, that must have a higher thermal stability to resist the melting temperature and a higher mechanical strength to resist the mixing pressure of asphalt in practical application. Furthermore, the shells should break when a microcrack appears, and cannot be ruptured without releasing the rejuvenator, in order to reach the self-healing's goals based on microencapsulation technology.

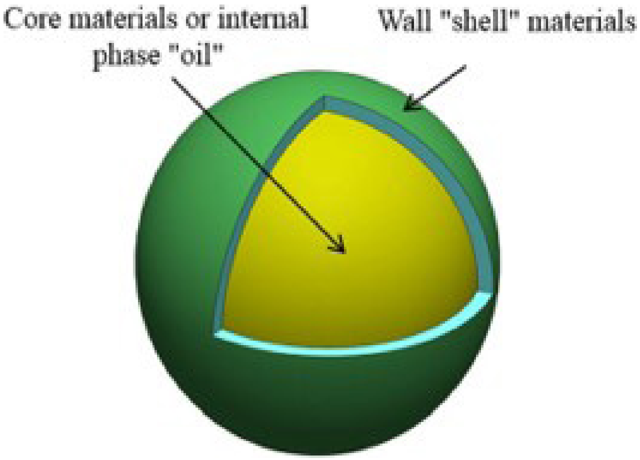

Figure 2: Composition of microcapsules in a simplified form [51].

Encapsulation Methods

As described previously, the encapsulation methods are classified into three categories. Table 3 summarizes the different techniques used in each method [10]. Figure 3 illustrates the microcapsule fabrication process.

Chemical methods

Microencapsulation by chemical methods is mostly based on using polymer as coating agent of the rejuvenator's materials chosen to be encapsulated [57]. According to Kanellopoulos et al. [50], the encapsulation takes place, in this case,

Table 2: Some materials used in microencapsulation technology for self-healing asphalt pavements.

Microencapsulation technique	Wall material	Core material	Applications	Ref.
Interfacial polymerization	Isophorone di-isocyanate	Asphalt rejuvenator	Preparation and performance of microcapsules for asphalt pavements using interfacial polymerization	[53]
In situ polymerization	Urea and formaldehyde	Rejuvenator composed from organic oil	Characterization of asphalt binder containing microcapsules	[54]
Ionic gelation	Biopolymer matrix of sodium alginate	Bio-oil obtained from liquefied agricultural biomass waste	Microencapsulated bio-based rejuvenators for the self-healing of bituminous materials	[8]
Coacervation	Prepolymer of melamine-Formaldehyde modified by methanol	Aromatic oil	Synthesis and physicochemical properties of high compact microcapsules containing rejuvenator applied in asphalt	[39]
Ionic gelation	Ca-alginate polymer	Commercial rejuvenator	Ca-alginate capsules encapsulating rejuvenator as healing system for asphalt mastic	[49]
In situ polymerization	Melamine-urea formaldehyde	Commercial rejuvenator	Aided regeneration system of aged asphalt binder based on microcapsule technology	[55]
Coacervation	Melamine-formaldehyde modified by methanol	Waste cooking oil	Investigation the possibility of a new approach of using microcapsules containing waste cooking oil: *In situ* rejuvenation for aged bitumen	[40]

Table 3: Different techniques used in microencapsulation technology according to the three categories.

Chemical method	Physico-mechanical method	Physico-chemical method
- Interfacial polymerization - *In situ* polymerization	- Spray drying - Pan coating - Extrusion-based method	- Ionic gelation - Coacervation

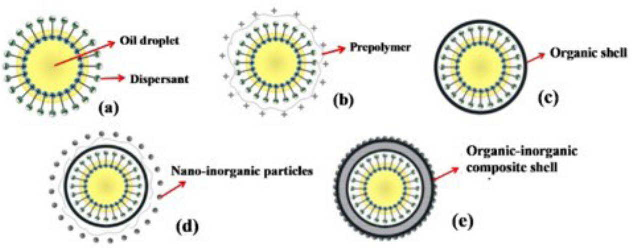

Figure 3: Illustration of the microcapsule fabrication process [56].

by the polymerization or poly-condensation of substances to constitute the wall of capsules, which is produced as the result of chemical interactions between the used materials. The techniques used in this chemical method for microencapsulation are: Interfacial polymerization and *in situ* polymerization [58]. Figure 4 illustrates microencapsulation process by the two chemical methods.

Interfacial polymerization

In this technique microcapsule formation results from the rapid polymerization between two reactive monomers soluble in the immiscible solvent used [60]. On one side, a lipophilic monomer is dispersed at the interface to form an oil-in-water emulsion in an aqueous phase with the core materials used. A hydrophilic monomer added on the other side to the solution [61]. And then rapid polymerization occurs, which is the result of the reaction between these two monomers cited above, at the interface of the two emulsified phases [50]. The polymer surrounds the core material and forms the capsule wall. Typically, emulsifiers are also added into the system to ensure the proper dispersion of the formed particles. Polyester, polyamides, and polyurethane are the commonly materials used for the formation of shells in interfacial polymerization [36]. This method can be used to fabricate microcapsules with sizes in the range from a few microns up to around 100 μm [50]. Thus, it allows the synthesis of capsules with high loading capacities. In construction related materials, interfacial polymerization has been used to encapsulate phase change materials, it is a simple and reliable method with a relatively low cost [62].

In situ polymerization

This technique is a direct polymerization that involves a chemical reaction, carried out on the oily particle surfaces and occurring between the interface of the continuous phase and the core materials [50]. The formation of capsules is generated by the deposits of the prepolymer on the surface of core materials, which is responsible for the shell formation in the microcapsules [52]. The commonly materials used in this technique are aminoplast resins such as urea-formaldehyde, melamine-formaldehyde, urea-melamine-formaldehyde, or melamine-formaldehyde polymers modified with resorcinol [10]. The difference between *in situ* and interfacial polymerization is that in the former no reactants are present in the

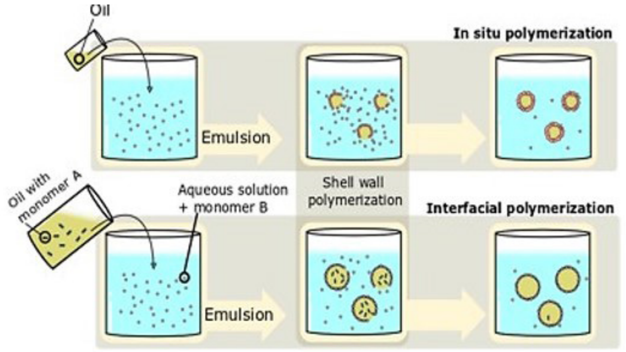

Figure 4: Microencapsulation by chemical methods [59].

core material. And in the same way most of the studies reported in the literature used in situ polymerization to produce microcapsules with average diameters as well as the interfacial polymerization [50]. The produced microcapsules depend on such factors as the acidic conditions [44]. Thus, after reaching a stable emulsion and adding water-soluble melamine resin, this *in situ* polymerization is generally controlled by pH levels adding a strong acid to initiate the polycondensation [63]. This technique was used in several industrial applications, and among these applications the encapsulation of healing compounds for self-healing based on *in situ* polymerization became the primary method used in this field.

Physico-mechanical methods

For physico-mechanical methods, microcapsules are produced because of either mechanical action such as coating, drying or a physical process like evaporation [50]. In other words, shell material is mechanically applied or condensed around the microcapsule core. The most common physico-mechanical encapsulation procedures are spray drying, pan coating, and extrusion-based methods.

Spray-drying

The spray drying technique is a low-cost commercial process, based-on physico-mechanical mechanism using mechanical interventions like tumbling process, with an appropriate and compatible shell matrix [50]. Microcapsules obtained by using this technique by dispersing the core material, which is immiscible with water in a polymer solution, they are sprayed in a hot chamber until the desired oil droplets are attained, then rapidly dried. This dehydration leads to a solidification of the shell material on the core particles while the solvent evaporates [64, 65]. Spray drying technique is readily available in many industries, and it has been used in the construction field to develop microcapsules for self-healing, which their size range is around 10 - 50 µm [66]. Figure 5 illustrates microencapsulation by spray-drying process.

Pan coating

The microencapsulation based-on pan coating is one of the oldest techniques used specially in the pharmaceutical field for forming small, coated particles or tablets. Also, relating to construction materials and to develop self-healing concrete, pan coating has been used to manufacture granules encapsulating many agents [10]. Microcapsules obtained by this technique are the results of applying as a solution the shell material in liquid form using a high-pressure nozzle to the desired solid core material to encapsulate in the coating pans. Figure 6 explains the microencapsulation principle by pan coating technique. Based on the literature, this technique presents some issues related to the thickness and integrity of the shell which can affect the mechanical properties and structural components of the materials [67].

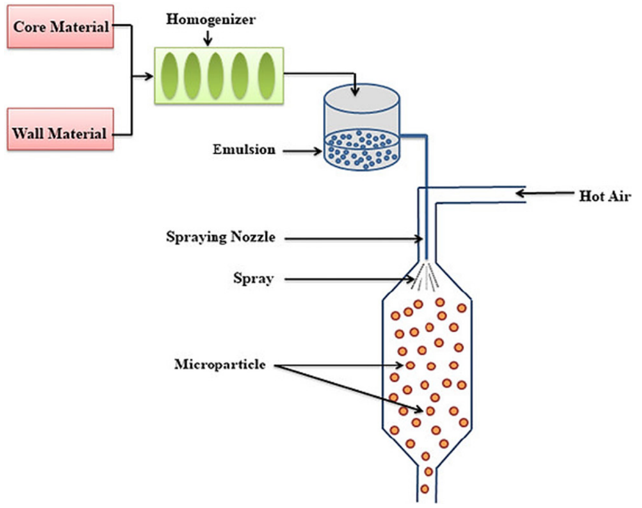

Figure 5: Schematic representation of microencapsulation by spray-drying process [51].

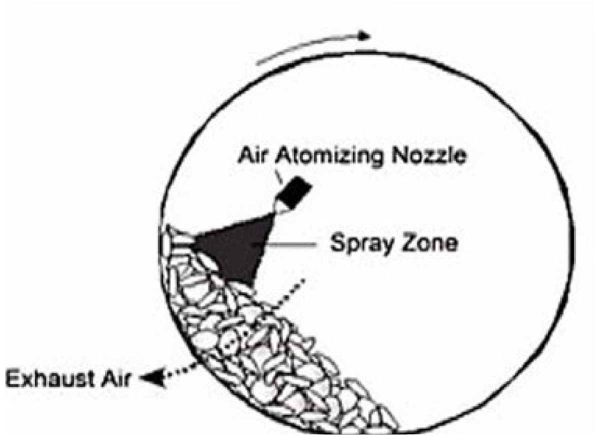

Figure 6: Microencapsulation principle by pan coating technique [68].

Extrusion-based methods

This technique is widely used to produce microcapsules, based on the principle of extrusion process of a liquid through a nozzle. During the vibrating, and as the core material in its liquid phase immiscible with the coating material, they emerge from the orifices and break up into spherical drops because of surface tension forces applied [69]. As shown in figure 7. Afterwards the droplets produced by extrusion are solidified to microcapsules by either physical or chemical means [65]. This technique has two methods: Prilling which is used to manufacture microcapsules or microspheres, while spinning used to produce encapsulating fibers based on alginate as a

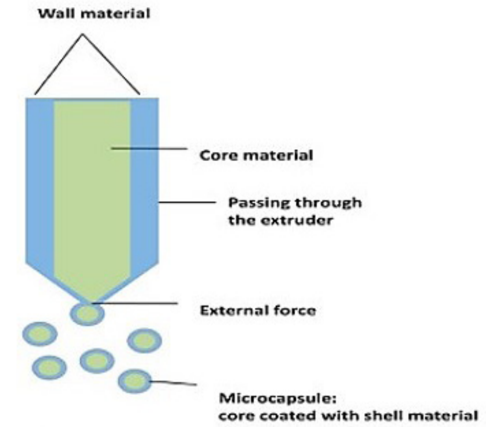

Figure 7: Schematic illustrates extrusion microencapsulation method [65].

polymeric fiber [70]. There are other factors influencing the formed drops such as the flow rate of the laminar jet, the nozzle diameter, and the viscosity of the extruded liquid, amongst other factors [71].

Physico-chemical methods

Physico-chemical methods have been used for producing microcapsules are based on the formation of the microcapsule wall from preformed polymers either natural or synthetic following the variation of temperature, pH value, or electrolyte concentration. The techniques commonly used are ionic gelation and coacervation.

Ionic gelation

In the physico-chemical methods, ionic gelation is widely used to form microcapsules based on the ability of polyelectrolytes to crosslink in the presence of multivalent counter ions to form hydrogels [72]. It is considered as a sustainable approach to extend the lifetime of roads producing core-shell and polynuclear encapsulating rejuvenators with size range 2 - 7 mm, using bio-based polymers like alginate, bio-oils as rejuvenating agents and calcium ion would lead to the calcium alginate microcapsules as illustrated in figure 8 [58]. This approach led to extending the lifespan of roads due to the polynuclear capsules which have the ability of not releasing all the rejuvenator agent while reaching the crack. Furthermore, they have been designed to provide structural reinforcement that allows the capsule to survive during the asphalt manufacturing process. Thus, anticipate multiple crack-healing and long-term healing [73].

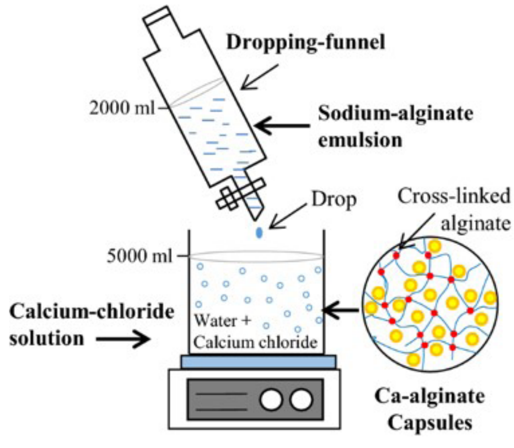

Figure 8: Ionic gelation microencapsulation process [74].

Coacervation

The other technique in physico-chemical methods is coacervation. This last is defined as the partial desolvation of homogenous polymer solution into a polymer-rich and a poor polymer phase. This technique is divided into two processes: simple or complex [60]. As shown in figure 9; In the first one, a desolvation agent is added for phase separation which triggers the interaction of the dissolved polymer with a low-molecular substance. While the second coacervation involves complexation between two oppositely charged polymers through their interactions, including three basic steps: preparation of emulsion; encapsulation of core material; and rigidization of the coating [10]. For further details, the process starts with the formation of the immiscible phases and the preparation of the emulsion by dispersing the core material into the polymer solution; secondly, the deposition of the coating onto the core material by adding the second polymer solution, changing the pH, the temperature, or by dilution of the medium to form the complex by the two polymers; and finally, the stabilization of the microcapsules by crosslinking [52].

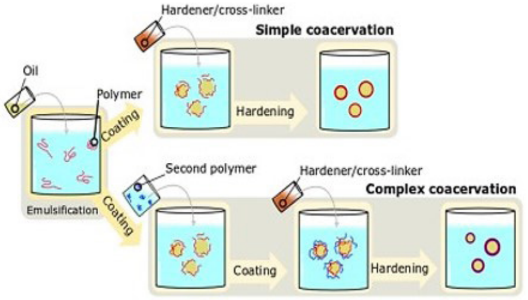

Figure 9: Simple and complex coacervation processes [59].

Conclusions

Microencapsulation technology is a promising approach for developing self-healing asphalt pavements. This review article has explored several microencapsulation techniques employed for the encapsulation of healing agents in asphalt mixtures, highlighting their advantages and limitations. Among the microencapsulation methods discussed:

- The physical coating method involves applying a protective layer around the healing agent, offering simplicity and cost-effectiveness. Furthermore, challenges with physical coating method include mechanical stability of the coating and release kinetics of the healing agent.
- Chemical methods form a polymer shell around the healing agent, providing controlled release and environmental protection. Moreover, chemical methods offer enhanced stability and controlled release kinetics, but may require specialized equipment.
- Selection of microencapsulation method depends on factors like desired compression strength, temperature stability as well as nature of healing agent.
- Future research should focus on optimizing the existing microencapsulation techniques, developing novel encapsulation methods, and conducting comprehensive evaluations of their performance in real-world conditions.

Microencapsulation technology, with its diverse range of methods, provides a promising avenue for the development of self-healing asphalt pavements. By selecting appropriate microencapsulation techniques and optimizing their parameters, it can be unlocked the full potential of healing agents in repairing and rejuvenating asphalt pavements, thereby enhancing their durability and extending their service life. Continued research and advancements in microencapsulation technology will undoubtedly contribute to the realization of self-healing asphalt pavements, revolutionizing the field of transportation infrastructure for the benefit of society.

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Conflict of Interest

None.

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