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RESEARCH ARTICLE-CIVIL ENGINEERING

Rheological and Morphological Characterization of Cup Lump Rubber-Modified Bitumen with Evotherm Additive

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Abstract

Natural rubber (NR) in the form of "cup lump" is used to significantly enhance the thermal stability and elasticity of bitumen. Despite these benefits, the paving industry has raised concerns about its increased energy use and carbon emissions when applied in hot mix asphalt (HMA). Warm mix asphalt (WMA) was invented to reduce this negative effect. In WMA, an additive is first added to the bitumen, which acts as a surfactant and allows the production and compaction of asphalt mixture at temperatures up to 50 °C less. As a result, the amount of energy consumed and carbon dioxide emissions during asphalt production are considerably reduced. In this study, CLR-modified bitumen (CMB) was blended with five percentages of Evotherm warm mix additive (0.3, 0.4, 0.5, 0.6, and 0.75%), and the properties were examined. According to the findings, the Evotherm modification lowered the viscosity of the binder by 26% and the contact angle by 6°, while the binder's crack resistance at low temperatures marginally improves. Quantitative analysis from Fourier transform infrared spectra revealed a reduction in C=C stretch, C−O stretch, and C−H absorbance in response to the addition of Evotherm. Also, atomic force microscopy (AFM) scan shows an increase in the number of catana phases with a separation of peri- and para-phases. Only CMB with 0.75% Evotherm possesses 100% aggregate coating with sufficient air void; hence, it was selected as the optimum to be used in producing the warm mix asphalt mixture. Overall, the mixing temperature of the CMB can be lowered by 40 °C.

Keywords Warm mix asphalt · Natural rubber · Cup lump · Polymer-modified bitumen · Wettability · Evotherm

1 Introduction

The aggregate content of hot mix asphalt (HMA) is around 95% and the bitumen content is approximately 5%. Despite the fact that bitumen makes up such a small fraction of the total, it dictates the flexible pavement's performance.

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Natural rubber (NR) has been used to improve bitumen performance for almost a century; in fact, the use of NR for bitumen modification was patented in England in 1840. In 1920, the United Kingdom and the United States made several attempts to construct rubberized roadways without the use of bitumen [1]. NR is a renewable, economic elastomer with outstanding thermal stability and elasticity. Its application in bitumen modification increases the rutting resistance, fatigue life, dynamic stability, resistance to moisture damage, and low-temperature cracking resistance of a pavement [2–4]. Currently, Malaysia is committed to increasing NR consumption and increasing the price of NR through road construction. More than 1000 kms of cup lump-modified asphalt (CMA) roads were built to increase NR domestic consumption. The project consumed about 42,000 metric tons of NR [5]. Cup lump rubber (CLR) is a coagulated NR that has not gone through any production processes [6]. The use of CLR-modified bitumen (CMB) in road construction gained popularity after CMA roads were constructed in Malaysia in 2015. Laboratory and field results obtained after two years of monitoring show improvements in resilience and durability, rutting resistance, and cracking performance compared the conventional HMA [7]. Despite the CMA's excellent performance, its use in HMA raised the cost of asphalt manufacturing by 80% due to the higher amount of heating energy required during the mixing and compaction of the CMA. According to [8, 9], the high cost of rubberized asphalt pavements is due to the increased energy consumption when compared with the standard asphalt mix. The authors found that increasing the asphalt manufacturing temperature directly leads to a corresponding increase in the quantity of total energy consumed, thereby resulting in a higher asphalt production cost. Therefore, the conversion of CMA to warm mix asphalt (WMA) is anticipated to substantially lower the mixing and compaction temperatures, cost, and fume emissions without affecting the in-service performance of the CMA mix. WMA is a technology that reduces HMA production temperature by up to 50 °C by adding chemicals that reduce the viscosity of the bitumen or increase its volume, thereby facilitating aggregate coating to be achieved at a lower temperature than HMA [8–11].

Despite the several advantages of WMA over HMA, little is known about the morphology of NR-modified bitumen with warm mix additives due to earlier research focusing on HMA [12]. Asphalt binders are typically subjected to tests in one of the three categories: physical, rheological, and morphological. Physical tests are empirical procedures used to ascertain the consistency of the binder. Because bitumen is viscoelastic, rheological tests are carried out to determine the binder's resistance to flow. Morphological tests are used to analyze the interaction between bitumen and different bitumen modifiers. Morphological tests aid in explaining or understanding the bitumen's rheological results and the mechanical performance test results of the asphalt mixture. The literature matrix presented in Table 1 examined the application of NR-modified bitumen; it confirms the lack of sufficient literature investigating the application of CLR-modified bitumen with Evotherm warm mix additive. Only Sani et al. [15, 16] analyzed the properties of NR latex-modified binders with Zycotherm warm mix additive. They discovered that NR latex-modified binders have a disjointed agglomerative structure with long voids, indicating high a moisture content that tends to lower the shear stress resistance of the binder. NR latex contains more than 40% water content, and its blending with bitumen produces foam in large quantities which generates concerns about the moisture damage susceptibility of the binder. As a result, using CLR with lower water content will be more beneficial in improving bitumen performance. Abdulrahman et al. [13, 14] investigated the physical properties and mechanical performance of cup lump rubber-modified bitumen in another study. They discovered that the WMA had outstanding rutting resistance (rut depths of less than 2 mm at 8000 load cycles), which represents a 21% improvement over the conventional HMA. In addition, moisture damage analysis revealed that the WMA has a tensile strength ratio (TSR) of 99 percent and the aggregates preserved over 90 percent of the bitumen coating. The production of CMA at a lower temperature reduced the CO_2 by 5 kg, resulting in a 23% reduction in the global warming potential of the asphalt mixture. Despite its enormous success, the rheology and morphology of the Evotherm-modified bitumen remained unknown. Hence, the current study intends to fill this research gap. The present study explored the properties of CLR-modified bitumen incorporating Evotherm warm mix chemical. The first step toward achieving long-term use of NR in pavement construction is determining the binder properties. Also, because of the complicated interactions that exist between the combination of bitumen, rubber, and WMA additives, characterization of the binders first is crucial before applying them at the mix design level [15]. This research becomes necessary since bitumen modification with NR increases viscosity, which necessitates greater mixing and compaction temperatures during the asphalt manufacturing process. As a result, an additional 2.5 kg of CO_2 per ton is produced for society, as well as CO, NOx, SO_2, and other environmental contaminants. Likewise, it consumes more heating energy (0.5–1.0 L/ton) thus raising the entire asphalt production cost [16, 17]. As such, the asphalt production industry should reduce the production temperature of HMA without affecting pavement performance [18]. The outcome of this study will provide clearer knowledge on the effects of bitumen modification with Evotherm and promote WMA in the asphalt industry.

Table 1 Literature matrix summarizing the application of natural rubber-modified binder

Author	Natural rubber type	Bitumen properties			Mixture Performance			
		Physical	Rheology	Characterization	Rutting	Moisture damage	Fatigue cracking	Thermal cracking
Abdulrahman et al. [13, 14]		✓	Gap to be filled by the current research		✓	✓	✓	Research Area yet to be fully explored
Ghafar et al. [19, 20]	Cup lump	✗	✗	✗				
Mazlina Mustafa Kamal et al. [21]		✓	✓					
Hazoor Ansari et al. [22]		✗	✗	✗				
Azahar et al. [23–25]		✗	✗	✗	✗		✗	
Razali et al. [26]					✗		✗	
Othman et al. [5]		✗	✗		✗			
Shaffie et al. [27]					✗			
Sani et al. [28, 29]	Latex	✓	✓	✓				
Abu Bakar et al. [30]		✗	✗					
Siswanto [4]					✗			
Shafii et al. [31]		✗						
Shaffie et al. [2]					✗			
Siswanto [32]					✗			
Krishnapriya [33]		✗	✗		✗	✗	✗	
Wen et al. [34]		✗	✗	✗				
Shaffie et al. [3]						✗		
Tuntiworawit et al. [6]		✗	✗	✗	✗		✗	
Tinavallie [35]		✗		✗				

✓ = With warm mix additive ✗ = Without warm mix additive

2 Materials and methods

2.1 Materials

Cup lump rubber-modified bitumen (CMB) produced in the laboratory was modified with five percentages of Evotherm in this study. Table 2 summarizes the properties of the CMB used and shows that the CMB has properties similar to PG82. The chemical Evotherm, made by Ingevity in the United States, was utilized as a surfactant warm mix additive. It

Table 2 Properties of the cup lump rubber-modified bitumen (CMB)

Method	Test	Value
ASTM D5	Penetration at 25 °C (dmm)	36
ASTM D36	Softening point (°C)	54
ASTM D4402	Viscosity at 135 °C (Pa s)	1.9
ASTM D113	Ductility (cm)	94
ASTM D6084	Elastic recovery (%)	31

Fig. 1 Evotherm warm mix additive used for this research

is an oily, dark liquid that is just slightly water-soluble, as shown in Fig. 1. The parameters of the Evotherm employed in this investigation are listed in Table 3.

2.2 Bitumen preparation

First, CMB is produced by blending 60/70 penetration grade bitumen with five percent cup lump rubber (in gel form) using a high shear mixer (at 4000 revolutions per minute) at 160 ± 10 °C for two hours. More detail on the process of converting solid rubber into gel is reported in [14]. A storage stability test in accordance with the ASTM D7173 standard was conducted to examine the tendency of separation; the result shows that the CMB is stable (the difference between the top and bottom softening temperatures is 1 °C). In the second stage, the CMB is modified with 0.3, 0.4, 0.5, 0.6, and 0.75% of Evotherm using a high shear mixer at 160 °C for five minutes. The Evotherm dosage of (0.3–0.75%) and blending time were selected based on the manufacturer's recommendation. Figure 2 presents the experimental flowchart used for the study. Physical, rheological, and morphological tests were conducted to characterize and compare the properties of the modified binders at high, intermediate, and low temperatures. For each test conducted in this study, at least three replicate samples were produced and tested, and the average value is reported. Error bars are provided to show the variability of the data obtained.

2.3 Experimental methods

2.3.1 Penetration and softening point

A penetration test is performed to determine the consistency of a binder by applying a load of 100 g from a standard needle into a mold filled with a bitumen specimen in a water bath maintained at 25 °C for 5 s [36]. A softening point test was conducted to identify the transition temperature of bitumen from a semi-solid state to a semi-liquid. At the transition temperature, the bitumen can no longer maintain its mass and begins to deform. The test was carried out in accordance with ASTM D36 [37].

2.3.2 Ductility and elastic recovery

In the ductility test, a briquet of bitumen sample is pulled at 50 cm/min in a 25 ± 5 °C water bath. The ductility value is the length the material elongates before breaking [38]. One drawback of this test is that it only examines the bitumen's capacity to stretch but not its ability to rebound. As a result, an elastic recovery test was performed in line with ASTM D6084 to assess the binders' capacity to recover after deformation [39]. Figure 3 presents an image of the prepared sample.

2.3.3 Viscosity

The modified binders were put through a viscosity test to guarantee that they could be blended easily at production and construction temperatures. The test was carried out using a Brookfield rotating viscometer (DV-II + Pro) and test specimens were prepared according to ASTM 4402.

2.3.4 Dynamic shear rheometer

The bitumen stiffness, viscoelastic behavior, and resistance to deformation under load are determined using a dynamic shear rheometer (DSR) at 46–88 °C different pavement temperatures and a loading rate of 10 rad/s (1.59 Hz) [40, 41]. In this test, a sample of bitumen is inserted between the geometries of a HAAKE rheometer and subjected to a sinusoidal

Table 3 Physical and chemical properties of Evotherm [14]

Physical state	Color	Odor	Specific gravity	pH values	Boiling point	Flashpoint (closed cup)
Viscous oil	Dark brown	Amine-like	1.03–1.08	10–12	> 200 °C	> 260 °C

Fig. 2 The study's experimental flowchart

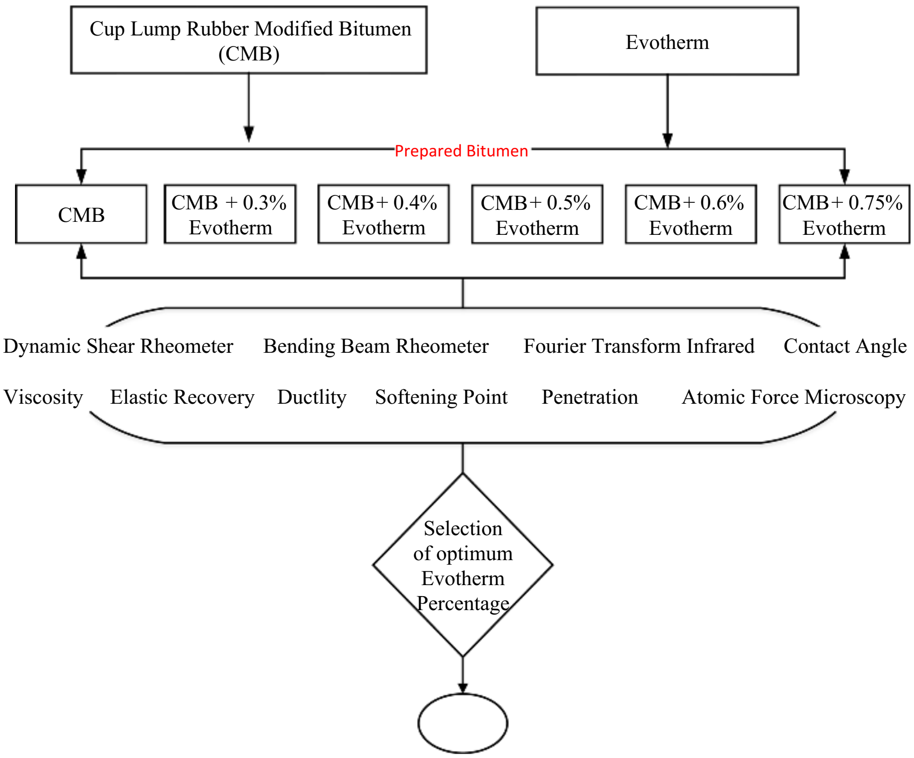


Fig. 3 Cup lump rubber-modified sample prepared for ductility test

strain. The applied strain's reaction is the creation of matching sinusoidal stress, but the material's response is out of phase with the applied strain, referred to as the phase angle (δ). This test revealed two properties of bitumen: rutting and fatigue resistance [42, 43].

2.3.5 Bending beam rheometer

The probability of crack development increases when pavement is exposed to low pavement temperatures due to the bitumen's transition from a time-dependent ductile to a brittle behavior [44]. The creep stiffness behavior of bitumen may be obtained using a bending beam rheometer (BBR) test. BBR test was conducted in line with the ASTM 6818 standard [45]. The *m*-value and creep stiffness of the bitumen are determined after a loading duration of 60 s [42, 46].

2.3.6 Fourier transform infrared spectroscopy

Using attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopy, the chemical alterations in the modified bitumen were examined. To investigate the oxidative aging of asphalt binders, carbonyl (C=O) and sulfoxide (S=O) absorption, as well as carbon–carbon (C=C) stretching vibrations of the aromatic ring, are all measured [43, 44]. The region covered by the peaks (from valley to valley) was used for quantitative analysis of the IR spectra using TQ Analyst software [45, 46]. Carbonyl oxidation (A_{1700}/A), aromaticity index (A_{1600}/A), aliphatic ($A_{1452} + A_{1373}/A$), presence of a sulfoxide group (A_{1030}/A), aromatic heteroatoms ($A_{874} + A_{809} + A_{747})/A$, and lengthy chains ($A_{720}/A_{1452} + A_{1373}/A$) are among them. The total area for each IR spectra is calculated using Eq. 1.

$$\begin{aligned}\Sigma A = {} & A_{2950} + A_{2860} + A_{1700} + A_{1600} + A_{1452} \\ & + A_{1373} + A_{1030} + A_{874} + A_{809} + A_{747} + A_{720} \qquad (1)\end{aligned}$$

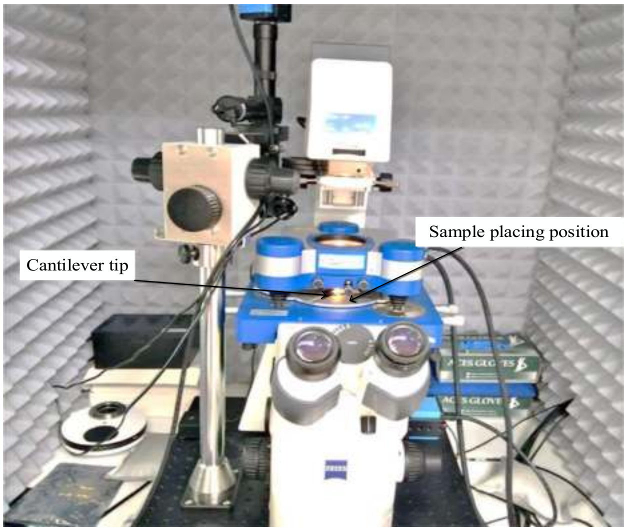


Fig. 4 Atomic force microscopy test setup

2.3.7 Atomic force microscopy

The atomic force microscopy (AFM) test is used to determine the forces generated between a cantilever beam's tip and the material being tested as a function of the distance between the two structures [47]. Test samples were made by heating bitumen to 135 degrees Celsius until it became fluid enough. A bitumen sample of around 2 mm diameter is dropped on a clean microscope glass slide, which is then returned to the oven at 135 °C for 5 min to provide a flat surface [48]. During testing, the system generated a two- and three-dimensional surface profiles of the bitumen specimen using the tiny cantilever tip (in a non-contact mode of operation). The phase matrix included in the tested specimen is depicted in 2D, while the three-dimensional topography (3D) offers data on the relative heights of the various characteristics present in the bitumen sample [49]. Figure 4 presents the atomic force microscopy test setup.

2.3.8 Contact angle

One of the major issues with WMA's durability is the possibility of moisture-induced bond breakdown. Because of the lower mixing temperature, a thin water layer forms at the aggregate—bitumen point of contact. Failures at this critical contact are most likely caused by the existence of water in the aggregates or outside the bitumen coated particle [50–52]. As a result, a robust adhesion bond connecting the aggregate and the bitumen is critical. Anything that compromises this bond will shorten the pavement's intended life span. The addition of warm mix additive to bitumen can change the binder's adhesive properties. Hence, it is important to look at the binders' ability to attract moisture at the bitumen-aggregate interface, which tends to lower the bitumen-aggregate adhesion bond [51, 52]. Samples for the contact angle test were prepared by heating the bitumen in a conventional oven until it turns to liquid, which is then applied to a 3 cm × 5 cm piece of tiny glass. A micro-syringe needle is used to release a sessile drop of distilled water from a height of 5 mm. Then, a video contact angle measuring device (VCA optima) was employed to capture the sessile drop image, and the left and right contact angles were determined. Five replicate samples were prepared for each of the bitumen produced in the study. The repeatability of the measurement was estimated using a single test method, and the results show that the variability ranges from 1.38 to 2.59, which is within the recommended limit of ± 5° [53, 54]. Figure 5 shows the contact angle measurement process.

3 Results and discussion

3.1 Penetration and softening point

The addition of 0.3–0.75% Evotherm continuously increased the penetration amount of the CMB, as depicted in Fig. 6, while a marginal decrease in softening point temperature was observed at 0.6–0.75% Evotherm content. Because the Evotherm tends to dilute the CMB, resulting in the formation of bitumen with a softer matrix, an increase in penetration value with decreasing softening point temperature is expected. This can be attributed to the lower melting and boiling point components of the Evotherm additive [55]. It was observed that the softening temperature was not affected by the addition of 0.3–0.5% Evotherm content. This is because during the blending of CMB with Evotherm, rubber particles tend to absorb the freely available Evotherm oil and cause swelling until the saturation limit is reached at 0.5% Evotherm content [56]. Furthermore, thermal analysis of NR using a differential scanning calorimeter reveals that the first endothermic peak and mass loss at 50 °C are caused by water evaporation from the NR cavities, and flowable bitumen constituents (saturates and aromatics) permeate and fill the pores left in the NR [57]. This interaction occurs at a temperature similar to the softening point testing temperature (50–60 °C); as such, Evotherm oil flows and fills the pores, hence the reason for the lack of decrease in the softening temperature of the bitumen at 0.3–0.5% Evotherm content. But adding 0.6 and 0.75% Evotherm causes the softening point temperature to decrease, signifying that the pores have reached saturation level.

3.2 Ductility and elastic recovery

The results of the ductility and elastic recovery tests are shown in Fig. 7. First, it is worthy of mention that the base

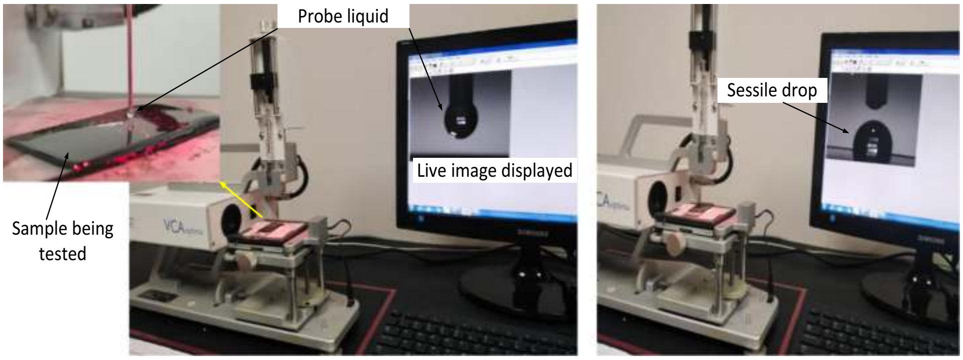


Fig. 5 Contact angle test setup used in measuring the affinity of the bitumen

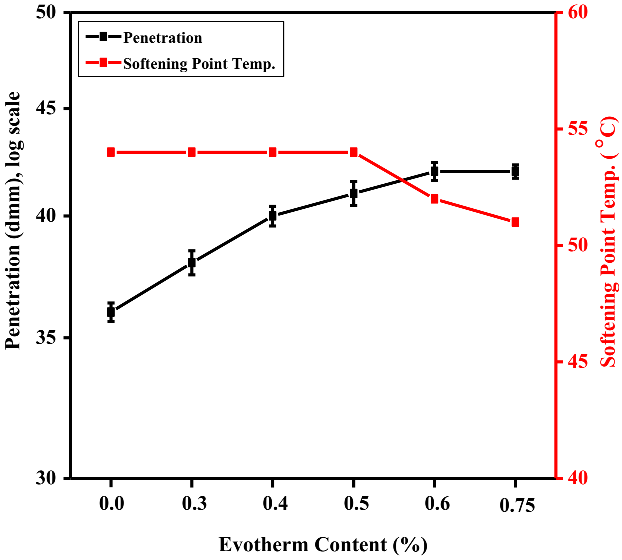


Fig. 6 Penetration and softening point temperature test result

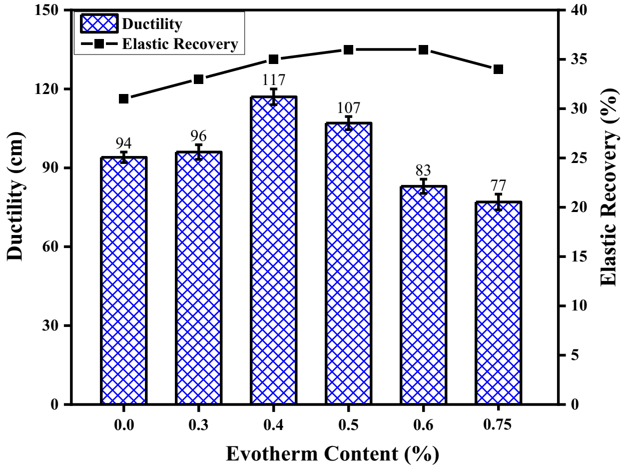


Fig. 7 Ductility and elastic recovery result

60/70 PEN bitumen used in this research has a ductility value 120 cm. The addition of 5.0% CLR reduced the ductility value to 94 cm. This can be attributed to the absorption of light oils by the CLR from the bitumen as a result of the bitumen–rubber interaction during the blending process [56]. Hence, the addition of rubber leads to the production of stiff bitumen with less stretching ability. The addition of 0.3 to 0.5 percent Evotherm shows an increase in the ductility value, indicating an improvement in the stretching ability of the CMB. At 0.6 and 0.75% Evotherm content, the ductility decreased. This demonstrates that a lower Evotherm dosage increased CMB stretching ability by lowering stiffness, whereas a higher dosage reduced the stretching ability of the CMB. A similar increase in ductility with the addition of Evotherm was reported by [58]. This is because the blending of conventional bitumen with CLR stiffens the bitumen, leading to improved high-temperature performance, but renders the bitumen brittle at 25 °C testing temperature due to a reduction in cohesion [22, 56, 59]. Thus, incorporating Evotherm into the blend tends to reduce the stiffness of the CMB (by replenishing the light oils absorbed by the CLR) until full saturation level is attained at 0.5% Evotherm content. Similarly, the elastic recovery curve showed a tendency in which increasing Evotherm concentration improves the CMB's recoverability until a maximum value is reached at 0.6 percent Evotherm. According to [60], a bitumen should have at least 60% elastic recovery; however, none of the tested binders met this requirement.

3.3 Viscosity

Figure 8 presents the viscosity test results. It demonstrates that adding 0.3 percent Evotherm to CMB reduced viscosity by 10% at 135 °C, whereas increasing Evotherm dosage to 0.4 and 0.5 percent results in a four percent additional reduction in viscosity. A cumulative reduction of 26% in viscosity was achieved at 0.75% Evotherm content, indicating that it is possible to use lower mixing and compaction temperatures for the CMB asphalt mixture. The viscosity of the CMB decreased by 11% when the testing temperature was raised to 165 °C, regardless of the Evotherm concentration. This is because the warm mix additive functions as a surfactant by lowering the surface tension between aggregate and bitumen. As such, its influence on viscosity is sometimes undetectable

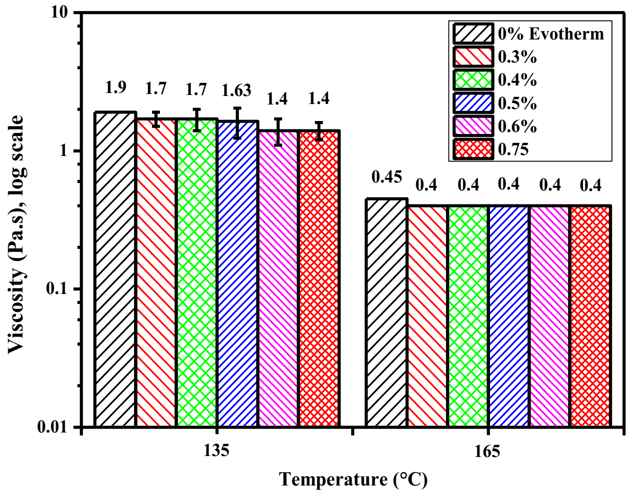


Fig. 8 Viscosity result of CMB with various percentages of Evotherm

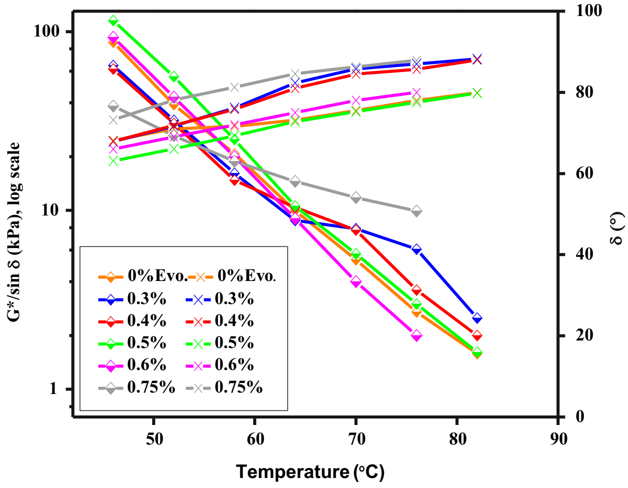


Fig. 9 Isochronal graph of rutting resistance and phase angle at a constant rate of recurrence of 1.59 Hz

at 165 °C [55, 61–63]. A similar slight decrease in viscosity was obtained by [64–66].

3.4 Dynamic shear rheometer

Figure 9 shows the isochronal graphs of rutting resistance and phase angles of the tested binders. The specification limit of $|G^*|/\sin(\delta) = 1$ kPa was employed to establish the bitumen's rutting resistance [67]. It shows that the CMB has a rutting resistance of 82 °C which is similar to the rutting resistance of the bitumen with 0.3 to 0.5% Evotherm. Meaning the addition of 0.3–0.5% Evotherm did not affect the rutting resistance of the CMB. This can be attributed to the low Evotherm dosage incorporated into the blend (3 to 5 g for every 1000 g of CMB). However, at 0.6 percent and 0.75 percent Evotherm content, rutting resistance decreased by one grade. This is due to the high Evotherm content, which tends to dilute and reduce the viscosity of the CMB. Similar finding was reported by [58]. On the contrary, [65] discovered that Evotherm raised the failure temperature of polymer-modified bitumen. A similar trend was observed in the phase angle curves, where Evotherm marginally reduced the elastic response of the CMB. As a result, the Evotherm modification has no significant negative impact on the binders' high-temperature performance.

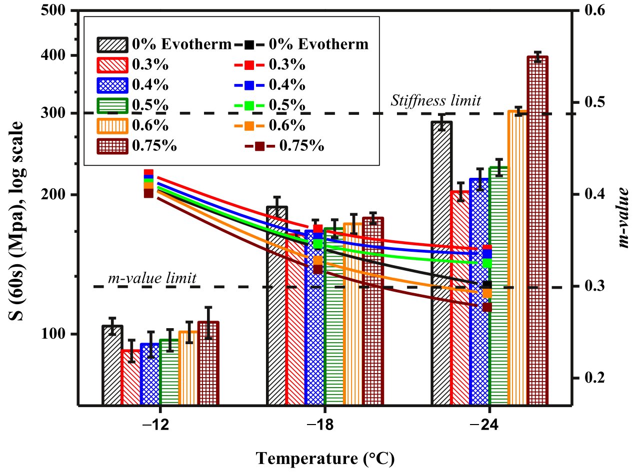


Fig. 10 Binders low-temperature cracking resistance

3.5 Bending beam rheometer

Figure 10 illustrates the findings from the BBR test. *m*-value denotes the ability of bitumen to dissipate applied stress via plastic flow, whereas creep stiffness (*S*) denotes the bitumen's stiffness at low temperatures. Preferably, the bitumen should be less stiff (lower creep stiffness) and capable of quickly releasing stress for better low-temperature performance (higher m-value) [68]. Also, the tested binder must satisfy the AASHTO M320 specification limits of $S \le 300$ MPa and *m-value* ≥ 0.3. The figure shows that the CMB (0% Evotherm) has a creep stiffness of 104 MPa at − 12 °C, the value increased to 188 MPa and 287 MPa − 18 °C and − 24 °C testing temperatures, respectively. The addition of 0.3 percent Evotherm at − 12 °C results in a 12 percent reduction in CMB stiffness, and the deflection of the beam increases by 40%, which is logical due to the Evotherm's oily nature, which tends to physically diminish the binder's hardness. At 0.4 and 0.5 percent Evotherm concentration, the creep stiffness decreased in a similar way. When the temperature was raised to − 18 °C, the Evotherm-modified bitumen showed a steady rise in creep stiffness with a lower *m*-value when compared to the 0% Evotherm bitumen. A similar stable trend was observed at − 24 °C (excluding the 0.6% and 0.75% Evotherm), which indicates that the incorporation

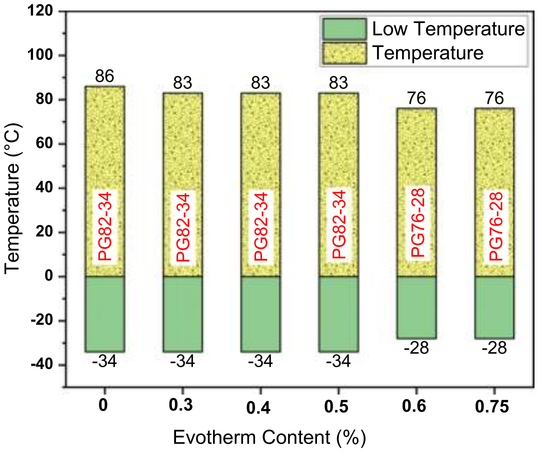


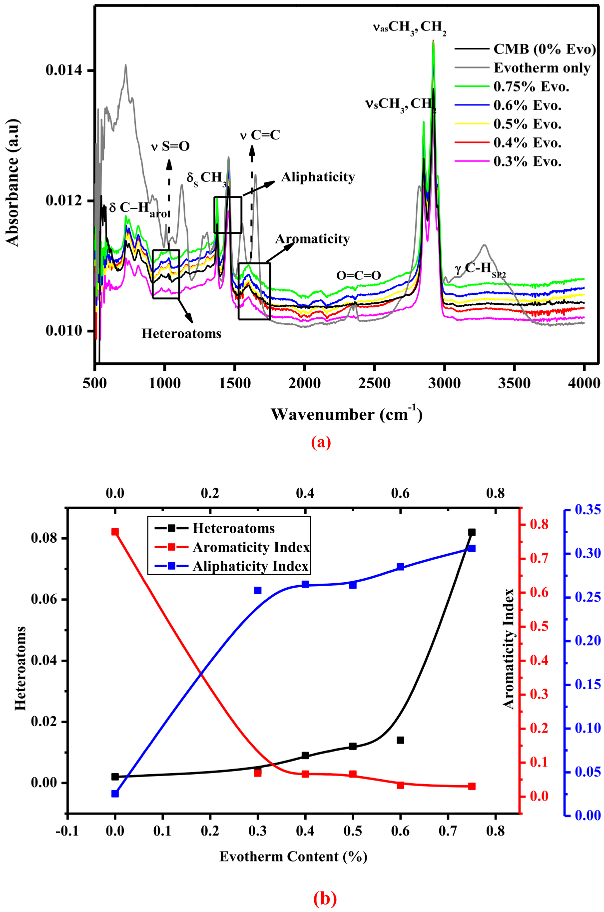
Fig. 11 High- and low-temperature limits of cup lump rubber-modified bitumen added with Evotherm

of Evotherm to the blend retards the rate of creep stiffness increase at low pavement temperatures. Reduced creep stiffness implies that the binder's elasticity has improved, and is expected to resist the formation of microcracks at low temperatures [69]. Moreover, the high *m*-value result indicates that Evotherm significantly improved the stress dissipation property of the CMB. At − 24 °C, the 0.3, 0.4, and 0.5 percent Evotherm-modified binders met the *S* and *m*-value criteria and were classified as PG82-34. However, more testing at a much lower temperature is required until failure occurs. While the bitumen with 0.6% and 0.75% Evotherm did not meet the S(60 s) and m-value requirements at − 24 °C testing temperature, they were classified as PG7618. Essentially, modifying the CMB with a little Evotherm lowered the creep stiffness and improved the CMB's stress release capabilities. Figure 11 summarized the performance grades of the tested bitumen at high and low temperatures.

3.6 Fourier transform infrared (FTIR)

Figure 12a depicts the changes in the Evotherm-modified bitumen's infrared spectrum. In addition, the FTIR of Evotherm alone was presented to show a specific footprint. Visual inspection of the IR shows that the Evotherm-modified binders display similar absorbance to the CMB (0% Evotherm). Meaning that adding Evotherm marginally affect the primary functional set of compounds present in CMB. Furthermore, the lack of a peak at 1700 cm^{-1} (carbonyl absorption) indicates that no oxidation occurred throughout the modification process.

However, significant changes were detected at wavenumbers ranging from 600 to 2000 cm^{-1}. Cis 1.4-polyisoprene absorption corresponding to =C−H out of plane bending was detected at 842 cm^{-1}, while a strong out of plane absorption band belonging to the toluene group was detected at 619–775 cm^{-1}. Figure 12b also displays the quantitative examination of the spectra, it indicates a decrease in absorption at 1600, 1030, and 720 cm^{-1} wavenumbers, which describe the C=C, C−O, and C−H stretch. The decrease in absorption is attributed to the decrease in density of C−H, C−O, and C=C stretch in response to the addition of the Evotherm [65, 70]. The decrease in aromaticity index at 1600, 900, 810, and 720 wavenumbers decreased with increasing Evotherm content, which confirms the decrease in density reduction. This shows that the Evotherm-modified bitumen has a less compact structure as well as less mobility and elasticity compared to the CMB. The structure becomes less rigid as the Evotherm dosage increases, as evidenced by lower viscosity values of the Evotherm-modified binders and an increase in the aliphaticity index $(A_{1456} + A_{1378}/A)$. In conclusion, the FTIR bands of the "Evotherm-modified binders" are comparable to those of CMB without Evotherm.

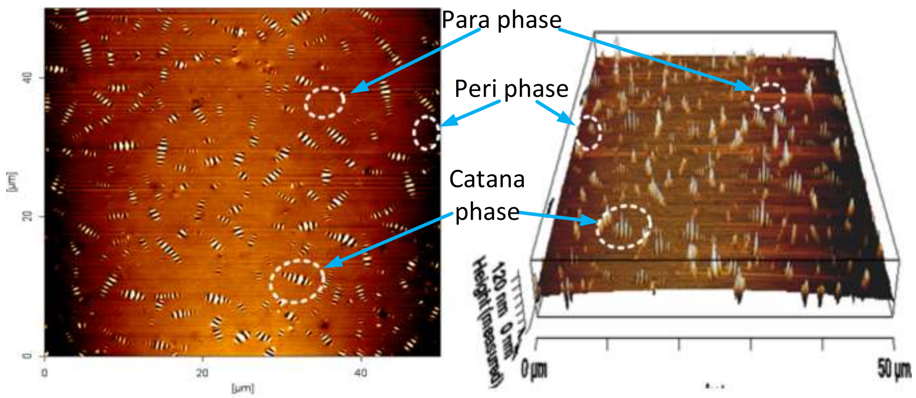


(a)

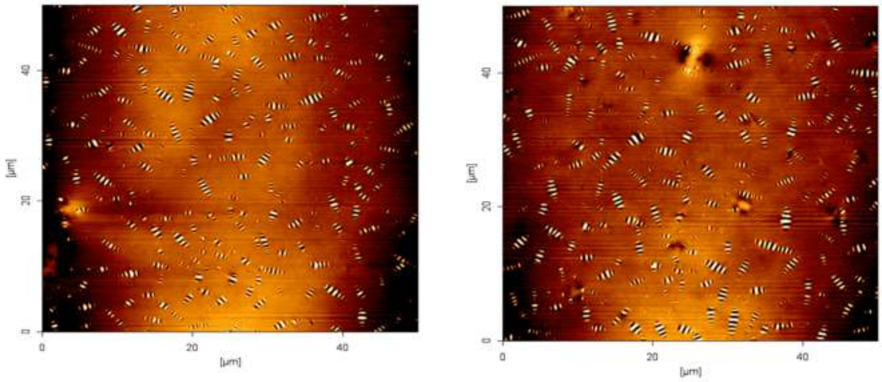
(b)

Fig. 12 **a** Evotherm-modified bitumen Fourier transform infrared spectra. **b** Changes in heteroatoms, aromaticity, and aliphaticity index in response to Evotherm modification

Fig. 13 Atomic force microscope scan of Evotherm-modified bitumen

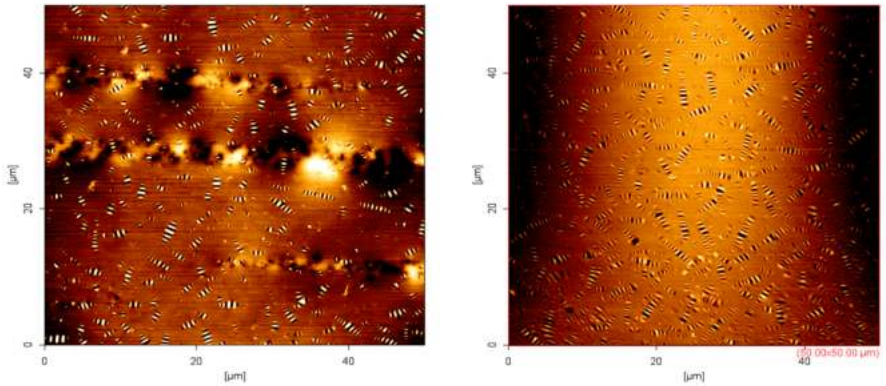


CMB (0% Evotherm)



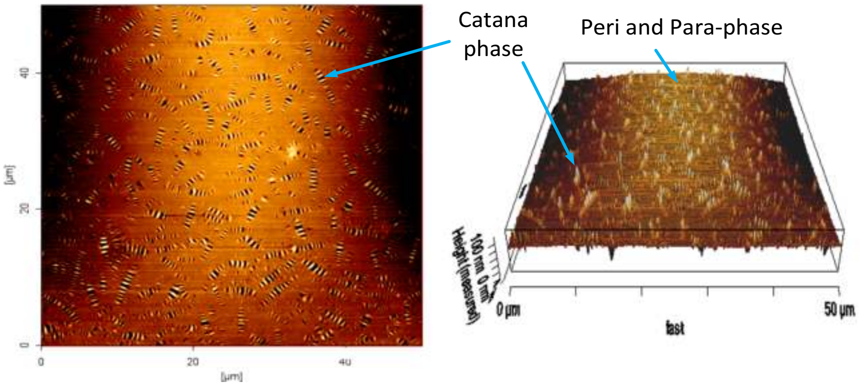
0.3% Evotherm

0.4% Evotherm



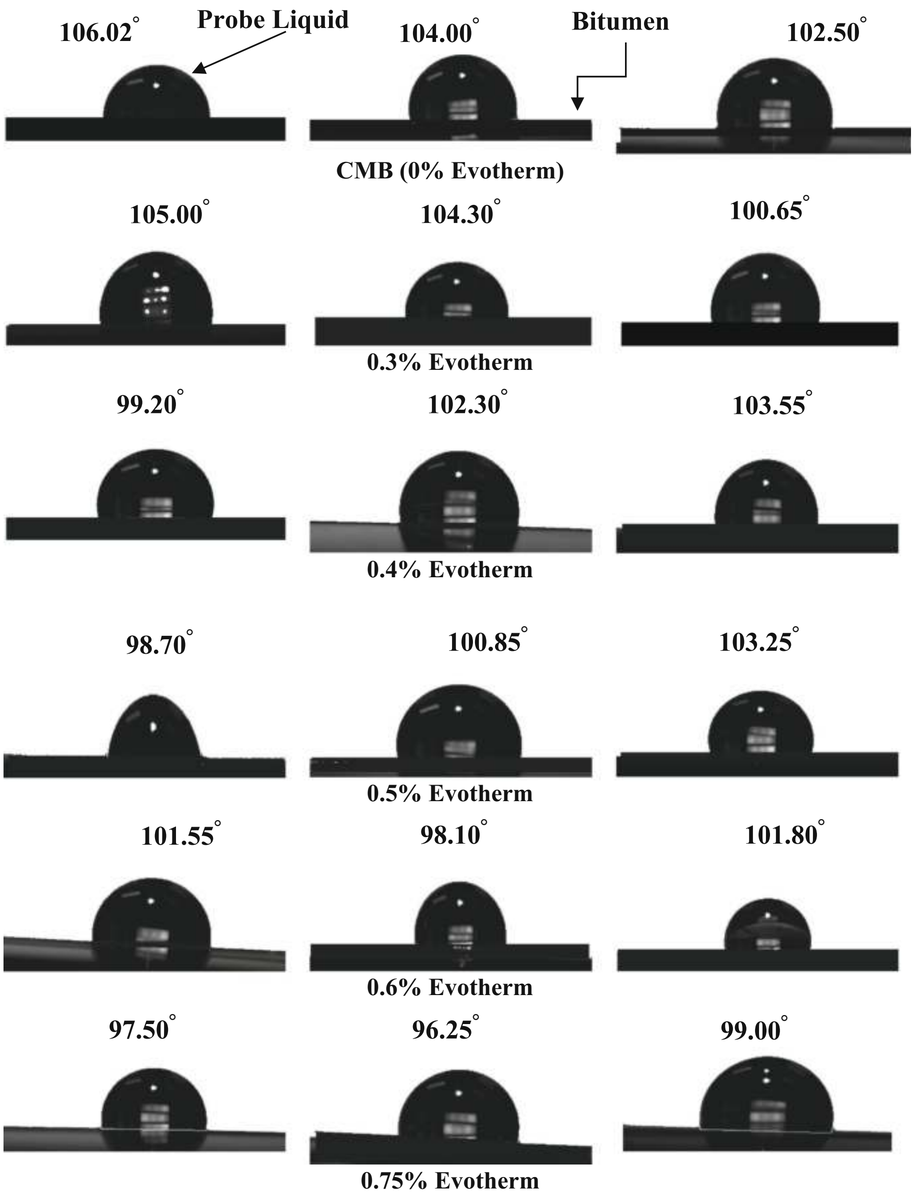
0.5% Evotherm

0.6% Evotherm



0.75% Evotherm

Fig. 14 Effect of Evotherm modification on contact angle. The average contact angle value decreases with increasing Evotherm content



3.7 Atomic force microscopy

Figure 13 depicts a scanning picture of the modified bitumen at 50 × 50 nm. In AFM test results, a three-phase network structure is commonly observed. They are the Catana phase (bee-structure), the peri-, and the para-phases [71]. The para-phase has a low stiffness and high cohesiveness, whereas the peri- and catana phases have a high stiffness (low cohesion) [72, 73]. A scanning image of modified bitumen shows that the Catana phase is more noticeable, whereas the peri- and para-phases are nearly indistinguishable. This is because the bitumen source and origin of the base binder determine its morphology at the microscale level. At 0.3–0.4% Evotherm modification, no significant changes in the catana, peri- and para-phases were observed. But at 0.6 and 0.75% Evotherm content, an increase in the number with a decrease in the size of the catana phase was detected, indicating a decrease in binder stiffness. Changes in the shape and size of the catana phase indicate changes in the molecular structure of the bitumen [74]. The lack of clear separation of the peri- and para-phases makes it difficult to examine the changes in bitumen cohesion and adhesion properties; this could open a new line of research into finding another technique for measuring these properties with reliability.

3.8 Contact angle

Figure 14 presents the results of the contact angle measurement carried out to examine the affinity of the modified bitumen to water. Bitumen is either hydrophilic (attracts water) or hydrophobic (rejects water and has less susceptibility to moisture-induced failure) [52, 75]. On the CMB (0% Evotherm), the distilled water forms an average angle of 104° and can be termed hydrophobic (contact angle greater than 90°) and tends to repel the dropped water on the bitumen surface. The addition of 0.3–0.75% Evotherm reduced the average contact angle of the CMB from 104° to 103°, 102°, 101°, 100°, and 98°. This implies that Evotherm modification decreased the hydrophobicity of the CMB as Evotherm content increased. Meaning there is an increase in the wettability of the dropped water on the surface of the Evotherm-modified bitumen, which may weaken the bond at the bitumen-aggregate interface. The spreading of water on the bitumen surface leads to fewer points of contact between the aggregate and the bitumen. The results of this test indicate that adding warm mix additives to bitumen reduces its water-repelling performance. Previous laboratory research has linked WMA's moisture sensitivity to insufficient aggregate drying; however, the current findings imply that bitumen's water-repelling power may also play a role in WMA's moisture susceptibility. The biggest issue with WMA's durability is the possibility of moisture-induced bond breakdown, which is caused by the lower mixing temperatures resulting in the formation of a thin water film between the aggregate and the bitumen surface [76]. Thus, the existence of water in the aggregates or outside the bitumen coated aggregate is a leading cause of asphalt moisture damage failure at this critical interface [52].

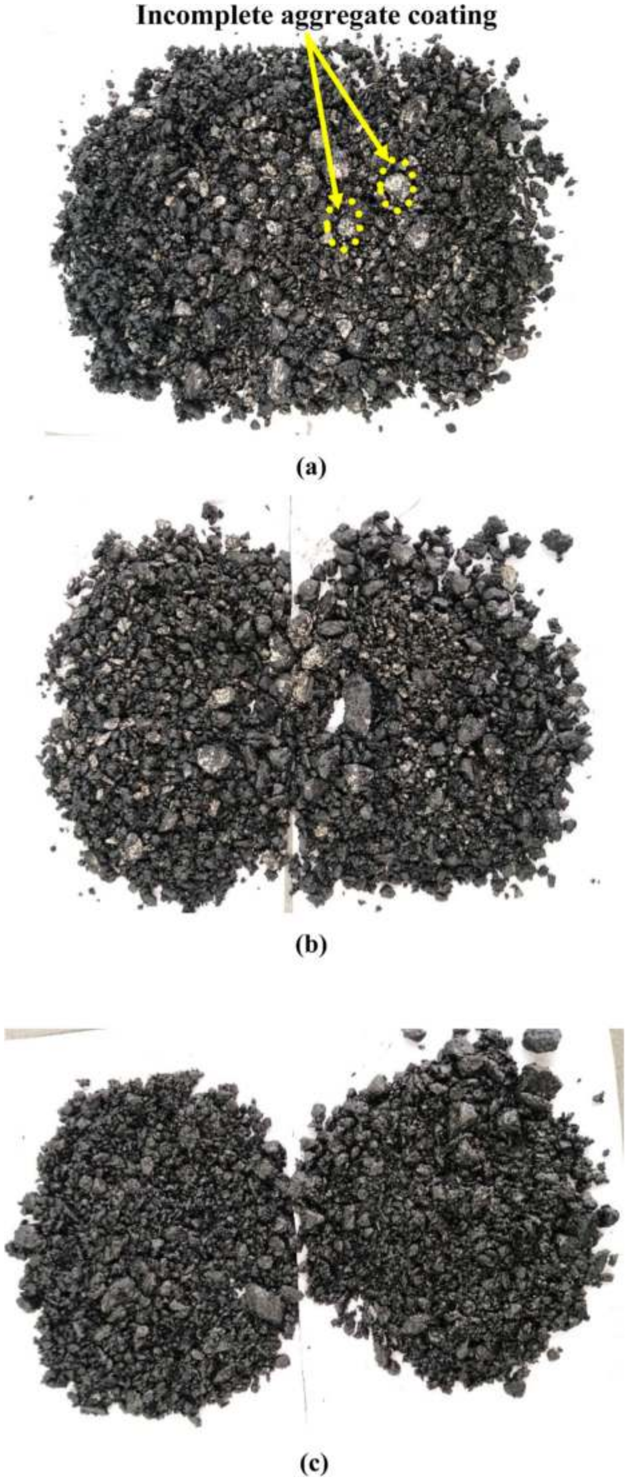


Fig. 15 Selection of optimum Evotherm content for reducing the mixing temperature of cup lump-modified asphalt with Evotherm. **a** After mixing at 130 °C with 0.5 percent Evotherm, white dots signify an incomplete aggregate coating. **b** When mixed with 0.5 percent Evotherm at 140 °C, uncoated aggregate is visible. **c** Complete aggregate coating when mixed at 140°C with 0.75% Evotherm

3.9 Selection of optimum Evotherm content and production temperature for asphalt mix production

This section presents the process of selecting the optimum percentage of Evotherm suitable for asphalt mix production using aggregate coating and compactability testing (as recommended by the manufacturer of Evotherm). The chosen approach complies with the AASHTO R35 criteria and is practiced by [14, 77]. It is a trial-and-error method where asphalt mixtures are prepared by varying the Evotherm content (0.3–0.75%) and at various mixing and compaction temperatures. Observations are then made to identify the mixture that has a 100% aggregate coating and sufficient air voids between 3–5%. The aggregate coating test is summarized in Fig. 15a–c. The first trial mix was prepared with the CMB + 0.5% Evotherm content at mixing and compaction temperatures of 130 and 120 °C, respectively. The loose mixture shows less than 100% aggregate coating with more than 5% air void, indicating the need to increase the mixing temperature or the Evotherm content (Fig. 15a). Another mixture was made by raising the mixing and compaction temperature to 140 and 130 °C, respectively, while keeping the Evotherm dosage at 0.5 percent; this resulted in a minor increase in aggregate coating but not the desired 100% (Fig. 15b). Another sample was made by raising the Evotherm value to 0.75 percent while the mixing temperature was maintained; this resulted in a 100 percent aggregate coating and 3–5% air void (Fig. 15c). As a result, 0.75% Evotherm content was chosen as the best dose for making the CMA mixture in WMA.

4 Conclusion

This study looked into the effect of Evotherm modification on the rheology and morphology of cup lump rubber-modified bitumen. It was discovered that adding Evotherm to CMB slightly reduced its viscosity. A lower percentage of Evotherm (0.3–0.5%) has no effect on CMB rutting resistance, whereas a higher percentage (above 0.5%) reduces rutting resistance by one grade at high temperature. The CMB's cracking resistance at low temperatures increased after the addition of 0.3–0.5 percent Evotherm to the blend. Furthermore, the FTIR results show that incorporating Evotherm lowered the absorption of double and single bonds, but the primary functional groups remained unaffected. The bee-structure is clearly dispersed in the AFM scan, although the para-phase and peri-phase are not clearly separated. Contact angle measurement revealed that the inclusion of Evotherm marginally lowered the CMB's water-repelling performance. According to the aggregate coating test results, 0.75% Evotherm content is the optimum dosage to be used in lowering the manufacturing temperature of cup lump-modified asphalt. Finally, the rheology and morphology of Evotherm-modified bitumen are comparable to cup lump-modified bitumen, so the binder can be used at the mix design level for environmentally friendly and sustainable road construction. It is worthy of mention that all the tests conducted in this study were at the binder level. As a result, future research will look into the mechanical interfacial bonding performance of the Evotherm-modified binders as well as cost–benefit analyses of the binders at the mix design level.

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