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Revolutionizing Self-Healing Asphalt: Optimized Encapsulated Rejuvenators for Enhanced Durability and Sustainability

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Abstract

In an era where sustainable infrastructure is crucial, self-healing asphalt emerges as a transformative solution to enhance pavement longevity and reduce maintenance costs, addressing the global challenge of deteriorating road networks. This study presents a pioneering investigation into the development and performance evaluation of encapsulated rejuvenators for self-healing asphalt, utilizing two distinct compositions; waste cooking oil (WCO) and Fischer-Tropsch bright stock oil (FTBSO), across three capsule sizes (1 mm, 2 mm, and 3 mm). Through the experimental tests on compressive strength, thermal stability, and rupture resistance under wet conditions, the ongoing study highlights the critical influence of capsule size and composition on the mechanical performance, as well as the resistance to degradation and oxidation under similar asphalt production conditions, including applied stresses and temperatures. The findings indicate the superior performance of 3 mm FTBSO-based encapsulated rejuvenators, which exhibit exceptional compressive strength (155 N), minimal weight loss (2% at 200° C after 1-hour short-term aging), and high rupture resistance (80 minutes to break under moisture at 100° C), making these capsules ideal for withstanding mechanical and thermal stresses, while ensuring effective crack repair. In addition, both 2 mm and 3 mm FTBSO- and WCO-based rejuvenator capsules demonstrated high resistance to compressive stresses, excellent thermal stability, and strong rupture resistance, making these capsules suitable for self-healing asphalt applications. In contrast, 1 mm WCO-based rejuvenator capsules exhibited the lowest compressive strength (32 N), the highest weight loss (10% after 1 hour of short-term aging at 200° C), and the fastest rupture under moisture (18 minutes to break at 100° C), making these capsules the least suitable for self-healing asphalt applications.

Keywords: Self-Healing Asphalt; WCO; FTBSO; Encapsulated Rejuvenators; Sustainable Pavement; Thermal Stability.

1. Introduction

1.1. Background

Asphalt pavements form the backbone of today's infrastructure. However, these pavements deteriorate with the passage of time due to the effect of oxidation, temperature fluctuations, and loads, causing the formation of cracks and the shortening of their lifespan [1]. The traditional forms of maintenance, including the processes of resurfacing and crack sealing, prove costly and result in serious environmental impact. For countering these issues, self-healing asphalt containing rejuvenating agents in the form of capsules has emerged as an advanced method. It allows the cracks in the pavement to self-heal, thereby extending the lifespan of the pavement [2]. The rejuvenators restore the maltenes lost in aged asphalt binders, thereby providing flexibility, decreasing brittleness, and enhancing anti-cracking features. From

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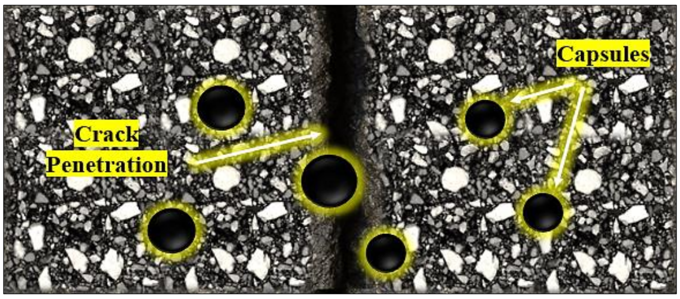
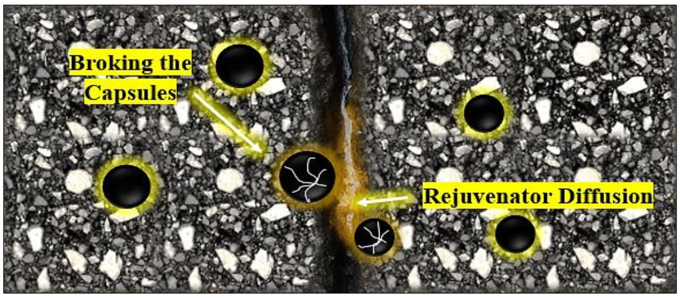
the different rejuvenators investigated, the bio-based and petroleum-based rejuvenators specifically emerged as efficient and asphalt binder-compatible [3]. In the current work, two common rejuvenators were investigated: waste cooking oil (WCO), consisting of a bio-based component from reused vegetable oils [3], and Fischer-Tropsch bright stock oil (FTBSO), an oil-based rejuvenator with high thermal stability and oxidation stability [4].

WCO is an ecofriendly rejuvenator extracted from waste vegetable oils in the domestic market and households. It is advantageous in possessing high penetration, low viscosity, and fiscally viability. It, however, presents some challenges, including the likelihood of excessive softening of the asphalt binder and the potential for oxidation, and needs the use of stabilization measures to improve performance [5]. In contrast, FTBSO is an advanced petroleum-based product with high molecular weight hydrocarbons and low aromatic composition. It has excellent oxidation stability, thermal stability, and controlled softening of the asphalt binder. FTBSO also presents environmental concerns linked to its petroleum-derived composition, particularly regarding its non-renewable aspects and the release of volatile organic compounds (VOCs) [6].

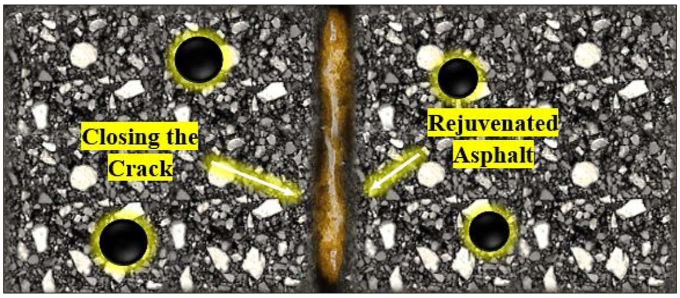
Capsule self-healing technology holds the potential to enhance the durability of asphalt pavement. It uses capsules containing rejuvenators as an integral part of the asphalt mix. When the development of cracks happens, the stress on the crack tip bursts the capsules, releasing the rejuvenator. It seeps into the crack through capillary action, revitalizing the original properties of the asphalt as presented in Figure 1. Capsules should be high in thermal stability, mechanically strong, and well-dispersible in order to sustain process conditions such as compaction, mixing, and the initial traffic load without failure early on [7]. Uniform distribution needs to be achieved for the capsules in order to prevent clustering, as this would hamper the performance of the asphalt.



A- Expanding the crack



B- Healing the crack



C- Closing the crack

Figure 1. Cross-section of asphalt demonstrating how encapsulated rejuvenators aid the self-healing of cracks over time

Earlier researchers show the self-healing effectiveness in asphalt mixtures are affected by several parameters with the dependence including temperature, healing time, and the dosage of capsules. Most notably, the use of higher temperatures is important in speeding the process significantly [8]. It is essential to know the interactions among the components in order to ensure the optimum performance of self-healing technology in actual implementations. Capsule size is extremely important in defining the mechanical properties of self-healing asphalt, such as its strength against compression, its thermal endurance, and its rupture strength. Capsule size plays an important function in order to achieve an optimum balance amongst strength, durability, and healing efficiency in self-healing asphalt mixtures.

1.2. Literature Review

Many researchers have explored the possibility of using encapsulated WCO and FTBSO in self-healing asphalt and showed that they have high potential. It is discovered that these rejuvenators are able to function well in rejuvenating old asphalt through the improvement in its properties, its increase in self-healing capacity, and its prolongation of service life. Wang et al. [9], evaluated waste oil-based capsules for self-healing asphalt, finding that waste rapeseed oil performed best in restoring aged asphalt. However, their study focused mainly on material characterization, leaving gaps in optimizing capsule size, dosage, and distribution. The ongoing study addresses these gaps by enhancing encapsulated rejuvenator design to improve self-healing efficiency and mechanical performance.

The study by Zghoundi & Akkouri. [10], investigated the durability of capsules in asphalt pavements, identifying stability challenges and suggesting improvements. However, their work did not address optimal composition or environmental interactions. The current study advances these findings by optimizing encapsulated rejuvenators for enhanced self-healing and durability. Furthermore, Su [11], developed oily rejuvenator-filled capsules for self-healing asphalt, evaluating their fabrication, dispersion in bitumen, and healing efficiency. The study showed that the capsules remained intact during mixing and effectively released rejuvenator upon cracking, restoring aged bitumen through capillary action. While multi-recovery abilities and self-healing performance were demonstrated, optimal capsule size and dosage were not explored. The present research aims to fill these gaps by optimizing encapsulated rejuvenator design for improved asphalt durability. On the other hand, Dong et al. [12], studied the self-healing characteristics of rejuvenated asphalt, modified with both crumb rubber and waste cocking oil. Even though their study has revealed the efficiency in recovering the deteriorated asphalt, capsule size and dose has not been explored practically.

The current study expands upon their results by concentrating on the optimization of capsule design to improve the efficiency of self-healing in asphalt. Likewise, Gao et al. [13], implemented an investigation into using waste cooking oil and rubber powder as asphalt rejuvenators demonstrated effective rejuvenation; however, it did not optimize capsule size and dosage. The current study builds on this research by optimizing the design of encapsulated rejuvenators to enhance performance. Moreover, Xing et al. [14], examined the use of environmentally friendly microcapsules containing waste soybean cooking oil as a rejuvenating agent for bitumen. Although the research indicated potential anti-aging benefits, it did not address how the size and spatial arrangement of the capsules might affect self-healing effectiveness. This study builds upon their previous work by refining the encapsulation of the rejuvenator to improve the longevity and functionality of asphalt mixtures.

1.3. The Novelty of The Study

The uniqueness in this work lies in its novel approach, combining encapsulated rejuvenators produced using Fischer-Tropsch bright stock oil (FTBSO) and waste cooking oil (WCO) for self-healing asphalt. In addition to presenting comparative analysis in their efficiency, the work goes deep into the effect of particular sizes (1 mm, 2 mm, 3 mm) on the compressive strength, thermal stability, and rupture when subjected in wet conditions. The novel approach offers a revolutionary framework for optimizing the design of encapsulated rejuvenators. It highlights their excellent performance under the same asphalt production conditions comprising applied stress and temperatures. It paves the way for further investigations on both synthetic and natural rejuvenator encapsulation for the purposes of enhancing the longevity of the pavement.

1.4. Research Objectives

The objectives of this research are to reveal encapsulated rejuvenators based on the use of Fischer-Tropsch bright stock oil (FTBSO) and waste cooking oil (WCO) for the benefit of the novel idea of self-healing asphalt. Undertaking comparative analysis of their performance while investigating the varying capsule sizes (1 mm, 2 mm, 3 mm) affecting compressive strength, thermal stability, and rupture behavior in wet conditions. Apart from that, the work aims at determining the best capsule size and type that yields durable and efficient crack repair, paving the way for the development of an advanced asphalt technology in the future.

1.5. Research Structure

The following sections in the paper adhere to the following format: In section 2, the work methodology and the used materials are described. It incorporates in detail the process in which the encapsulated rejuvenators were prepared utilizing WCO and FTBSO. In addition, the explanation for the used experimental procedures in the measurement of the wet rupture strength, thermal stability, and the compressive strength. In section 3, the results are presented and the performance test results for the different capsule sizes and composition are discussed. In section 4, the work is summarized with the major findings, referencing the limitations in the study, and providing avenues for further work in the optimization of the encapsulated rejuvenators in self-healing application in asphalt.

2. Experimental Part

This study explores the development and performance of encapsulated rejuvenators for self-healing asphalt, using waste cooking oil (WCO) and Fischer-Tropsch bright stock oil (FTBSO) as rejuvenating agents encapsulated in different sizes. The theoretical approach is grounded in self-healing materials and encapsulation technology, aiming to restore bitumen properties by releasing the rejuvenators upon crack formation. The encapsulated agents are designed to withstand high asphalt mixing temperatures (up to 200° C) and resist mechanical stresses until triggered by cracking. The study evaluates compressive strength, thermal stability, and rupture resistance in wet conditions, hypothesizing that optimal capsule size and composition will enhance fatigue resistance, oxidative aging mitigation, and overall asphalt durability.

2.1. The Used Materials

2.1.1. The Adopted Bitumen

Bitumen 60/70 is a semi-hard penetration-grade bitumen widely used in road construction, asphalt pavements, and waterproofing applications. It is classified based on its penetration value (60-70 dmm at 25°C), which indicates its hardness and consistency. This type of bitumen provides a balance between flexibility and strength, making it suitable for moderate to warm climates. The most important physical properties which needed in this research are listed below in Table 1.

Table 1. The conducted physical properties of the used bitumen

Property	The Result	Typical Range	Test Standard
Penetration (25°C, 100g, 5s)	64	60 - 70 dmm	ASTM D5
Softening Point	51	46 - 54° C	ASTM D36
Ductility (25°C, 5 cm/min)	128	> 100 cm	ASTM D113
Viscosity at 135°C	332	200 - 400 cSt	ASTM D4402

2.1.2. West Cocking Oil (WCO)

It is a byproduct of frying and food processing, primarily composed of degraded vegetable oils and animal fats. It is increasingly used as a bio-based rejuvenator in asphalt to restore aged bitumen's flexibility and reduce brittleness. WCO contains free fatty acids, glycerides, and minor impurities, which influence its physical and chemical behavior when mixed with asphalt. The most important physical properties which needed in this research are listed below in Table 2.

Table 2. The conducted physical properties of the used WCO

Property	The Result	Typical Range	Test Standard
Viscosity (cSt, 40°C)	33	30 - 50	ASTM D445
Acid Value (mg KOH/g)	4.5	1 - 5	ASTM D974
Water Content (%)	0.3	< 1.0	ASTM D6304
Saponification Value (mg KOH/g)	205	180 - 220	ASTM D5558
Boiling Degree (°C)	220	200 – 300	ASTM D2887

2.1.3. Fischer-Tropsch Bright Stock Oil (FTBSO)

FTBSO is a synthetic oil derived from the Fischer-Tropsch (FT) process, which converts syngas (a mixture of CO and H_2) into hydrocarbons. It is a highly refined, paraffinic-based oil with excellent oxidative stability, ultra-low aromatic content, and a narrow molecular weight distribution. Due to these properties, FTBSO is commonly used as a bitumen modifier or rejuvenator in asphalt pavements to restore flexibility, reduce aging effects, and enhance long-term performance. The most important physical properties which needed in this research are listed below in Table 3.

Table 3. The conducted physical properties of the used FTBSO

Property	The Result	Typical Range	Test Standard
Viscosity (cSt, 40°C)	620	500 - 1100	ASTM D445
Pour Point (°C)	- 18	- 5 to - 25	ASTM D97
Aromatic Content (% mass)	< 0.5	< 1.0	ASTM D1319
Saturates Content (% mass)	> 100	> 98	ASTM D2007
Boiling Degree (°C)	430	400 to > 500	ASTM D7169

2.2. Preparation Process of the Used Rejuvenators

Based on previous studies, a mixed blend of WCO or FTBSO with virgin bitumen is more effective for self-healing asphalt than using WCO or FTBSO alone, as it balances softening, enhances durability, improves adhesion, and maintains structural integrity [15]. While WCO or FTBSO can rejuvenate aged asphalt, they may evaporate and oxidize quickly, whereas virgin bitumen ensures long-term performance and prevents excessive softening. This blended approach improves fatigue resistance and meets performance standards, making the asphalt more durable and reliable. The mixing percentages of the used rejuvenators and virgin bitumen, formulated to create more reliable rejuvenators, are (70% WCO or FTBSO + 30% virgin 60/70 bitumen). To mix 70% WCO or FTBSO with 30% virgin 60/70 bitumen, first heat the virgin bitumen to 160° C to ensure proper fluidity. In a separate container, heat the WCO or FTBSO to 120-140°C to lower its viscosity. Gradually add the pre-heated WCO or FTBSO into the hot bitumen while continuously stirring to ensure uniform dispersion. Use a high-shear blending machine as shown in Figure 2, set to 3000-5000 RPM and mix for 10-15 minutes to achieve a homogeneous blend. Maintain the temperature throughout the process to prevent phase separation. Once the mixing is complete, allow the blend to cool gradually to ensure stability and consistency before use.

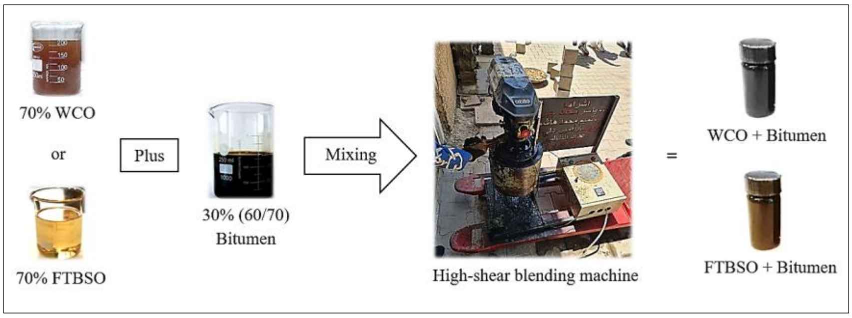


Figure 2. Production process of rejuvenators

2.3. The Process of Encapsulating the Used Rejuvenators

To create the shell of capsules, melamine-formaldehyde (MF) has been adopted for encapsulating WCO or FTBSO + 60/70 bitumen. First, prepare an aqueous solution of melamine and formaldehyde in a molar ratio of 1:1.5 at a concentration of about 5 – 10% by weight, adjusting the pH to around 8 – 9 using an alkaline solution such as sodium hydroxide (NaOH). Add a surfactant, such as sodium dodecyl sulfate (SDS) ($C_{12}H_{25}NaO_4S$), to stabilize the emulsion. Slowly heat the solution to around 60 – 70° C and gradually add the rejuvenator blend while stirring at a high shear rate to form uniform droplets. Once the emulsion is stable, reduce the pH to about 3 – 4 using acetic acid (CH_3COOH) to initiate polymerization, which will harden the MF prepolymer into a solid shell around the rejuvenator droplets. Allow the reaction to proceed for 2–4 hours to fully polymerize the MF, ensuring complete shell formation as shown below in Figure 3. After polymerization, cool the mixture, filter out the capsules, wash them with distilled water to remove any unreacted chemicals, and then dry the capsules at 40 – 60° C until they are solid and stable. These capsules, typically ranging from 1 – 3 mm in diameter as shown below in Figure 4, are now ready to be incorporated into the asphalt mix, where they will release the rejuvenator when cracks form. Capsule uniformity was ensured across the different sizes (1 to 3 mm) and compositions through a controlled encapsulation process. Precise temperature control, pH adjustments, and high-shear stirring were applied to form uniform droplets, while consistent polymerization and careful drying maintained even shell thickness and structural stability for both WCO and FTBSO-based formulations. Melamine-formaldehyde (MF) was chosen as the encapsulation material for its durability, thermal resistance, and chemical stability, ensuring capsule integrity during asphalt mixing and trafficking. Its robust shell structure protects the rejuvenator, contributing to controlled release and effective interaction with the binder, which is crucial for long-term performance.

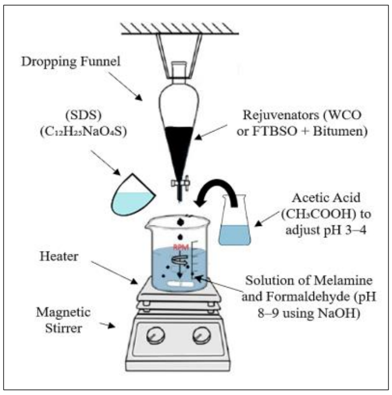


Figure 3. Preparing of rejuvenator capsules

Figure 4. (1 – 3 mm) diameter capsules

2.4. Physical Properties of the Generated Rejuvenator Capsules

2.4.1. Compressive Strength

To test the compressive strength of generated rejuvenator capsules using ASTM E2546 (nanoindentation method) at 20 °C, a nanoindentation device equipped with a Berkovich or spherical indenter as shown below in Figure 5, is used to apply a controlled compressive load on individual capsules (1, 2, and 3 mm) in diameter. The test involves pressing the indenter onto the capsule surface at a precise loading rate (0.2 mm/min), measuring the force-displacement curve until rupture. The compressive strength (σ_c) is calculated using $\sigma_c = F / A$, where F is the peak force at failure and A is the contact area. This method provides high-resolution mechanical properties, including hardness and modulus, making it suitable for evaluating capsule durability. Higher compressive strength values (> 5 MPa) indicate a strong shell, while lower values suggest weaker encapsulation [16], which may affect self-healing performance in asphalt applications.

Figure 5. Hit 300 nanoindentation tester

2.4.2. Thermal Stability

ASTM Method E1952 is a test method for the determination of the thermal stability of those materials exposed to elevated temperatures, and it is most suited for the evaluation of rejuvenator capsules for self-healing asphalt. For the test, the capsules are subjected to the typical temperatures between 150° C and 200° C, simulating the high temperatures during asphalt mixing. The capsules then undergo in a thermal chamber or oven, as shown in Figure 6, for 1-24 hours in short-term stress and 7-30 days for long-term thermal stress [17]. The capsules, after the test, are carefully inspected for physical and chemical degradation, such as rupture, leakage, and structural changes. The weight loss, changes in dimension, and other properties of the material are also measured to determine the effectiveness of the capsules in sustaining its performance at high temperatures, still being able to function for self-healing in asphalt.

Figure 6. (101-2ES) Lab. drying oven

2.4.3. Rejuvenator Release Efficiency (Trigger Mechanism)

In order to determine the rejuvenator release efficiency for waste cooking oil (WCO) and Fischer-Tropsch bright stock oil (FTBSO) encapsulated rejuvenator capsules, the Water Immersion Test (following ASTM D5404) is used in order to test for rupture in hot water (100° C in water bath) as shown below in Figure 7 in order to simulate moisture exposure [18]. The test entails dipping the capsules in the hot water and monitoring the time for each capsule to rupture and release the rejuvenator. The process yields an efficient and pragmatic method for the measurement of the trigger mechanism of encapsulated rejuvenators in self-healing asphalt applications.

Figure 7. Laboratory water bath

Figure 8, displays the flowchart for the work methodology, including the materials used, the process of bitumen modification preparation and mixing, and the rejuvenators encapsulation process.

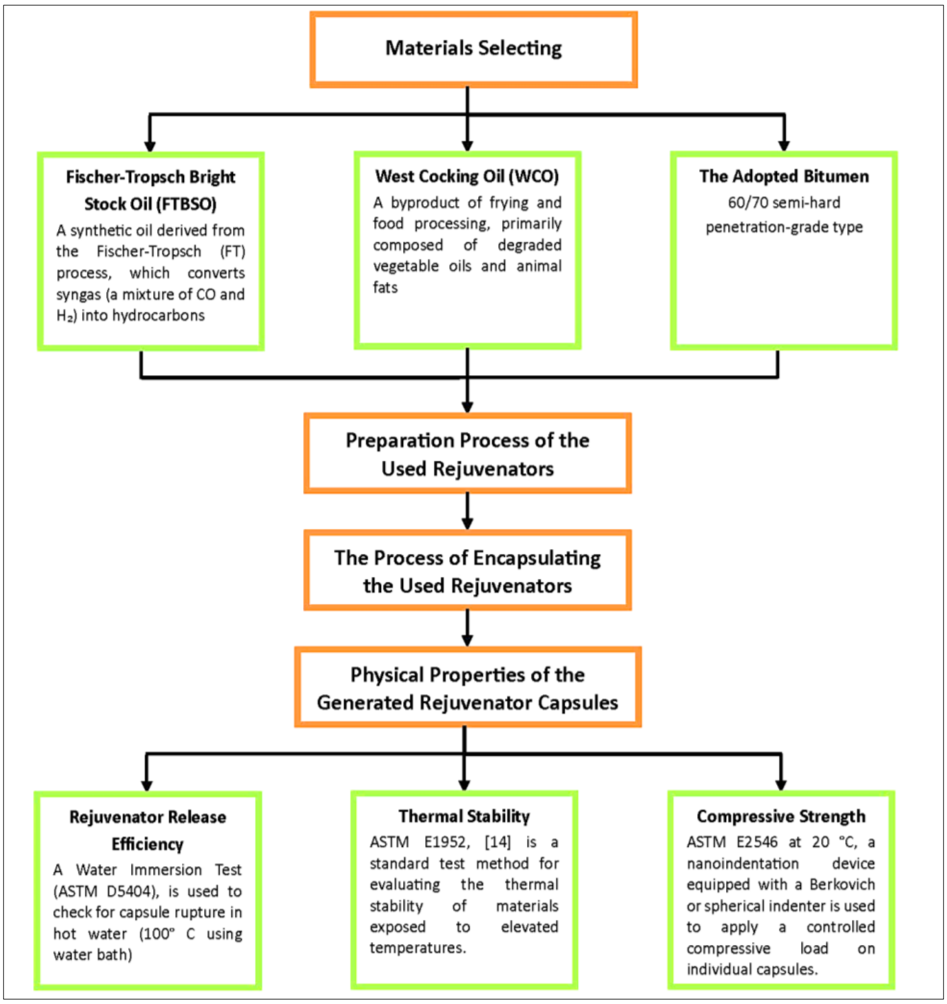


Figure 8. Flowchart of the work methodology

3. Results and Discussion

3.1. Compressive Strength Test Results

Compressive strength tests were performed on 1 mm, 2 mm, and 3 mm encapsulated capsules filled with 70% WCO + 30% bitumen, as well as 70% FTBSO + 30% bitumen, in order to determine their mechanical strength against stress. The findings here reveal that the 3 mm and 2 mm capsules, both WCO- and FTBSO-based, prove to be the strongest, having the ability to sustain high stresses. This is largely due to the greater surface area and the thickness of the encapsulation shell, providing greater strength against deformation under stress application. However, 1 mm WCO capsules were the lowest and least suitable. Furthermore, capsules prepared with WCO showed lower compressive strength and greater displacement, mainly as a result of its softer and less viscous nature, inherently providing less rigidity in its structure. Thus, the capsules become easily deformed under compressive stress. In contrast, FTBSO-based capsules showed greater strength in compression with less displacement. The performance is superior as the chemical nature of FTBSO is rigid in nature and has greater stability in its structure under pressure. These results indicate that both formulations are feasible for self-healing asphalt, with FTBSO-based capsules having greater mechanical strength, hence being more suitable for high load-bearing capacity application. Improved strength and stability not only enhance their performance in the process of asphalt mixing, leading to higher quality mixture, but ensure durability under sustained pavement loading, hence extending the self-healing nature of the modified asphalt mixtures. Similarly, the research conducted by Tabaković et al. [19] investigated encapsulated bitumen rejuvenator in compartmented alginate fibers. Their best fibers, with 70/30 rejuvenator-to-alginate composition, corresponded with some formulations studied in the present work. The fibers were thermally stable and mechanically strong, allowing them to endure the stresses that occur in the course of asphalt compaction and mixture.

The findings of the current study align with the broader trends observed in previous research by Tabaković et al. [19] underscoring the significant influence of rejuvenator type and encapsulation materials on mechanical performance. While both WCO- and FTBSO-based capsules exhibit comparable strength characteristics, FTBSO-based capsules generally show greater resistance under compressive loads. Figures 9 to 12 illustrate the archived results for each size and type of capsules.

An admirable study conducted previously by Micaelo et al. [20] aimed to investigate the impact of calcium-alginate encapsulated rejuvenators on the stiffness, fatigue, and rutting properties of asphalt. This study has recommended a minimum compressive force threshold to be withstood by asphalt self-healing capsules, as shown in Table 4.

Table 4. Minimum applied load on the rejuvenator capsules in self-healing asphalt

Process	Estimated Minimum Load (N)
Asphalt Mixing	30 N
Compaction (Paving)	50 N
Traffic Loading (Initial)	70 N

By comparing the results of the ongoing study with the minimum applied load on rejuvenator capsules, as presented in Table 4, it is concluded that 3 mm and 2 mm capsules of both WCO and FTBSO-based formulations are the most robust, capable of withstanding production and traffic loads, while 1 mm WCO capsules are the weakest and least suitable. The current study expands on previous research by exploring WCO and FTBSO-based capsules of various sizes, broadening the horizon for improved asphalt self-healing and material optimization.

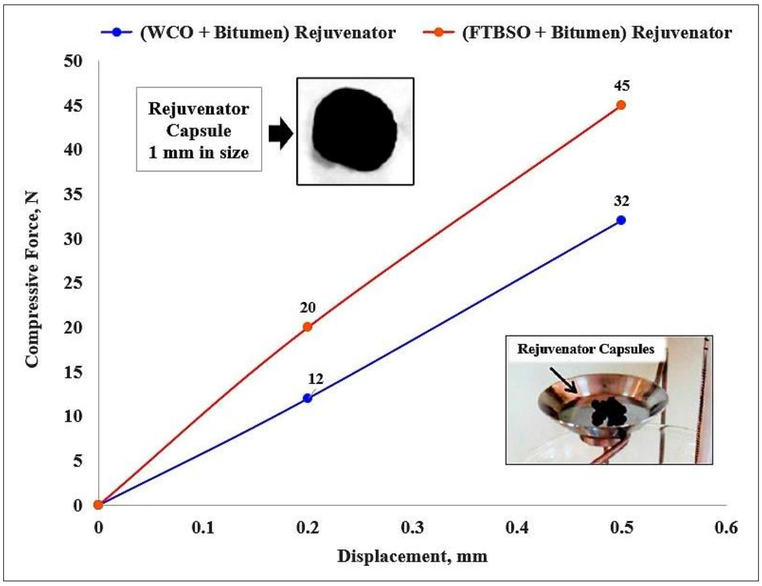


Figure 9. Compressive force vs. displacement or a 1 mm rejuvenator capsule

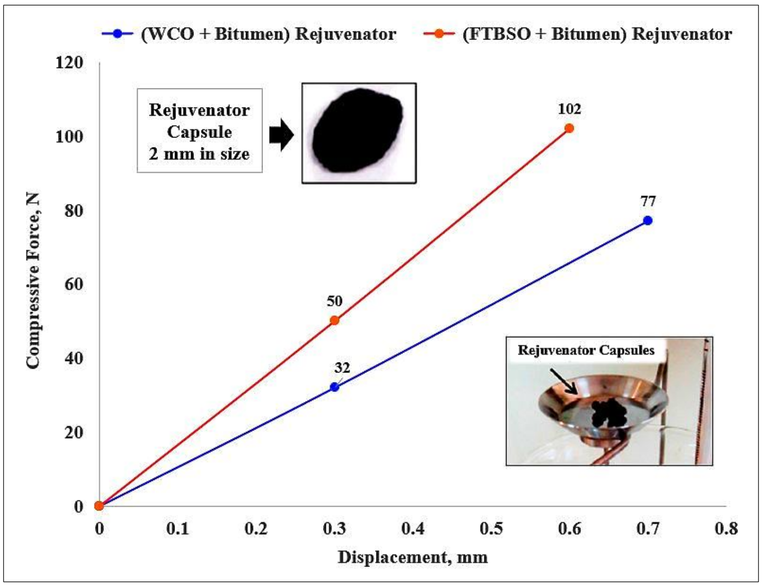


Figure 10. Compressive force vs. displacement for a 2 mm rejuvenator capsule

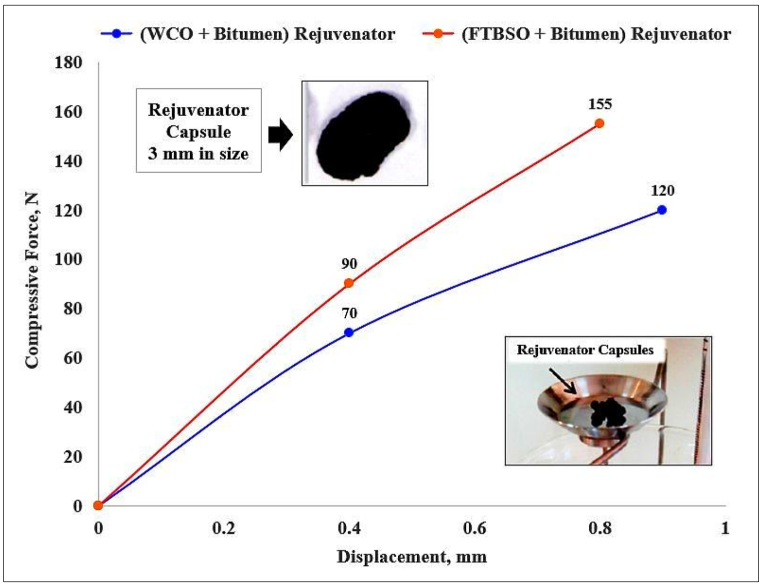


Figure 11. Compressive force vs. displacement for a 3 mm rejuvenator capsule

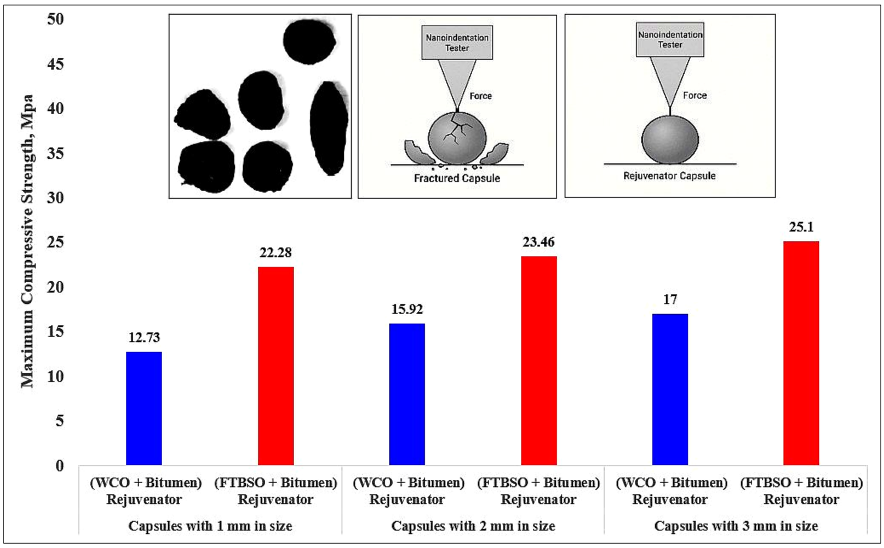


Figure 12. Maximum compressive strength of rejuvenator capsules in terms of size and type

3.2. Thermal Stability Test Results

The thermal stability test for the encapsulated rejuvenators (1 mm, 2 mm, and 3 mm in size) containing either (70% WCO + 30% bitumen) or (70% FTBSO + 30% bitumen) is conducted to evaluate their resistance to degradation, weight loss, and oxidation under high temperatures (typically 150° C – 200° C). The test assesses how the capsules maintain their structural integrity and functionality over a short-term duration (1 hour). Smaller capsules (1 mm) exhibited the highest weight loss and the lowest thermal stability, primarily due to their larger surface-area-to-volume ratio, which increases their susceptibility to oxidation and accelerates degradation. Thus, 1 mm capsules are less ideal for the high-temperature conditions of asphalt mixing. In contrast, 2 mm and 3 mm capsules showed better thermal stability, having much less weight loss and retaining their structure intact. The greater volume and lower surface area exposure of bigger capsules function as protective barriers against quick oxidation, increasing the stability towards high-temperature conditions. Such stabilized rejuvenators remain encapsulated until the desired release at the process of formation of asphalt cracks. FTBSO capsules perform better than WCO-based counterparts because of the better synthetic stability, lower oxidation propensity, and greater strength in the structure, granting them greater thermal stability and the ability to withstand high temperatures, as well as suitability for self-healing asphalt usage. Their greater thermal stability guarantees greater reliability in the blending process for asphalt, as well as prolonged pavement performance, and maintains rejuvenator integrity until crack propagation induces self-healing. Likewise, Norambuena-Contreras et al. [21] investigated the use of WCO-based capsules for self-healing asphalt, focusing on encapsulation techniques, capsule characterization, and short-term thermal stability.

Their work investigated the ability of WCO in order to improve the self-healing property of asphalt through the release of rejuvenators in a controlled manner on the initiation of crack formation. In the present work, FTBSO-based capsules along with WCO-based formulations were used, thereby providing enhanced thermal stability and sustainability. In addition, thermal stability tests were further carried out for short (1-hour) exposure, with the focus being the impact of capsule size (1 mm, 2 mm, and 3 mm) on structural integrity, softening, and weight loss at high temperatures. The thorough analysis of capsule size differences and the short-term performance provides new insights into self-healing asphalt technology and the durability evaluation of encapsulated rejuvenators. Figures 13 to 15 illustrate the archived results for each size and type of capsules.

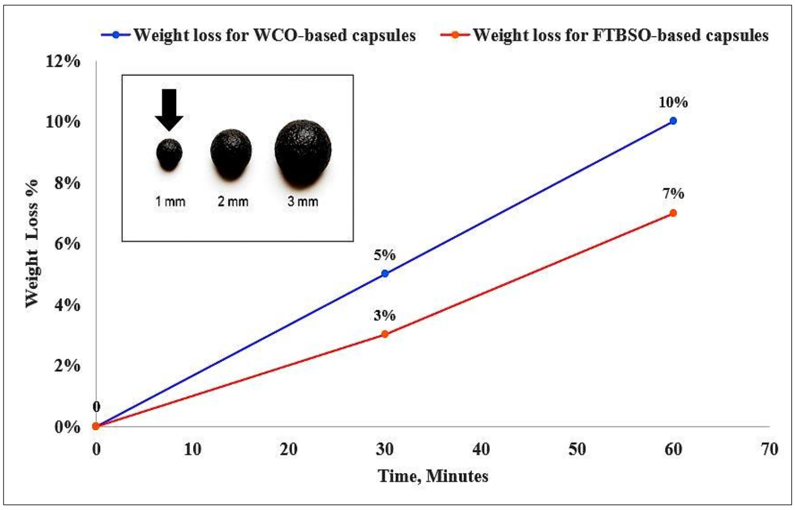


Figure 13. Weight loss vs. time for a 1 mm rejuvenator capsule

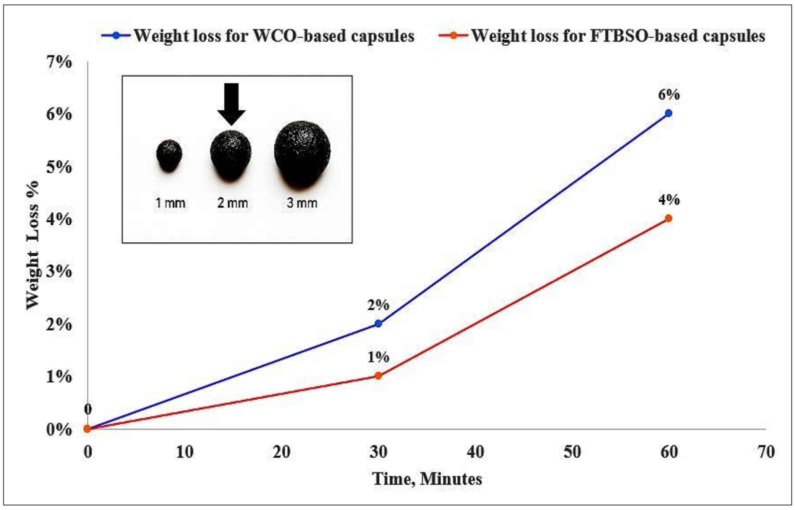


Figure 14. Weight loss vs. time for a 2 mm rejuvenator capsule

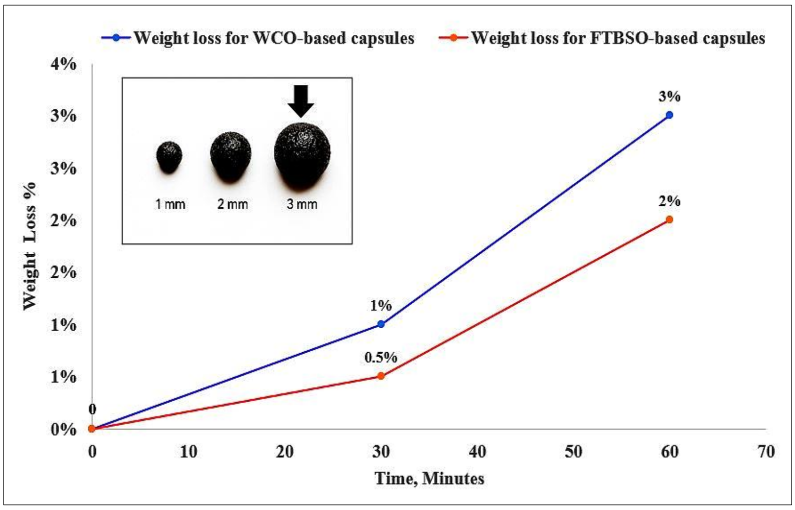


Figure 15. Weight loss vs. time for a 3 mm rejuvenator capsule

3.3. Rejuvenator Release Efficiency Test Results

The water immersion test at 100° C evaluates the rupture times of rejuvenator capsules for self-healing asphalt, containing WCO and FTBSO, in 1 mm, 2 mm, and 3 mm sizes. The capsules were tested for moisture-induced degradation to assess their durability and release efficiency. Figure 16, below summarizes the results of the water immersion test for the capsules.

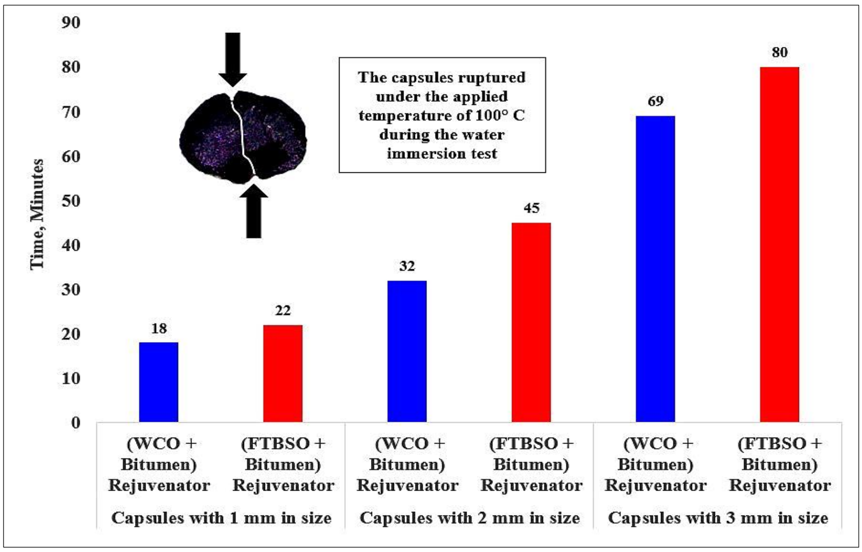


Figure 16. Time vs. rupture occurrence in the water immersion test

FTBSO capsules took longer to rupture than WCO capsules during the water immersion test at 100°C. This delay is due to FTBSO's significantly higher boiling point (above 400° C), resulting in negligible vapor pressure within the capsule, thus relying primarily on gradual, water-induced shell hydrolysis to trigger rupture. In contrast, WCO, with a lower boiling range of 200 – 300° C, generates internal vapor pressure more readily, accelerating shell degradation and hastening rupture. Regarding their applicability in self-healing asphalt, the enhanced stability and slower rejuvenator release from FTBSO capsules may be advantageous for sustained long-term healing, whereas the quicker rejuvenator release from WCO capsules could offer benefits for rapid crack repair. Therefore, optimization of both capsule types is necessary based on specific pavement performance requirements. Additionally, both formulations are capable of withstanding typical asphalt mixing temperatures of around 160° C.

A study conducted by Wang et al. [22] investigated the thermal stability and mechanical rupture of calcium alginate capsules encapsulating rejuvenators such as sunflower oil, demonstrating stability up to 200° C under dry conditions and effective healing under compressive stress. However, the current tests introduced a novel moisture-induced durability assessment conducted at 100° C. The capsules evaluated in this research exhibited the ability to withstand typical asphalt mixing temperatures of 160° C, consistent with the thermal resilience observed in prior studies. Nevertheless, observed rupture times (18–80 minutes) underscore water's significant accelerating influence on capsule shell degradation. This bridged a gap by revealing WCO's faster rupture due to volatility and FTBSO's slower release due to stability, enhancing the understanding of moisture's role in capsule performance for self-healing asphalt.

The behavior of encapsulated rejuvenators, composed of either 70% WCO or 70% FTBSO combined with 30% bitumen, during thermal stability tests is influenced primarily by their capsule size and rejuvenator composition. Smaller capsules display reduced resistance to degradation and oxidation due to their higher surface-area-to-volume ratio, which facilitates greater exposure to heat and oxygen. In contrast, larger capsules exhibit enhanced thermal stability by minimizing this exposure. Furthermore, the rejuvenator type significantly impacts thermal performance: WCO, being an organic oil, is inherently more prone to thermal breakdown and oxidation, whereas FTBSO, a synthetic oil, provides greater thermal and oxidative stability. Based on the previously discussed results on weight loss for all sizes and types of rejuvenator capsules, the degree of degradation and oxidation can be summarized as shown below in Table 5.

Table 5. The degree of degradation and oxidation of rejuvenator capsules in terms of type and size

Capsule Size	Composition	Resistance to Degradation	Reasons Behind that	Resistance to Oxidation	Reasons Behind that
1 mm	70% WCO + 30% Bitumen	Poor	Small size increases surface-area-to-volume ratio, enhancing heat penetration. WCO's natural instability accelerates breakdown.	Low	WCO's unsaturated fatty acids and impurities are highly reactive, promoting rapid oxidation at 160°C.
2 mm		Moderate	Larger size reduces surface exposure, but WCO's inherent thermal instability still causes degradation over time.	Low to Moderate	WCO oxidation occurs due to unsaturated fats, though larger size slightly limits oxygen access.
3 mm		Good	Largest size minimizes heat and oxygen penetration. WCO's instability is mitigated but not eliminated.	Moderate	Reduced oxidation due to lower surface area, but WCO's reactive components still degrade over long exposure.
1 mm	70% FTBSO + 30% Bitumen	Fair	Small size increases heat exposure. FTBSO's stability delays degradation, but size limits overall resistance	High	FTBSO's synthetic, saturated hydrocarbons resist oxidation, though small size allows some oxygen penetration.
2 mm		Good	Larger size reduces heat and oxygen effects. FTBSO's thermal stability enhances resistance	High	FTBSO's stable molecular structure minimizes oxidation, with size further limiting oxygen exposure.
3 mm		Excellent	Largest size and FTBSO's synthetic nature greatly reduce heat penetration and degradation.	Very High	FTBSO's lack of reactive sites and large size prevent significant oxidation even over long periods.

4. Conclusions

The conclusions drawn from the evaluation of encapsulated rejuvenators, utilizing two distinct types (70% waste cooking oil (WCO) + 30% bitumen) and (70% Fischer-Tropsch bright stock oil (FTBSO) + 30% bitumen), across three sizes (1 mm, 2 mm, and 3 mm), focus on their performance in terms of compressive strength and thermal stability. This analysis gives important insight into the suitability of these capsules for the self-healing application in asphalt, demonstrating how the size, along with composition, affects the mechanical stability and durability against degradation at high temperatures. Recommendations as described below follow from the findings and above discussions:

- Compressive strength increases with larger capsule sizes, with 3 mm and 2 mm capsules demonstrating the highest resistance to mechanical stress, while 1 mm capsules remain the most fragile. Moreover, FTBSO-based capsules outperform WCO-based ones due to their higher viscosity and greater structural stability.
- The thermal stability of encapsulated rejuvenators is significantly influenced by capsule size and rejuvenator type. The 3 mm FTBSO-based capsules demonstrated superior resistance to thermal degradation, with only 2% weight loss at 200° C, while 1 mm WCO-based capsules showed the highest degradation, with a 10% weight loss under the same conditions.
- WCO-based capsules demonstrate lower thermal stability in comparison to FTBSO, due to the natural oil's unsaturated fatty acids and impurities, which promote oxidation, with 1 mm capsules being the least resistant.
- FTBSO-based capsules outperform WCO counterparts, with excellent thermal stability driven by FTBSO's synthetic, saturated hydrocarbons, making 3 mm FTBSO capsules highly resistant to degradation and oxidation.
- Capsules with WCO exhibited faster rupture than those with FTBSO due to WCO's volatility-induced degradation compared to FTBSO's greater stability under moisture conditions.

In short, 3 mm FTBSO-based capsules yield the best performance, providing superior compressive strength, thermal stability, and tailored rejuvenator release, with durability for asphalt mix and compaction. In comparison, 1 mm capsules perform the worst, with WCO-based capsules being especially likely to rupture and deteriorate early. On the other hand, 2 mm FTBSO-based capsules yield balanced performance, with durability matched with effective self-healing properties.

The work highlights the need for capsule design optimization for structural and thermal stability, with the assessment of the compressive strength, thermal stability, and rupture resistance being the critical inputs for the selection of the right material in asphalt technology. The principal limitations in the present study pertain to the absence of stress evaluation under sustained environmental conditions like moisture, sunlight, and cyclic traffic loading. Additionally, it did not include simulated asphalt mixing tests to assess survivability under shear forces, relying instead on compressive strength and rupture tests to approximate these stresses. The absence of thermal cycling and extended mechanical fatigue tests also limits the understanding of capsule performance under real-world conditions. Subsequent works should seek to fill the above gaps, making an all-round analysis on durability and self-healing effectiveness in actual asphalt usage.

5. Declarations

5.1. Author Contributions

Conceptualization, T.M.H.; methodology, T.M.H.; formal analysis, T.M.H.; investigation, T.M.H.; resources, T.M.H.; data curation, T.M.H.; writing—original draft preparation, T.M.H.; writing—review and editing, F.F.A.; visualization, T.M.H.; supervision, F.F.A.; project administration, F.F.A.; funding acquisition, F.F.A. All authors have read and agreed to the published version of the manuscript.

5.2. Data Availability Statement

The data presented in this study are available on request from the corresponding author.

5.3. Funding

The authors received no financial support for the research, authorship, and/or publication of this article.

5.4. Conflicts of Interest

The authors declare no conflict of interest.

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