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Article

Experimental Evaluation of a Partially Synthetic Bitumen in Road Paving Applications

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Abstract: Bitumen is produced from non-renewable natural resources, continuously deplete and intrigues researchers to look for alternative binders. Annually, tons of waste engine oil (WEO) and crumb rubber (CR) are discarded unsustainably and pose a significant environmental threat. Adding these industrial waste products to asphalt provides a safe and cost-effective way for their disposal and improves the bitumen's performance in parallel. This study uses various combinations of waste engine oil and crumb rubber with 60/70 penetration grade bitumen to produce a partially synthetic bitumen. Adhesion being one of the critical characteristics of the bituminous binder has been assessed using the bitumen bond strength test along with physical and rheological properties. Results showed that waste engine oil with crumb rubber inclusion increases penetration and decreases the softening point, viscosity, complex modulus, and bonding strength. Additionally, polyphosphoric acid (PPA) in smaller dosages was also incorporated into the optimum percentage of CR + WEO to improve the properties of the binder. The results confirm that binder modification with waste engine oil and crumb rubber can be more effective with PPA. It is concluded that 35% of waste can replace the virgin binder giving a cost-effective and environmentally friendly solution.

Keywords: bitumen; synthetic bitumen; adhesion; waste engine oil; crumb rubber; PPA; high-temperature performance

1. Introduction

Bitumen is a binding agent customarily used in flexible pavements that are usually built with hot mix asphalt (HMA). The increased road transport volume and construction-phase failures with environmental and external factors reduce asphalt pavements' useful life and increase the probability of permanent faults like rutting and moisture damage (Fareed *et al.* 2020, Haroon *et al.* 2022). More than 90% of global pavements are flexible because they are strong, last a long time, and can be repaired easily. Bitumen, a highly viscous material, is used as the asphalt's binder. It exemplifies the transition between the solid and liquid phases at different temperatures. Bitumen consumption has reached an all-time high of 700 million barrels per year. It is an organic compound composed of hydrocarbons and typically contains 1% carbon, 80% oxygen, and trace amounts of oxygen, sulfur, nitrogen, and other metals (Abdul Hassan *et al.* 2019).

A renewable resource, bitumen, is created through the distillation process. The depletion of natural resources has far-reaching consequences for both price increases and pollution. Bitumen has been altered with a wide variety of additives to combat the issue of waste management and boost the efficiency of traditional binders. Binders are expensive and harmful to the environment. However, using modified or substitute binders can reduce costs and help the environment (Ahmad *et al.* 2022). Various techniques were used to enhance the performance by modifying the binder, and the final product is designated synthetic bitumen. The partially synthetic bitumen reported by using various additives, i.e., polymers (Zhu *et al.* 2014), i.e., waste engine oil (Liu *et al.* 2018), modification of aggregate gradation (Aodah *et al.* 2012), Styrene Butadiene Styrene (SBS) (Yildirim 2007, Zhang and

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Hu 2013), crumb rubber (Xiao *et al.* 2009, Cetin 2013), PPA (Domingos and Faxina 2015), polyethylene (Mohanty 2013) and costly Nanomaterials (Fang *et al.* 2013, Fini *et al.* 2015).

Waste engine oil is a by-product of the automotive industry collected yearly from various automobiles. Zinc, lead, potassium, and magnesium are the most common contaminants, such as firewood. Due to the increase in population globally, every year, approximately 11 billion tons of waste are produced, indicating that each person produces more than one ton on average, and this number is increasing. According to estimates, waste generation will double by 2025 compared to 2000. Modern societies are based on using cars, which cannot operate without lubricants. The quantity of waste oil has increased with the population and vehicles. As the population increases, waste increases which causes landfill space and health problems (Counts 2023). Massive volumes of garbage, such as blast furnace slag, glass, steel slag, scrap tires, plastics, waste engine oil, building and demolition wastes, etc., are being dumped in landfills and stockpiles around the world, posing environmental and economic dangers (Abukhettala 2016).

Bitumen can be revitalized by adding used motor oil, making for an eco-friendlier product (Int *et al.* 2014). Although some of WEO is recycled, most ends up in the trash. Despite its changing production resources, the chemical composition of WEO matches that of bitumen. Bitumen treated with waste engine oil (WEO) was shown to have improved penetration and a lower softening point than unmodified bitumen (Abdul Hassan *et al.* 2019). Feng et al. (Feng *et al.* 2020) reported that a high concentration of WEO had unfavorable effects on the binder's properties. The injection of waste engine oil alters the elastic properties of bitumen, i.e., a decrease in complex modulus and a rise in phase angle. Liu et al. (Liu *et al.* 2018) reported that fatigue performance was improved by WEO, although bitumen loses some resistance to rutting. Liu et al. (Liu *et al.* 2019) concluded that the high rutting strength and performance are degraded, as reported by an analysis of the bitumen's rheological behavior. It was presented no proof of hydrophobic interactions between bitumen and WEO in their studies. More than that, they advocated a WEO adjustment of 4-8%. Jia et al. (Jia *et al.* 2014) concluded that the integration of WEO in binder reduces its excellent productivity, i.e., rutting resistance, as documented in the literature and through extensive evaluations of waste engine oil-modified bitumen. As the binder content was decreased, the mixture's fatigue resistance improved, and the concentration of organic aldehyde compounds in bitumen was raised through modification with used motor oil. The increased carbonyl groups in bitumen made it more vulnerable to oxidation. Furthermore, oil incorporation lowered stiffness at cold pressures, whereas high temperatures hampered the binder's elastic recovery and have been demonstrated.

Shoukat and Yoo (Shoukat and Yoo 2018) concluded that engine oil was found to have an improved influence on thermal cracking in dynamic evaluations of changed binders at the expense of decreased resistance to rutting. Filtered used engine oil decreased binder flexibility by 35% compared to virgin asphalt after only a few months of aging. Still, 2.5% oil lowered the Performance Grade (PG) of the top end by 0.3 °C. Overall, the performance of unprocessed waste motor oil was below that of both new and filtered crude oil. DeDene (DeDene 2011) reported that waste engine oil might make bitumen combined with Reclaimed Bituminous (RAB) less rigid and improve low-temperature characteristics. It is possible to reduce the hardness of asphalt roads by utilizing engine oil without reducing the roads' ability to withstand wet conditions.

Like Waste engine oil, the amount of automobile waste tires has also substantially increased worldwide in the past few years due to the evaluation of the automobile industries. Proper disposal or recycling of crumb rubber (CR) produced from automobile tires becomes very important for the environment. This massive waste material harms the earth's natural environment and pollutes the water, air, and soil. Eventually raises concern for global warming, economic crisis, energy preservation, and others. Using waste conserves road construction materials may minimize landfill space, reducing environmental impact. Therefore, scientists focus on innovating the disposal technique of these waste tires and waste engine oil as bitumen modifiers (Formela 2021). The examination of the automobile sector has helped to similar growth in the amount of waste tire produced around the world as has occurred with waste engine oil. Crumb rubber (CR), a by-product of tire recycling, must be disposed of or recycled correctly to prevent environmental damage (Lo

Presti 2013). Burning one ton of used tires produces 450 kg of harmful gases & 270 kg of soot is kept out of the atmosphere. The use of CR as a modifier in asphalt is an excellent example of its application (Rumyantseva *et al.* 2020). The optimal preparation process parameters for rubber-modified asphalt include shear temperatures of 180 °C, shear times of 45 minutes, and shear speeds of 5000 rpm (Liu *et al.* 2015).

The dosage of crumb rubber also imparts the properties of the modified asphalt. S. Mashaan et al. (S.Mashaan *et al.* 2011) concluded that the increased crumb rubber content significantly improves elasticity and ductility. Shafabakhsh et al. (Shafabakhsh *et al.* 2014) reported that asphalt mixtures containing 10% waste rubber powder improved their performance at higher temperatures, reduced asphalt mixtures' sensitivity to temperature, and increased their resistance to rutting. Further, it also reduced the binder's production costs; rubberized asphalt mixtures had superior performance at high temperatures compared to the control specimens. Similarly, (Gohar *et al.* 2022) reported that adding 15% crumb rubber increases the stiffness, viscosity, and high softening point and improves the rutting resistance of conventional bitumen. The crumb rubber size also played a vital role in altering the asphalt properties.

Ibrahim et al. (Ibrahim *et al.* 2013) concluded that lowering the crumb rubber size improves asphaltic mixtures' rutting resistance, resilience, and fatigue cracking resistance. Brasileiro (Brasileiro *et al.* 2019) reported that crumb rubber has lower complex modulus values at low temperatures, minimizing the likelihood of cracking and enhancing pavement performance and endurance. Wang et al. (Wang, Liu, Apostolidis, *et al.* 2020) concluded that the swelling of rubber dramatically modifies its characteristics, making it softer and more viscous.

Huang (Huang 2008) concluded that the rutting resistance improved with increased viscosity and flexibility at high temperatures. In addition, the low temperature increased fatigue resistance because of a decrease in viscosity. The studies (Attia and Abdelrahman 2009, Wang *et al.* 2012, Moreno *et al.* 2013) concluded that CR improves pavement performance and mechanical responsiveness of changed binders in asphalt binders. The studies (Yu *et al.* 2014, 2017, Guo *et al.* 2017, Sienkiewicz *et al.* 2017) confirmed that utilization of CR is known to significantly enhance pavement qualities such as resistance against fatigue & rutting, improvement overall durability, and reduce maintenance costs. The studies (Navarro *et al.* 2004, Kim and Lee 2013) established that the encapsulation efficiency of rubber-modified bitumen is not a significant issue. It becomes a problem at higher temperatures. That becomes more pronounced as a considerable percentage of CR is integrated into the asphalt. The mechanical behavior of hot-mixed asphalt and the mechanical/chemical qualities of bitumen were improved by using the WEO-CR rejuvenator, which was also found to raise the overall performance of the mixture (HMA) (ELTWATİ *et al.* 2022).

Recently, bitumen has become highly technical, with modifiers like polymers, acids, and mineral fillers controlling its performance properties (Masson 2008). PPA is a reactive reagent comprised of phosphoric acid oligomers. PPA is compatible with asphalt, which considerably improves the performance of binders at high temperatures. The penetration index and viscosity values of bitumen have been enhanced due to increased asphaltenes content and decreased saturate and resins (Varanda *et al.* 2016). PPA might also be regarded as a viable alternative to polymers. Numerous researchers have investigated the chemical reaction between PPA and asphalt (Polacco *et al.* 2005). It was observed that the rheological properties of CRB-modified binder were enhanced by adding up to 2% PPA. PPA also enhanced the storage stability by raising the viscosity of CR-modified asphalt. High asphaltenes content increases resilience to rutting but decreases fatigue resistance (Qian *et al.* 2019). PPA improves the bitumen performance significantly. In addition, it was concluded that a bitumen addition of up to 1% substantially improves high-temperature performance while having a negligible effect on fatigue characteristics (Hao *et al.* 2019).

According to the literature mentioned above, the WEO as a modifier into the bitumen compromised the performance properties of the asphalt binder (Liu *et al.* 2018, 2019, Shoukat and Yoo 2018, Feng *et al.* 2020, Abbas *et al.* 2022). However, Crumb rubber (Huang 2008, Mashaan and Karim 2014, Yu *et al.* 2014, 2017, Guo *et al.* 2017, Sienkiewicz *et al.* 2017) and Polyphosphoric acid (Polacco *et al.* 2005, Varanda *et al.* 2016, Hao *et al.* 2019, Qian *et al.* 2019) improves the performance of asphalt binders and mixtures. In this regard, the performance of WEO-modified binders can be improved by using these two additives, i.e., CR and PPA. Many previous studies are available where CR and PPA have been used separately for improvement of WEO-modified bitumen, however, very limited work is available on the combined behavior of CR and PPA on WEO-modified bitumen. So, an indepth analysis was required to evaluate the combined effect of these two additices in WEO-modified bitumen. They were used to scrutinize their impact on the performance of the virgin binder. The main aim behind this study is to use maximum industrial waste (WEO) in the original binder and enhance the performance of WEO-modified biunder.

2. Materials and Methodology

2.1. Bitumen

The bitumen used in this study was 60/70 pen-grade with a softening point of 50 °C was procured from a local refinery in Pakistan.

2.2. Waste Engine Oil

Waste engine oil was collected from local motor vehicle repair plants and markets. The waste engine oil used for this experiment was obtained from a local auto repair shop, as shown in Figure 1. Previous research has revealed that waste engine oil contains elements like zinc, lead, calcium, and calcium.

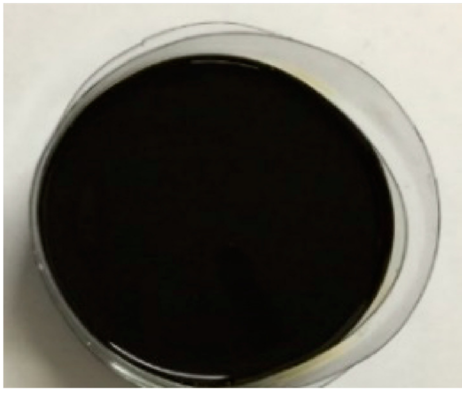

Figure 1. Waste Engine Oil.

2.3. Filter paper

Due to engine wear and tear during vehicle operation, the engine oil became contaminated with metal traces and was continuously heated and oxidized (Rosado and Pichtel 2003). In aged motor oil, metallic components such as potassium, calcium, zinc, and iron were detected (Zając *et al.* 2015). After modification, these metal residues may have affected the rheological properties of the bitumen. Therefore, waste engine oil was filtered with a Whatman™ grade filter to maintain the rheological characteristics, as illustrated in Figure 2.

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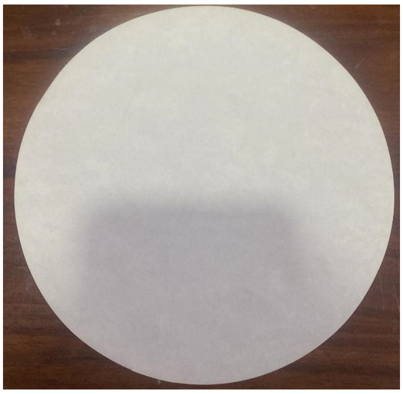

Figure 2. Filter Paper.

2.4. Crumb Rubber

Crumb Rubber (CR) was extracted from used tire scrape and grind them in shredder plants, as illustrated in Figure 3. After being crushed, the material passed through sieve No. 50/300 microns/0.2997mm (Kök *et al.* 2013, Kim and Lee 2015).

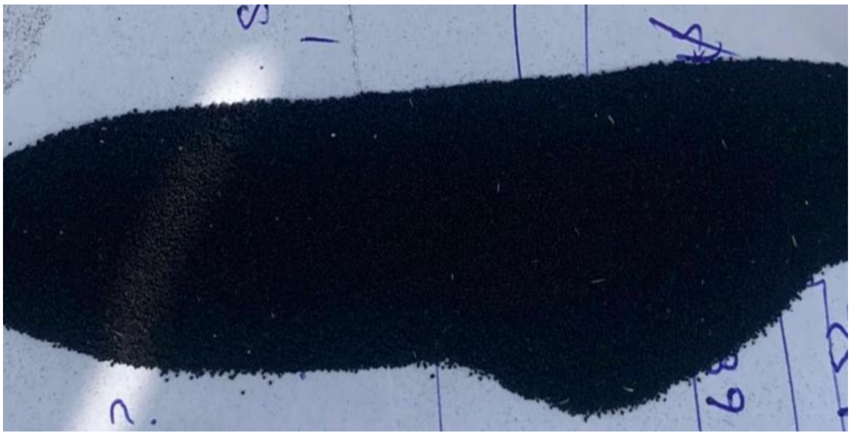

Figure 3. Sample of Crumb Rubber.

2.5. Polyphosphoric Acid

Polyphosphoric Acid (PPA) is a chemical most likely known as a liquid mineral polymer, as illustrated in Figure 4. It's been used since 1990 as a bitumen modifier. It is used to increase the stiffness and grades of binders. The right amount of PPA and proper utilization can bring significant results. It can drastically improve the performance of bitumen at high temperatures. PPA is an oligomer of H_3PO_4. PPA can be used as a straight modifier or in conjunction with other additives. Although PPA has many proven advantages and excellent outputs, some agencies have banned its use due to premature failures of pavements (Yadollahi and Sabbagh Mollahosseini 2011, Hao *et al.* 2019). The PPA used in this study is acquired from a local chemical supplier. PPA is a reactive reagent made up of phosphoric acid oligomers.

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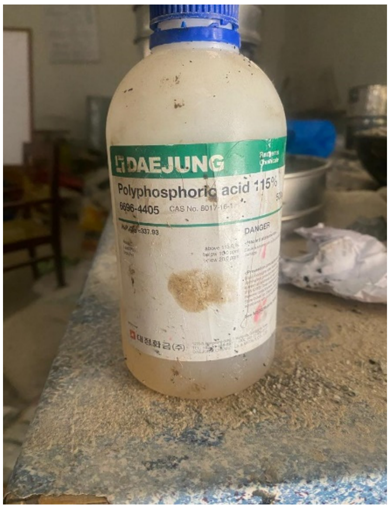

Figure 4. Poly-phosphoric Acid (PPA).

2.6. Preparation of Modified-Bitumen

The bitumen is heated on a hot plate before adding waste engine oil and CR into the bitumen, where the temperature is kept constant and mixed using a shear mixer. Waste engine oil was filtered out by using filter paper of grade Whatman 1002-150. Then, various proportions of used engine oil and crumb rubber were mixed with bitumen. The mixture was then heated and stirred using a high-speed mechanical stirrer at 3000 rpm for approximately 45 minutes at a temperature between 175 °C and 185 °C (Kamal *et al.* 2009, Jamal and Giustozzi 2020, Gohar *et al.* 2022). The mixing proportions are shown in Table 1.

Table 1. Mixing proportions.

Sr. No	Dosage
1	Control blend (B)
2	B+5% WEO+20% CB
3	B+5% WEO+25% CB
4	B+5% WEO+30% CB
5	B+10% WEO+20% CB
6	B+10% WEO+25% CB
7	B+10% WEO+30% CB
8	B+15% WEO+20% CB
9	B+15% WEO+25% CB
10	B+15% WEO+30% CB
11	B+5% WEO+30% CB+0.6%PPA
12	B+5% WEO+30% CB+1.2%PPA
13	B+5% WEO+30% CB+1.8%PPA

3. Experimental Program

3.1. Physical tests

The penetration test was performed on a bituminous sample at 25 °C to determine its consistency and the degree of stiffness. This test was done according to (ASTM D 5 1997) under standard conditions on both modified and new binders. The penetration value of a bitumen sample is the total distance, in units of 1/10 mm, that a specific-sized needle penetrates the sample. The typical needle weighs 100 g and has a loading time of 5 seconds and a pull speed of 50mm/60 seconds. The sample was conditioned for 1.5 hours at 25 °C in a water bath.

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The (ASTM D36/ D36M-14e1 2014) was executed using a ring and ball apparatus. In theory, the softening temperature is the point at which a binder (either the original or a modified version) can no longer hold a steel ball of the weight of 3.5 grams. The softening point is the temperature when the steel balls break the binder and extend about 1 inch.

A binder's mixing and compaction viscosity can be determined using Rotational Viscometer (RV) testing per the AASHTO TP48 standard to calculate the bitumen spinning viscometer values. Using a Brookfield rotating viscometer, bitumen's viscosity is tested. During the experiment, the temperature was maintained at 135 °C. The sample was sheared at a constant strain rate and temperature. Compared to other technologies, the 135°C (275°F) temperature used by rotational viscometers is more accurate and representative of asphalt laying field temperatures.

3.2. Fourier transform infrared spectroscopy

The FTIR test was performed according to ASTM E1252-98 to evaluate the chemical composition of virgin and (WEO+CR, WEO+CR+PPA) modified asphalt binder samples. One drop of a carbon disulfide (CS_2) solution containing samples of virgin and (WEO+CR, WEO+CR+PPA) modified asphalt binder was placed on a KBr table. The scan range ranged from 400 to 4000 cm^{-1}, and the analysis was performed at a resolution of four (4) cm^{-1}. The absorption of infrared light as a function of wavelength is measured by Fourier transform infrared spectroscopy (FTIR) to determine the chemical composition of molecules.

3.3. Rheological Testing of Asphalt binder

The mechanical properties of bitumen were measured using a KINEXUS DSR in compliance with AASHTO T315-19. The shape of the DSR plates was optimized for each temperature and type of test. Plates measuring 25 millimeters thick with a 1-millimeter air gap were selected for use at temperatures above 46 °C, while plates measuring 8 millimeters thick with a 2-millimeter air gap opted for use at temperatures below 40 °C. Performance Level PG was calculated using 25 mm plates revolving at 10 Hz (Haroon *et al.* 2022). DSR measured the failure temperature at G*/sin 1 kPa. Frequency sweep tests were conducted at significant stress and frequencies ranging from 0.1 to 100 rad/s on virgin and modified binders at 22 °C, 34 °C, 58 °C, 64 °C, 70 °C, and 82 °C. The complex modulus |G*| was calculated using the time-temperature superposition approach at various frequencies and temperatures (Fareed *et al.* 2020, Wang, Liu, Zhang, *et al.* 2020).

3.4. Bitumen Bond Strength Test

Bitumen Bond Strength Test (BBS) was performed to assess the bonding strength of bitumen binder (virgin/modified). For determining the effect of moisture, both dry and wet Pull-out tensile testing strength (POTS) was determined. Metal stubs were isolated from aggregate foundation slabs to quantify pull strength accurately and evaluated the Pneumatic Adhesion Tensile Testing Instrument (PATTI). The adapted binders were produced for testing in conformity with ASTM D4541-17 under standard test conditions. Stubs pressed 0.4 grams of cement pastes against base slabs, and the wet and dry pull-off force was recorded.

4. Results & Discussion

4.1. Physical Testing Results

To a large extent, the inclusion of waste materials such as waste engine oil, crumb rubber, and PPA alters the original/control/virgin binder's penetration, softening point and viscosity. The effect of WEO and CRB on the binder is depicted in Figure 5 and Figure 6. The incorporation of WEO increased the penetration values of modified bitumen. It is due to the WEO that reduced the binder's consistency and softening point (El-Shorbagy *et al.* 2019). Used motor oil contains aromatic solvents that break down asphaltenes (Wang, Jia, *et al.* 2020).

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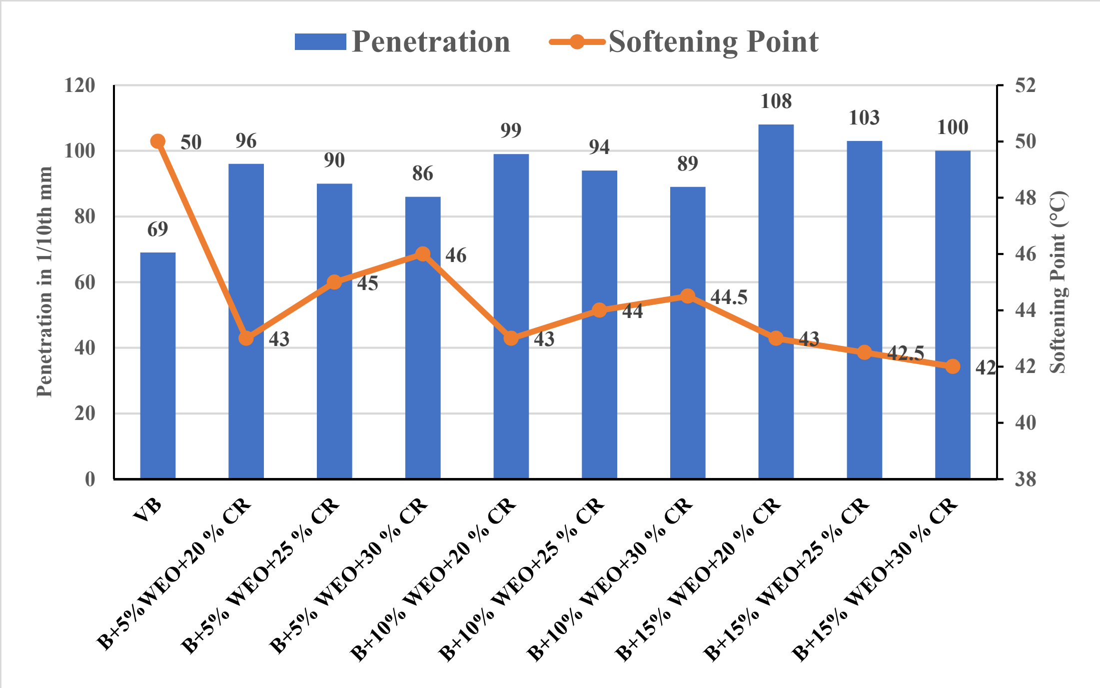

Figure 5. Penetration and Softening Point of Virgin and WEO+CRB Modified Binder Results.

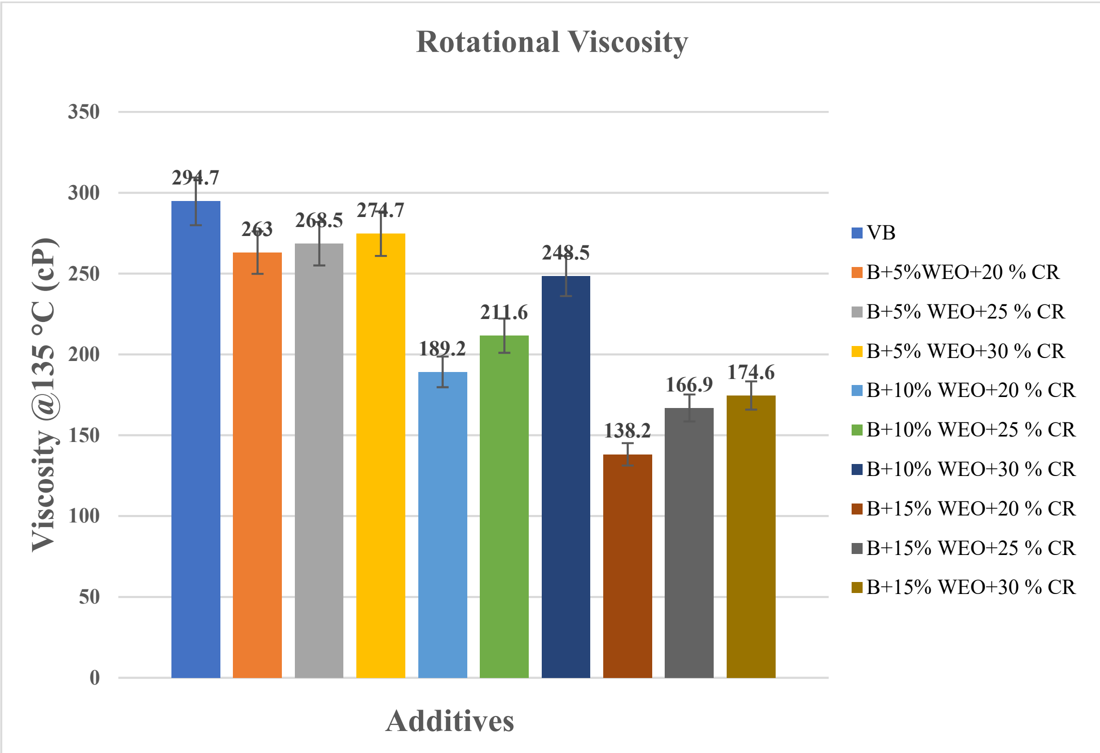

Figure 6. Virgin and WEO + CRB Modified Binder Viscosity Results.

Adding 5% WEO with 20%, 25%, and 30% CR by total binder content increases the penetration value by 28%, 23%, and 20%, respectively, while the softening point decreases by 14%, 10%, and 8%. In 10% WEO with 20%, 25%, and 30% CR, a 30%, 26%, and 22% increase in penetration value and an 8%, 14%, and 12% decrease in softening point value were observed. Similarly, the addition of 20%, 25%, and 30% CR with 15% WEO resulted in a 36%, 33%, and 31% increase in penetration value and a 16%, 15%, and 14% decrease in softening point, as presented in Figure 5 respectively. It indicates that increasing the dosage of WEO results in higher penetration values and a lower softening point, whereas increasing the dosage of CR results in lower penetration values and a higher softening point.

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The addition of WEO softens the binder, whereas the addition of crumb rubber hardens and stiffens it, thereby enhancing its conventional properties.

The control binder's viscosity is reduced when waste engine oil is added and increases when Crumb rubber is mixed, as illustrated in Figure 6.

Figure 7 and Figure 8 show that the combination of 5% WEO and 30% CR seems closest to virgin binder in terms of its properties. To improve it further, this combination has been further modified by adding 0.6%, 1.2%, and 1.8% PPA.

PPA being a cositly additive, the effect of PPA has only been studied on the best combination (5% WEO + 30% CR) for all the test techniques used in this study. The percentage of PPA was calculated based on studies conducted and recommendations from earlier research (Varanda *et al.* 2016).

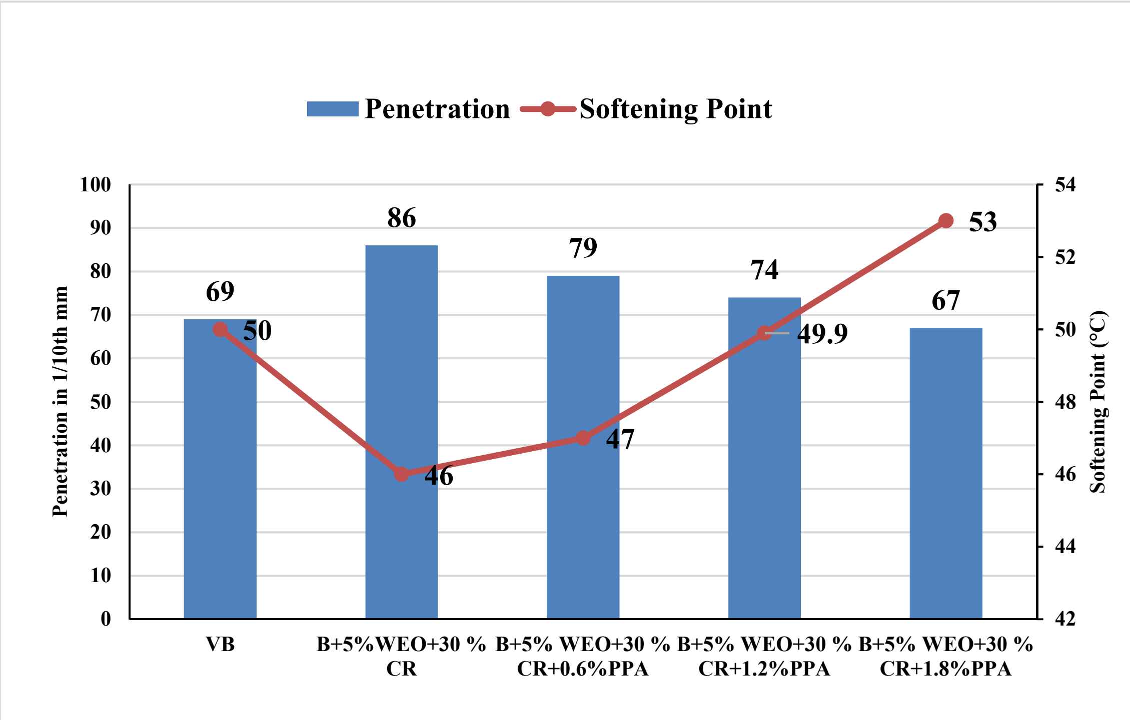

Figure 7. Penetration and Softening Point of Virgin and PPA Modified Binder Results.

The viscosity of partially synthetic bitumen was improved by adding PPA in the predetermined percentages determined by standard testing. There was an increase in viscosity values with lower PPA concentrations in a blend of 5% WEO + 30% CR. Viscosity values from WEO-modified bitumen after exposure to PPA are displayed in Figure 8. The results shows a clear improvement in viscosity and combinations with 1.2 and 1.8 %PPA looks very close to virgin bitumen.

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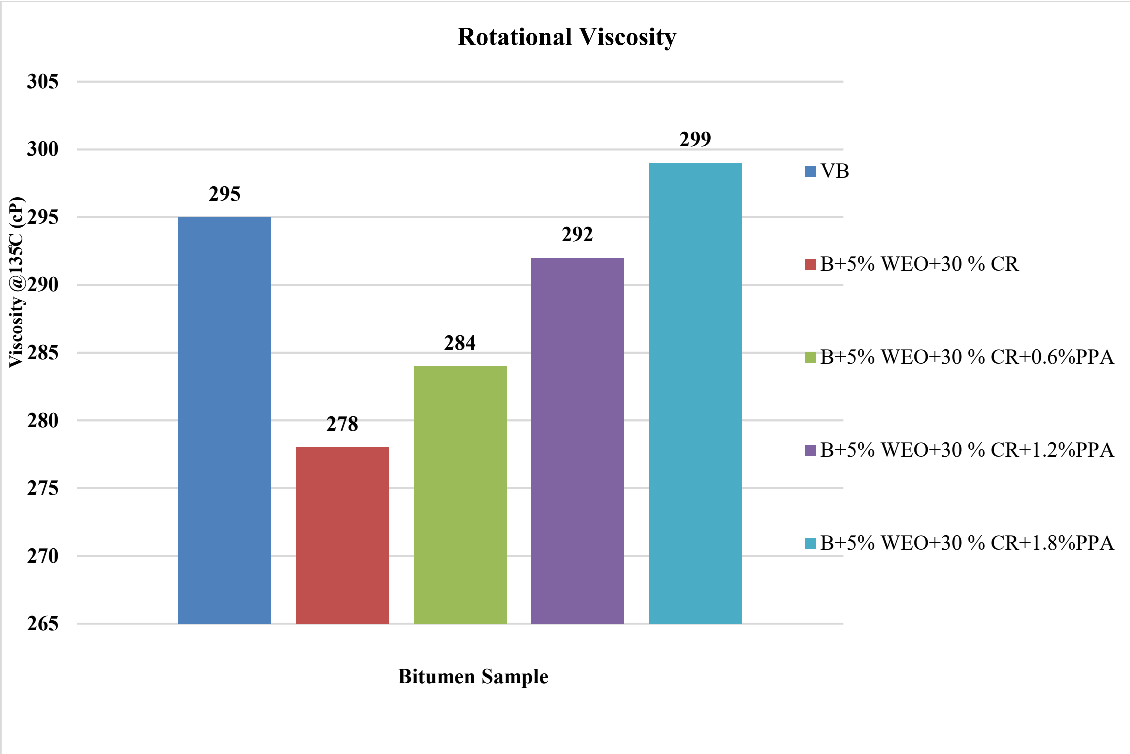

Figure 8. Virgin and PPA Modified Binder Viscosity Results.

The addition of PPA improved the binder's consistency, softening point, and penetration. The reactivity of PPA was boosted because of the polarity of the asphaltene component, which facilitated its dissociation into PPA and H+. PPA changes the asphaltene percentage distribution by disrupting the hydrogen bond network established within agglomerates of asphaltene micelles. It lowers asphaltene content and raises maltene solubility in the residue (Varanda *et al.* 2016).

4.2. FTIR Analysis

The analysis's stated goal is to learn more about how WEO + CR and WEO + CR + PPA change the chemical makeup of asphalt binder. Figure 9 shows that the FTIR investigation yielded spectra for all WEO + CR (5%, 10%, and 15%), and WEO + CR + PPA, respectively. The effect of PPA on FTIR peaks was also like the virgin binder shown in Figure 9. Furthermore, when WEO + CR and WEO + CR + PPA were combined in asphalt binder, no aging occurred, as shown by a quantitative examination of asphalt binder aging, which was investigated by determining the carbonyl index.

Figure 9 illustrates that two peaks are generated, with frequencies of 2860 cm^{-1} and 2920 cm^{-1}, which are associated with the growing oscillation in C-H and its aliphatic chains. The absorption bands of C=C have a peak energy of 1600 cm^{-1}, and this characteristic is found in aromatic compounds. Additionally, the C-H asymmetry within CH_2 and CH_3 is responsible for the peak at 1450 cm^{-1} and 1370 cm^{-1}. The 1050 cm^{-1} peak is associated with S=O sulfoxide, and the C-H benzene ring's vibrations account for the last peak between 690 and 920 cm^{-1}. This qualitative study is based on a review of the relevant literature (Yao *et al.* 2013, Jamal *et al.* 2020). The spectra and the observed peaks are consistent across all samples. In addition, the appearance and disappearance of peaks are not observed between functional groups, indicating any physical relationship between the samples (WEO+CR and WEO+CR+PPA) and the asphalt binder.

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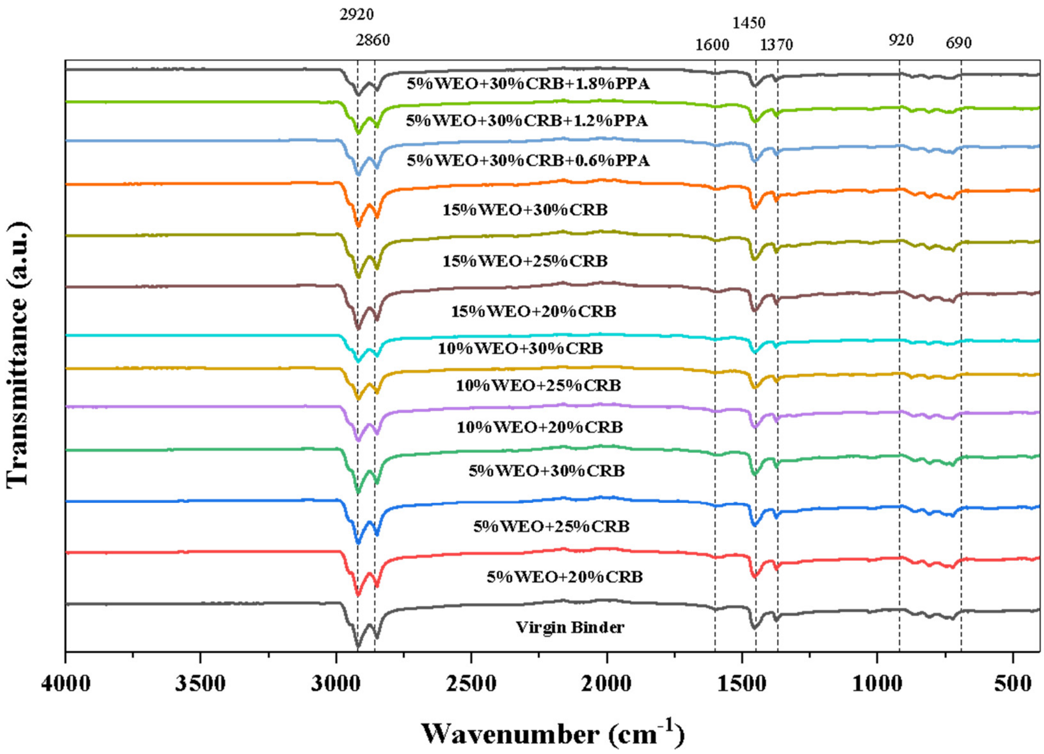

Figure 9. FTIR Analysis of all the samples (WEO, CR, and PPA).

4.3. Performance Grading Test Results

The failure temperatures are evaluated using the Performance Grade (PG) experiments according to the Superpave criteria. It provides an idea of how the modification mechanism has changed PG. The estimated value of temperature failure is 67.3 °C for the base binder, which comes in PG 64, which fulfills the requirement. Figure 10 depicts the binder's failure before and after modification, respectively. The virgin control binder received a pen grade of 60/70 and was determined to be PG-64. A specific binder can be used in an area with a maximum average pavement temperature of 64 degrees Celsius over seven days. The addition of the WEO and CR softened the bitumen and deteriorated it to PG-58, PG-52, and PG-46.

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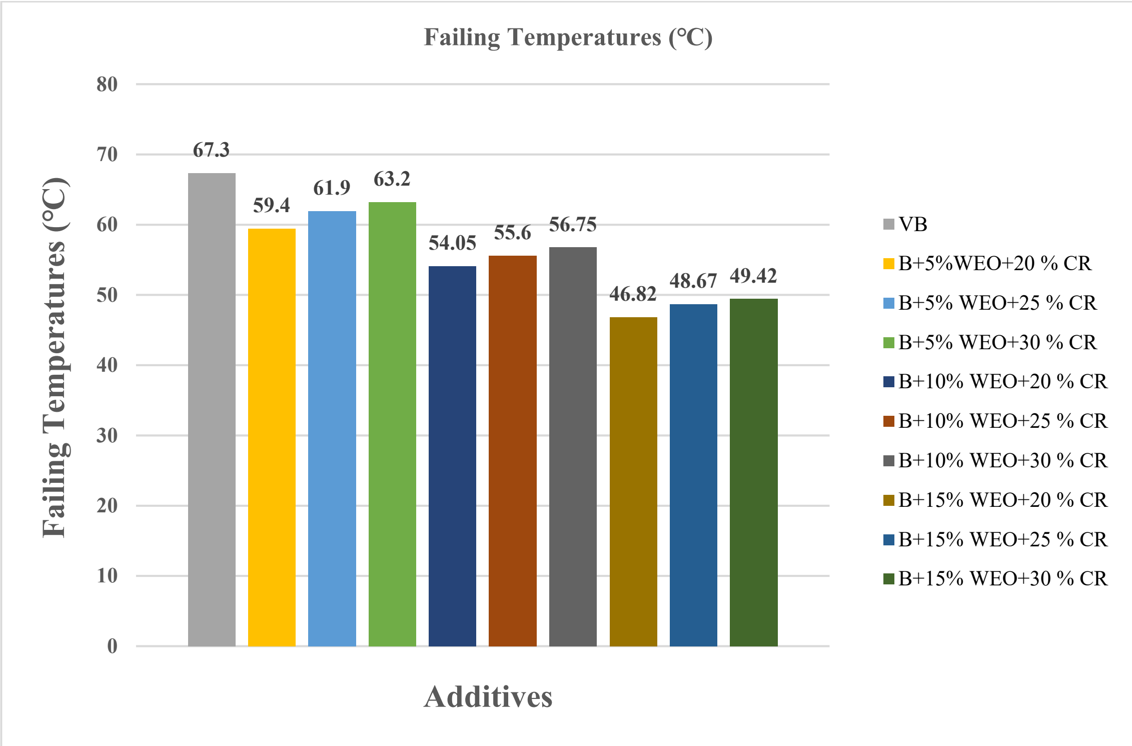


Figure 10. The failing temperature of Virgin and WEO+CRB Modified Binder.

Adding 0-10% recycled lubricating oil to bitumen raised the critical cracking temperature to around two °C at the expense of the high-temperature PG grade (Villanueva *et al.* 2008). Tire rubber powder added to bitumen has been found to enhance the qualities of the modified binder. A higher percentage of tire rubber powder was shown to boost resistance to rutting. The addition of tire rubber powder to bitumen is also being considered to increase the pavements' long-term economic and environmental viability (Hainin *et al.* 2015).

As previously explained, adding PPA causes the binder to become stiffer due to chemical reactions and network development, as illustrated in Figure 11. It resulted in a gradual improvement as high as PG70, which is relatively stiff and advantageous for regions with extremely high temperatures. In compliance with the existing environmental conditions in Pakistan, it is recommended that PG 70 be used in most regions (Mirza *et al.* 2011).

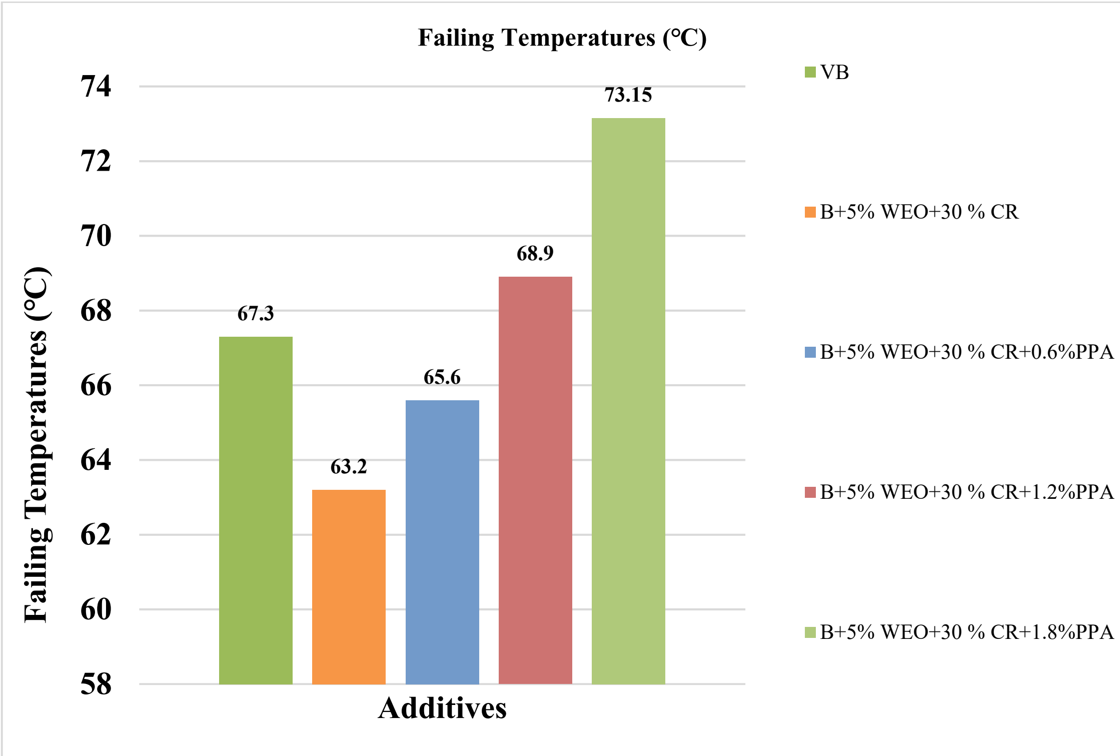
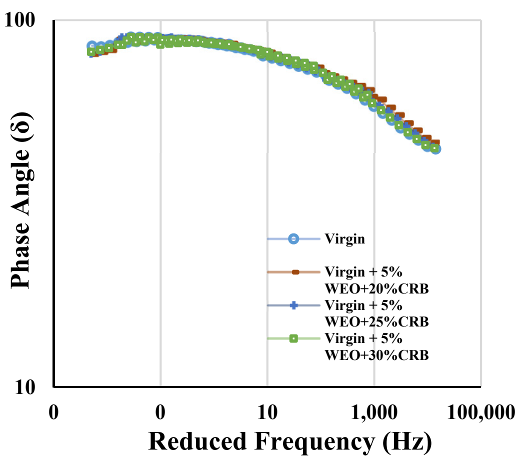


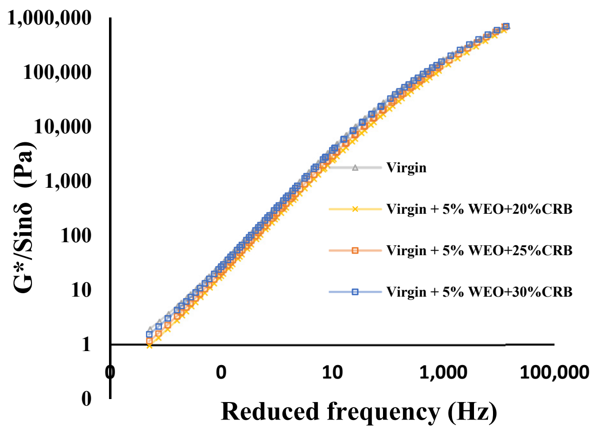
Figure 11. The failing temperature of Virgin and PPA Modified Binder.

4.4. Frequency Sweep Test Results

The results of the frequency sweep can be used for the evaluation of temperature & time dependence of laboratory-created specimens of asphalt binder mixed with WEO + CRB, WEO + CRB + PPA, and base binder. At various values of temperature and a frequency range from 0.1 to 100 Hz, the frequency sweep is executed. The master curves are developed with the help of a sigmoidal function for all customized binder samples. Figures 12 (a) and (b) show the Master curves drawn between the rutting parameter, and the phase angle, respectively, against reduced frequency after the binder modification with WEO and CRB.



(a)



(b)

Figure 12. Master Curve for Virgin and 5% WEO + CRB Modified Binder: (a) Phase Angle, (b) Rutting Resistance.

As illustrated in Figure 12 (a), phase angles are used to compare the viscoelasticity of the original binder to that of the modified one (Arabani and Tahami 2017). Additionally, the phase angle values made the material exceptionally elastic and softer. Waste engine oil reduces the asphaltenes percentage in bitumen, which is critical for the binder's stiffness for high-temperature performance. Combining 5% WEO with 20%, 25%, and 30% of Crumb rubber significantly degraded high-temperature performance, as illustrated in Figure 12 (b). Crumb Rubber's addition to bitumen helps restore the binder's natural viscoelastic equilibrium, making it more resistant to permanent deformation. The use of CRMB additionally lengthens the life of the roadway. Due to its greater adherence to a wide range of aggregates, CRMB mitigates the effects of deformations such as rutting and cracking. It provides exceptional resistance to any permanent deformation, improved aggregate-to-binder adhesion, enhanced performance in extreme climates, lengthened fatigue life of mixes, and is highly flexible and stable (Khan and Kamal 2008).

From Figure 13 (b), with the inclusion of 10% WEO with 20%, 25%, and 30% Crumb rubber in the dosage, the values of the G*/Sinδ decrease with the more content of WEO and low content of Crumb rubber at high temperatures relative to the base binder. Figure 13 (a) illustrates that including 10% WEO with 20%, 25%, and 30% Crumb rubber in the dosage increased the phase angle values making the material more elastic and softer, which shows increasing behavior in the elasticity of the asphalt binder (Abdalfattah *et al.* 2016). Figure 13 (b) illustrates that the increased content of WEO results in lowering bitumen binder stiffness and resistance against rutting within the range of high temperatures. But with the combined inclusion of WEO + CR, G*/Sinδ values decrease and are more prominent in the low-temperature range, which lowers the stiffness in a binder and rutting parameter values.

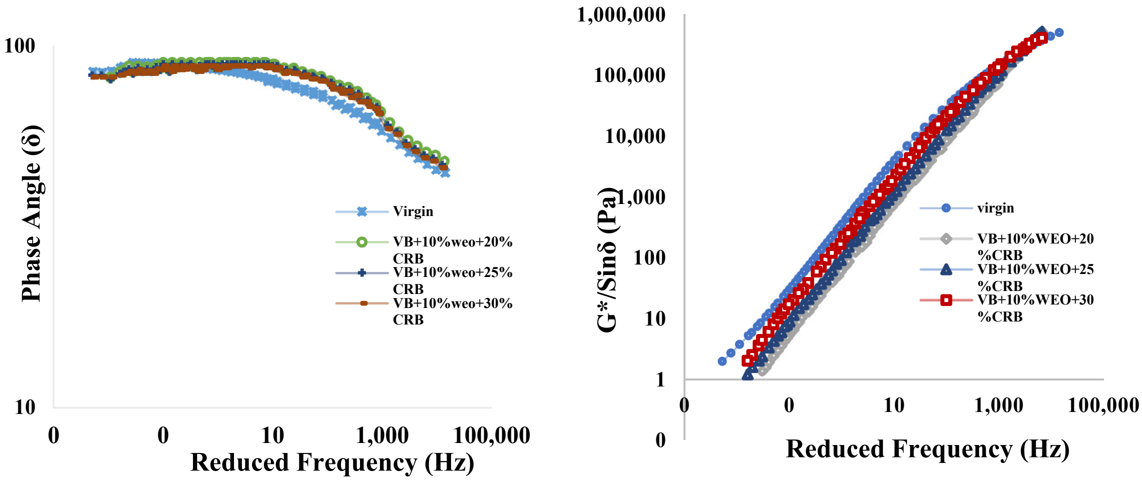
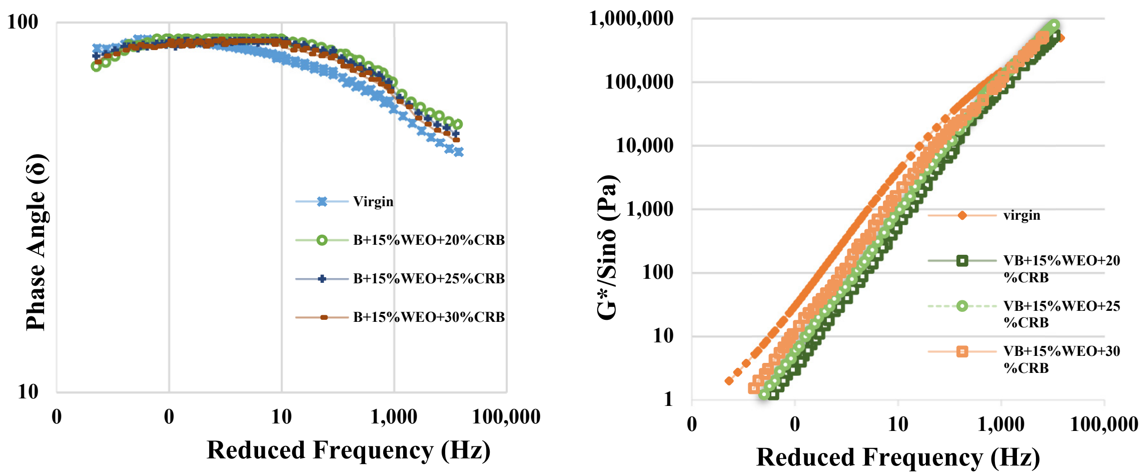


Figure 13. Master Curve of for Virgin and 10% WEO + CRB Modified Binder: (a) Phase Angle, and (b) Rutting Resistance.

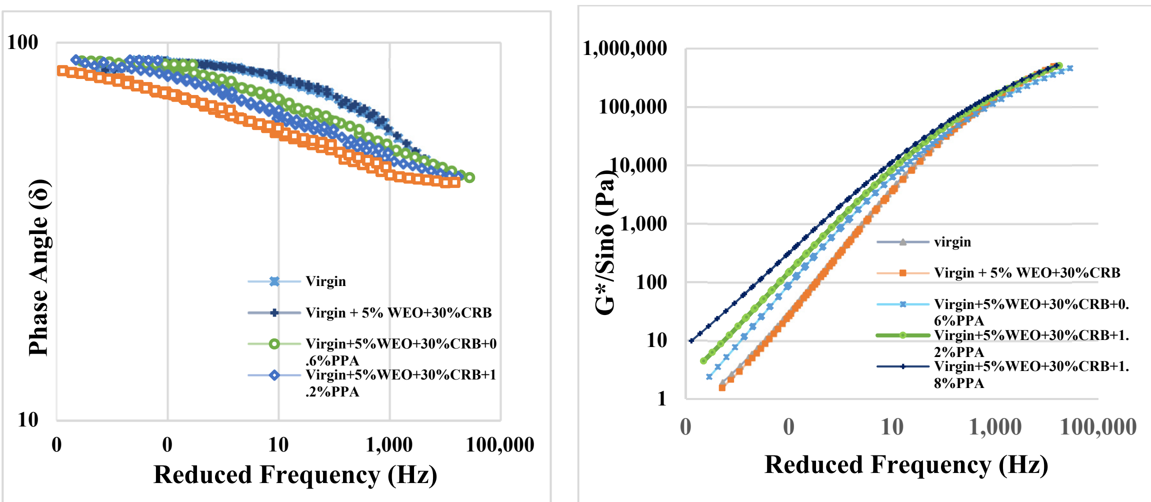
Figure 14 (a) illustrates that adding 15% WEO with 20%, 25%, and 30% Crumb rubber in the dosage increased the phase angle values making the material more elastic and softer, which shows increasing behavior in the elasticity of the asphalt binder. Figure 14(b) illustrates that G*/Sinδ is the factor for the rut plotted in opposition to a reduced frequency. The asphalt binder's durability against rut and fatigue can be calculated using the rut factors. There is a substantial decrease in rutting resistance compared to the virgin binder, which can be seen from the master curves of the rut factor. The values of the G*/Sinδ decrease with more content of WEO and low content of Crumb rubber at high temperatures relative to the base binder. It means that the increased content of WEO results in lowering bitumen binder stiffness and resistance against rutting within the range of high temperatures. But with the combined inclusion of WEO + CR, G*/Sinδ values decrease and are more prominent in the low-temperature range, which lowers the stiffness in a binder and rutting parameters.



(a) (b)

Figure 14. Master Curve of for Virgin and 15% WEO + CRB Modified Binder: (a) Phase Angle, and (b) Rutting Resistance.

The other objective of this experiment was to measure the effects of high temperatures on the phase angle, and the rutting parameter of asphalt binders, as illustrated in Figure 15 (a), and (b), that contained waste engine oil and crumb rubber with PPA. Figure 15 shows the addition of PPA improved the high-temperature performance of WEO with CRB-modified bitumen. It improved the high-temperature performance of the virgin binder shown in previous research (Abbas *et al.* 2022). The binder showed more elasticity as the phase angle decreased, as illustrated in Figure 15 (a), These values are desirable for rutting resistance by increasing the values of complex modulus and rutting parameters, as illustrated in Figure 15 (b). PPA greatly enhanced the high-temperature performance of WEO-modified bitumen, as proved in previous research (Sandeep *et al.* 2021).



(a) (b)

Figure 15. Master Curve for the Virgin and WEO + CRB + PPA Modified Binder Master Curve of for Virgin (a) Phase Angle, and (b) Rutting Resistance.

4.5. Bitumen Bond Strength Test Results

Based on the aggregate material and the conditioning of the material with moisture, bitumen exhibits varying bond strengths. The adhesive power of the binder on the aggregate base was

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measured with a BBS test conducted in dry and wet conditions. Figure 16 below shows a graph of the POTS values derived using equation (1):

$$\text{POTS} = \frac{(BP*Ag)-C}{Aps} \quad (1)$$

Here, POTS is the pull-off tensile strength, BP is burst pressure Ag is the contact area with a value of 2620 mm², and C is the piston constant with a value of 0.286.

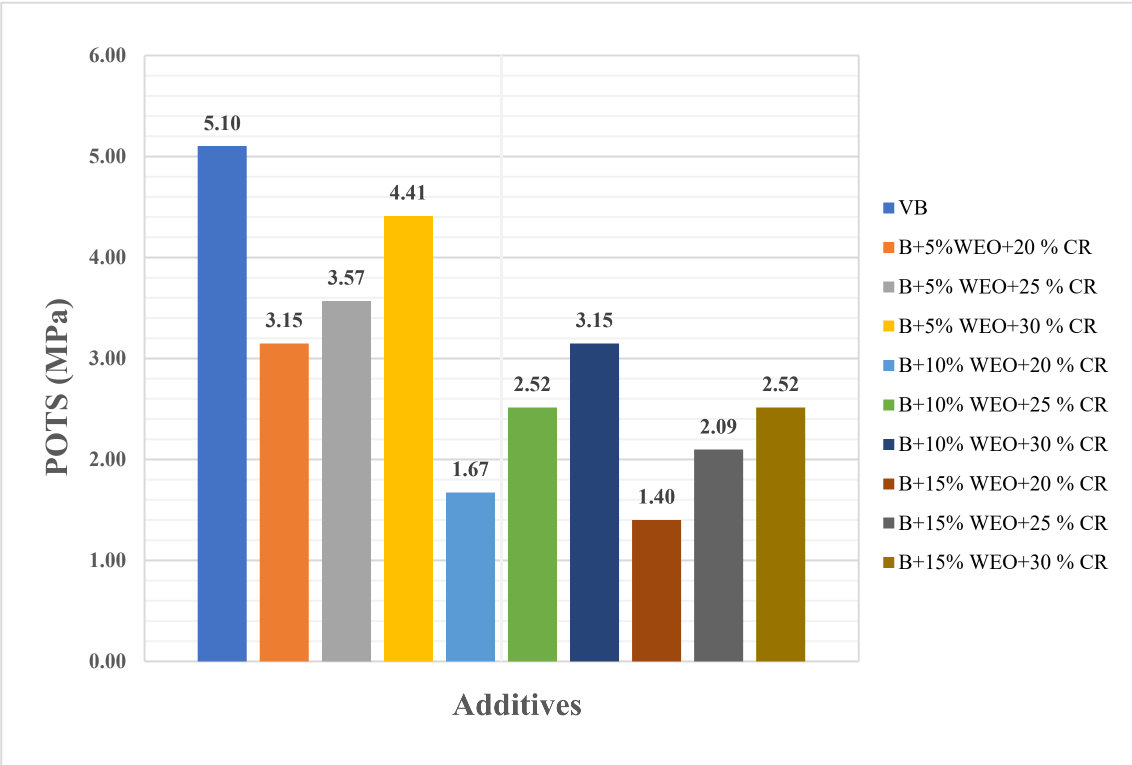

Figure 16. POTS in Dry Condition of Virgin and WEO+CRB Modified Binder.

The tendency is the same for both dry and wet samples. The POTS values of the various wet-state samples were lower than those of the dry-conditioned specimen. It is because bitumen and aggregate bonds are weakened by high moisture content, reducing POTS values (Moraes *et al.* 2011). Also, an increase in the value of POTS authenticates a stronger association between aggregates and binder specimens due to the incorporation of different waste material forms.

The effect of WEO and CR on adhesion was experimentally evaluated using PATTI. A Bitumen Bond Strength (BBS) test was performed for dry and wet conditions to determine the bond strength of the binder to the aggregate surface. Figure 17 shows the effect of PPA on POTS values in dry conditions of WEO-modified bitumen. For dry conditioning, the addition of 5% WEO with the inclusion of 20%, 25%, and 30% CR by total binder content, after 24 hours of dry conditioning, POTS values decrease by 38 %, 27%, and 13%, In 10 % WEO with the inclusion of 20%, 25% and 30% CR, a decrease of 67%, 50% and 38% in value of POTS was observed at dry condition. Similarly, when 15% WEO was mixed with 20%, 25%, and 30% CR, the POTS value of the dry sample decreased by 72%, 58%, and 50%, respectively, when compared to the virgin binder Lower dosages of PPA in a combination of 5%WEO showed increased values of POTS.

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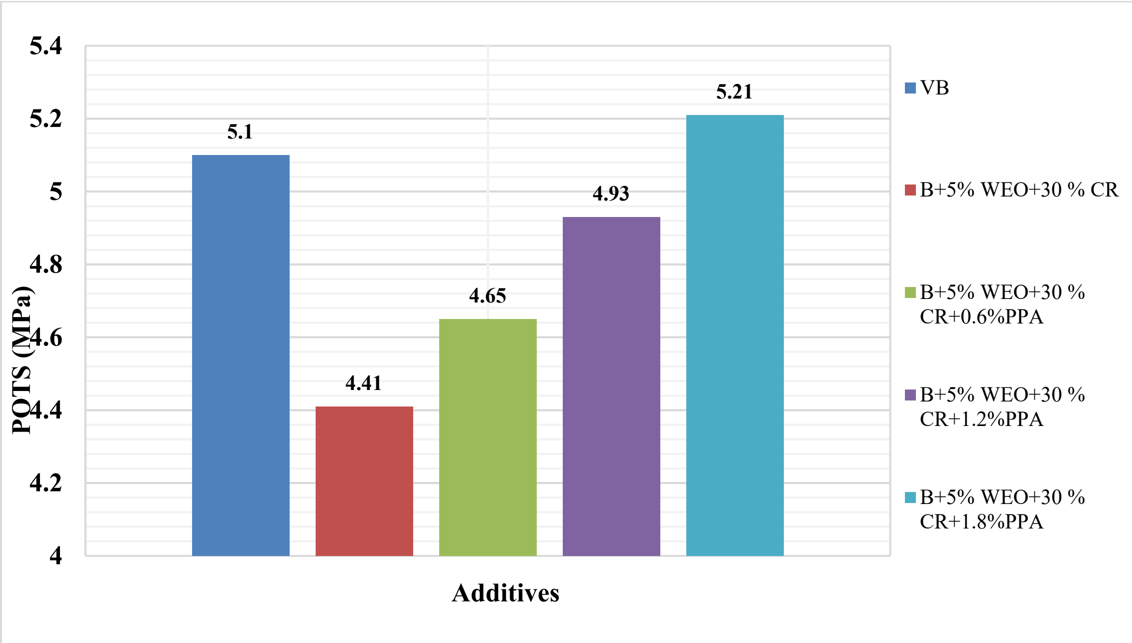

Figure 17. POTS in Dry Condition of Virgin and WEO + CRB + PPA Modified Binder.

Figure 18 illustrates that by adding 5% WEO with 20%, 25%, and 30% CR by the total binder content, POTS values decrease by 26 %, 11%, and 4% at wet conditions. In 10 % WEO with 20%, 25%, and 30% CR, a 36%, 19%, and 7% decrease in POTS values was observed. For that, in 15 % WEO with the inclusion of 20%, 25%, and 30% CR, a decrease of 47%, 36%, and 17% in the POTS value of the wet sample was observed.

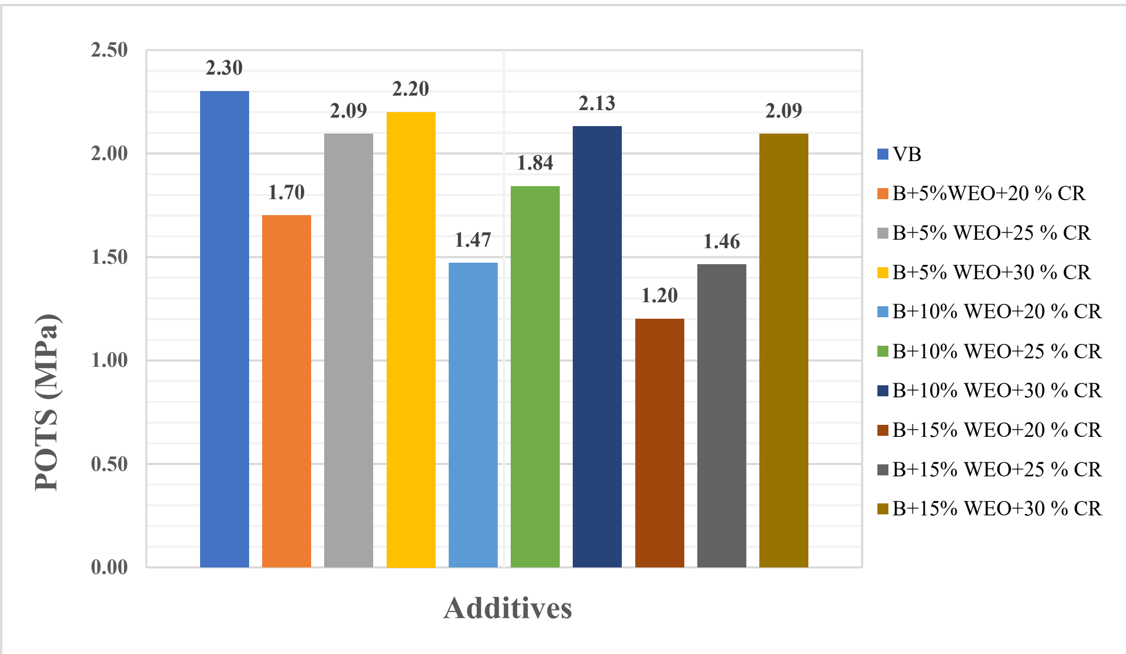

Figure 18. POTS in Wet Condition of Virgin and WEO+CRB Modified Binder.

Figure 19 show the effect of PPA on POTS in wet condition values of WEO-modified bitumen. Compared to dry conditions, the partially synthetic bitumen showed lower values after water conditioning because water permeates the bitumen-bitumen interface and the bitumen-aggregate interface, weakening the bond. It means that due to the incorporation of crumb rubber, an increase in the value of POTS authenticates a stronger association between aggregates and binder specimens. A decrease in POTS indicates a weaker bond between aggregates and binder specimens due to the incorporation of waste engine oil. Increased WEO dosage results in a lower POTS value, whereas increased CR dosage results in a higher POTS value, and vice versa. Lower dosages of PPA in a combination of 5% WEO showed increased POTS values.

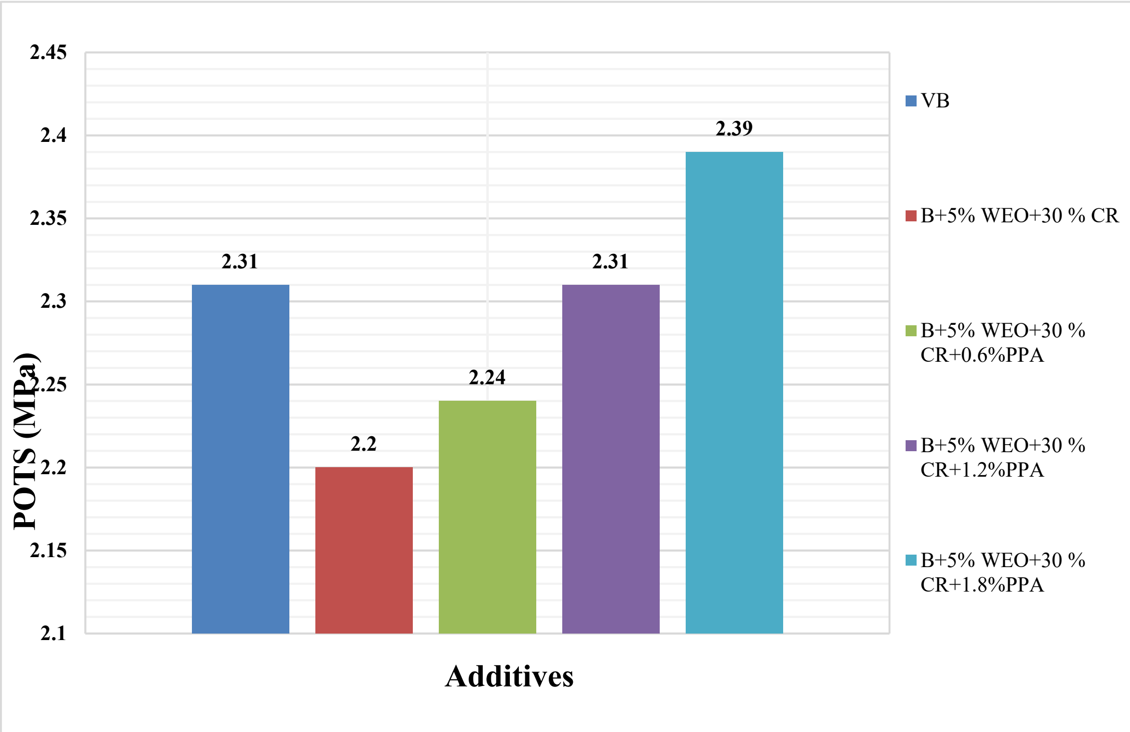

Figure 19. POTS in Wet Condition of Virgin and PPA Modified Binder.

5. Statistical Analysis

The statistical properties of tests for viscosity, moisture susceptibility, and Dynamic Shear Rheometer tests. Tukey's primary method was used for the statistical analysis. It takes one step to compare for various purposes. It is used to determine the considerable difference between means.

5.1. Viscosity Tests

The statistical analysis of viscosity results is shown in Table 1, in which only one dependent variable, i.e., test results and categorical independent variables of 13 categories. The interpretation of the output obtained from statistical analysis is based on the criteria that the mean values of categories lying in the same subset as that of the virgin category have a similar effect, while those in different subsets have a significantly different effect in contrast to the control category (Fareed *et al.* 2020). The statistical analysis deduced from SPSS software is explained as follows:

Table 1. Statistical Analysis of Viscosity.

Statistical Analysis of Viscosity							
Additives	N	Subset at 95% confidence interval					
		1	2	3	4	5	6
B+15%WEO+20%CR	3	138.2					
B+15%WEO+25%CR	3		166.9				
B+15%WEO+30%CR	3			174.6			
B+10%WEO+20%CR	3				189.2		
B+10%WEO+25%CR	3					211.6	
B+10%WEO+30%CR	3						248.5
Sig.		1.000	1.000	1.000	1.000	1.000	1.000

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Additives	N	Subset at 95% confidence interval					
		7	8	9	10	11	12
B+5%WEO+20%CR	3	263					
B+5%WEO+25%CR	3		268.5				
B+5%WEO+30%CR	3			274.7			
B+5% WEO+30 % CR+0.6%PPA	3				284		
B+5% WEO+30 % CR+1.2%PPA	3					292	
Virgin Binder	3					294.7	
B+5% WEO+30 % CR+1.8%PPA	3						299
Sig.		1.000	1.000	1.000	1.000	1.000	1.000

The addition of 5% WEO and 30% CRB with the addition of PPA affects the viscosity positively, and higher percentages of WEO negatively. It means by changing the dosages, the viscosity changes. All samples had different viscosity as their means lie in the same subset as the virgin binder.

5.2. Complex Modulus

The analysis of the complex fundamental value at 1 Hz is shown in Table 2. It has been observed that PPA significantly improved high-temperature performance. As indicated by statistical analysis, Polyphosphoric Acid has significantly increased the complex modulus values by making the binder stiff. According to prior studies, PPA significantly raises complex modulus values and stiffens the binder (Baldino *et al.* 2013, Hao *et al.* 2019). Therefore, lower dosages of PPA are recommended.

Table 2. Statistical Analysis of Complex Modulus.

Statistical Analysis of Complex Modulus							
Additives	**N**	**Subset at 95% confidence interval**					
		1	**2**	**3**	**4**	**5**	**6**
B+15%WEO+20%CR	3	147.65					
B+15%WEO+25%CR	3		167.34				
B+10%WEO+20%CR	3			189.47			
B+15%WEO+30%CR	3			191.95			
B+5%WEO+20%CR	3				207.96		
B+10%WEO+25%CR	3					218.87	
B+5%WEO+25%CR	3						246.41
Sig.		1.00	1.00	1.00	1.00	1.00	1.00
Additives	**N**	**Subset at 95% confidence interval**					
		7	**8**	**9**	**10**	**11**	**12**
B+10%WEO+30%CR	3	283.65					
B+5%WEO+30%CR	3		317.76				
Virgin Binder	3			353.72			
B+5% WEO+30 % CR+0.6%PPA	3				787.36		
B+5% WEO+30 % CR+1.2%PPA	3					1130.91	
B+5% WEO+30 % CR+1.8%PPA	3						2170.85
Sig.		1.00	1.00	1.00	1.00	1.00	1.00

5.3. Moisture Susceptibility Tests

Statistical analysis of POTS values acquired through BBS under both dry and wet conditions is given in Table 3. Adding 5% WEO and 30% CRB with 0.6% PPA affects the strength positively and

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higher percentages negatively. A concentration of PPA greater than 0.6% does not affect POTS (dry and wet) values. It is noticed that Virgin Binder, B + 5% WEO + 30% CRB + 1.2% PPA, and B + 5%WEO + 30% CRB + 1.8% are in the same subset as compared to the original binder. Although values increase slightly, there is no significant improvement effect as per statistics. That's why they are lying in the same subset. The values less than 0.05 have a significant effect, which means different performance as their means lie in a different subset than the virgin binder.

Table 3. Statistical Analysis of Moisture Susceptibility.

Statistical Analysis of Moisture Susceptibility						
Additives	**N**	**Subset at 95% confidence interval**				
		1	**2**	**3**	**4**	**5**
B+15%WEO+20%CR	3	1.3				
B+10%WEO+20%CR	3		1.57			
B+15%WEO+25%CR	3			1.775		
B+10%WEO+25%CR	3				2.180	
B+15%WEO+30%CR	3				2.305	2.305
B+5%WEO+20%CR	3					2.423
Sig.		1.000	1.000	1.000	1.000	1.000
Additives	**N**	**Subset at 95% confidence interval**				
		6	**7**	**8**	**9**	
B+10%WEO+30%CR	3	2.64				
B+5%WEO+25%CR	3	2.83				
B+5%WEO+30%CR	3		3.305			
B+5% WEO+30 % CR+0.6%PPA	3		3.445	3.445		
B+5% WEO+30 % CR+1.2%PPA	3			3.622	3.620	
Virgin Binder	3				3.70	
B+5% WEO+30 % CR+1.8%PPA	3				3.80	
Sig.		0.079	0.444	0.144	0.119	

6. Conclusions and Recommendations

The following conclusions have been deduced from this study:

- The conventional bitumen test results confirmed that WEO with crumb rubber in the binder increased the penetration and reduced the softening point and consistency because waste engine oil softened the bitumen. The best combination of 5% WEO and 30% CR seems closest to virgin binder in terms of its properties where an increase of 20% was observed in the penetration value, while an 8% decrease as found the softening point compared to a virgin binder. To improve it further, this combination has been further modified by adding 0.6%, 1.2%, and 1.8% PPA. Best results were observed at 1.8% PPA which improved the consistency of partially synthetic bitumen by decreasing its penetration up to 3% and increasing its softening point to 2%.
- The RV results revealed that incorporating CR and WEO into bitumen reduced viscosity. It was due to the dissolving of the asphaltene content of the binder, which is mainly responsible for its viscosity. Adding 5% WEO and 30% CR by total binder content reduced viscosity by 2 % The best combination of 5% WEO and 30% CR seems closest to virgin binder in terms of its viscosity. Further, the addition of 1.8% PPA increased the viscosity of the virgin binder by 2%.
- The frequency sweep test results concluded that combining WEO and CRB in the asphalt binder imparts high-temperature performance by decreasing the complex modulus. With the addition of 5% WEO and 30% CR by total binder content, the Complex modulus decreased by 21%, and the phase angle increased by 4%. The rut resistance and complex modulus improved by adding 1.8% PPA into the asphalt binder. Complex modulus increased by 20%, and the phase angle improved by 1%. Its mean PPA increased the complex modulus values by stiffening the binder.

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- With the addition of 5% WEO and 30% CR by total binder content, after 24 hours of dry and wet conditioning, POTS values decrease by 13% and 9%. It is because the WEO softens the bitumen, which makes it more susceptible to moisture damage. But the higher dosage of WEO, more than 5% WEO, proved harmful against moisture resistivity. However, adding crumb rubber can improve the resistance against moisture for better adhesion but does not fulfill the binder requirement. PPA improved the bonding strength of asphalt binder with the addition of 1.8% PPA, and POTS values increased by 5% and 4% after 24 hours of dry and wet conditioning.
- Statistical analysis results are compatible with the present results and previous literature. In comparison, PPA improved high-temperature performance remarkably well. The statistical study determined that PPA analyzed its susceptibility to moisture remarkably well.

Overall conclusions state that 5% WEO with the 30% CR addition can be utilized as a bitumen additive to make it sustainable by effectively and adequately disposing of tons of WEO and scrap tires. The dosages of PPA (1.8%) should be added with the WEO and crumb rubber to meet the performance requirement. The optimum percentage of industrial waste used to produce partially synthetic bitumen is 35% to make it workable. It can be used as a partial replacement for bitumen and to save non-renewable resources, protect the environment, and make it cost-effective.

7. Recommendations

This study is only limited to evaluating moisture resistance, rutting, and fatigue at binder level. There is still a requirement for in-depth analysis at asphalt mixture level to reach authentic findings based on the results.

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