

# CHARACTERISATIONS OF BIO-ASPHALT INCORPORATING PALM BIO-OIL FROM WASTE EMPTY FRUIT BUNCHES

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## ABSTRACT

*Bio-asphalts feature economic, social, and environmental benefits and may thus be potentially substituted for petroleum-based asphalt in asphaltic concrete mixtures. This article presents the experimental investigation of adding palm bio-oil derived from empty fruit bunches (WBO) into the conventional asphalt binder at 5, 10 and 15 wt%. The physical properties of bio-asphalt were assessed via the penetration, softening point, rotational viscosity, ductility, and mass loss after the rolling thin-film oven (RTFO) tests. Fourier-transform infrared spectroscopy (FTIR) and gas chromatography-mass spectrometry (GC-MS) tests were used to identify the chemical composition and functional groups of the bio-oil and bio-asphalt samples. The binder coatability was assessed by static water immersion and water boiling tests. The addition of bio-oil lowers the softening point and viscosity of the binder while increasing its penetration, ductility and mass loss after RTFO. The main absorption peaks for WBO bio-oil and WBO bio-asphalts were corresponding to C=H stretching of alkanes and aliphatic compounds, respectively. The bio-oil was mostly composed of aromatic and oxygenated compounds, as observed from GC-MS results, which justified the FTIR analysis for WBO bio-oil. The addition of WBO bio-oil improved the bonding characteristics of asphalt-aggregates, indicating that it has a significant potential for use in the production of bio-asphalt.*

**Keywords:** bio-oil, chemical composition, empty fruit bunches, physical properties, renewable resources.

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## INTRODUCTION

Crude oil is the largest energy resource in the world, including asphalt binder as the main ingredient in road paving material. Several challenges have emerged, such as increasing crude oil consumption, the uncertainty in crude oil prices, and its nature as a non-renewable resource (Lin and Bai, 2021; Maghyereh and Sweidan, 2020; Mensi *et al.*, 2020; Razek and Michieka, 2019). Malaysia is one of the oil producers, Petroliaam Nasional Berhad, known as Petronas, produces and handles crude oil production.

For the last 25 years, the price of crude oil has been unpredictably fluctuating as shown in *Figure 1*. In particular, the crude oil price was constantly increasing until it reached a peak of USD140.00 per barrel in 2008. The price thereafter began to fluctuate, reaching the second high of USD119.52 per barrel in January 2022 (Trading Economics, 2022). *Figure 2* also indicates that oil consumption has been increasing since 2006, with global demand estimated to reach 104.1 million barrels per day in 2026 (Statista, 2019). To address these challenging problems, industries and researchers have conducted extensive work in search for “green technology” to replace or substitute conventional binders. As a result, there is pressing demand for alternatives to conventional asphalt binders. The utilisation of biomass resources for bio-asphalt production as either a partial or complete substitute for asphalt binder derived from crude oil has been used as an alternate method for producing road paving materials. The substitution may reduce the dependency on the crude oil-based

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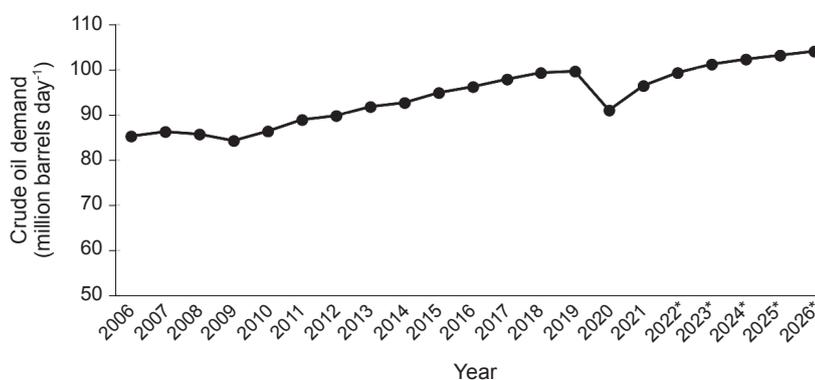
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Source: Trading Economics (2022).

Figure 1. The trend of crude oil price for the last 25 years.



Source: Statista (2019).

Figure 2. Global demand for crude oil.

asphalt binder (Sun *et al.*, 2016; Wen *et al.*, 2013; Yang *et al.*, 2013).

Biomass sources have come to the minds of researchers due to their accessibility and environmental friendliness (Wang *et al.*, 2020a). Biomass material is a viable alternative since it may be converted into bio-oil by pyrolysis or hydrothermal liquefaction. It is renewable, eco-friendly and has rheological properties comparable to conventional asphalt. Bio-oil may be derived from a wide range of biomass sources throughout the globe, including algae (Abdul Latif *et al.*, 2019), switchgrass (Yue *et al.*, 2017), corn stover (Yang *et al.*, 2017), swine manure (Fini *et al.*, 2011; Mills-Beale *et al.*, 2014; Wang *et al.*, 2020b), waste wood resources (Gürer *et al.*, 2020; Ingrassia *et al.*, 2020; Yang *et al.*, 2013; 2014) and waste cooking oil (Sun *et al.*, 2016; Wen *et al.*, 2013; Zahoor *et al.*, 2021). Particularly in Malaysia, the extraction of palm oil results in a substantial amount of biomass waste, including mesocarp fruit fibres (MF), palm kernel shells (PKS), palm oil mill effluent (POME), and empty fruit bunches (EFB). These palm oil wastes might serve as biomass feedstock for bio-oil production, especially the EFB (Abdullah *et al.*,

2011; 2017; Chang, 2018; Pogaku *et al.*, 2016; Rosli *et al.*, 2017; Yiin *et al.*, 2016).

Over the years, it has been established that much research has been undertaken in quest of an asphalt binder alternative. However, there is still limited research on EFB-based bio-asphalt. As oil palm plantations in Malaysia have generated a significant amount of biomass waste, it is suggested that EFB might be utilised to produce bio-oil in advance of bio-asphalt. Therefore, in this experimental study, bio-oil produced from EFB (designated as WBO bio-oil) was used to partially replace the asphalt binder at 5, 10 and 15 wt%. This research assessed the physical properties, chemical characteristics, and coatability of bio-asphalts.

## MATERIALS AND METHODS

### Materials

The Malaysian Palm Oil Board (MPOB), Bangi, Selangor, Malaysia, provided the WBO bio-oil used in this investigation, which was used in the experiment as received. It was derived from the

pyrolysis of EFB at 500°C for a residence time of 30 min. The elemental analysis revealed that carbon was the most prevalent element in the chemical composition of the bio-oil. The chemical formula of bio-oil can be written as  $\text{CH}_{1.92}\text{O}_{0.61}\text{N}_{0.03}$ , and its characteristics are shown in *Table 1*. In asphalt mixtures, the use of EFB will enhance waste recycling and sustainable material consumption. This will aid in reducing the depletion of non-renewable resources as well as the usage of substantial amounts of energy and greenhouse gas emissions. The higher heating value (HHV) or calorific value of bio-oil is 40% to 50% lower than that of petroleum fuel, indicating that bio-oil releases less energy in its compounded state, making it more stable or less energetic. Besides, conventional 60/70 penetration grade (60/70 PEN) binder and crushed granite aggregates with properties conforming to Malaysian Public Works Department (PWD) or Jabatan Kerja Raya (JKR) standard specifications (JKR, 2008) were adopted in this study. *Table 2* displays the physical parameters of the standard binder.

### Preparation of Asphalt Binder Samples

The 60/70 PEN asphalt binder was used as the base binder. The WBO was added to the base binder and mixed using a propeller mixer at 145°C and 1000 revolutions per minute (rpm) for 30 min to prepare bio-asphalt. The pre-heated asphalt binders were blended with WBO bio-oil at 5, 10 and 15 wt%. These products were designated as 5%, 10% and 15% WBO bio-asphalts, and their characteristics and performance were evaluated.

TABLE 1. CHARACTERISTICS OF WBO BIO-OIL DERIVED FROM EFB

Properties	Units	WBO bio-oil
Elemental analysis	C	49.80
	H	7.98
	N	1.93
	O	40.29
H/C molar ratio		1.92
O/C molar ratio		0.61
Water	wt%	18.74
Calorific value	MJ kg <sup>-1</sup>	21.41
Molecular formula		$\text{CH}_{1.92}\text{O}_{0.61}\text{N}_{0.03}$

### Preparation of Loose Asphalt Mixture Samples

Granite was used as the aggregate in the loose asphalt mixture prepared for the static water immersion and water boiling tests. The asphalt mixture preparation technique followed the standard of AASHTO PP2 (AASHTO, 2001). The asphalt mixture was prepared by mixing the base binder or bio-asphalt with the designated aggregate sizes. The asphalt binder was pre-heated at 160°C for 2 hr before mixing. Meanwhile, aggregates were pre-heated for at least 4 hr at the same temperature. During the mixing process, a designated percentage of binder was added to the aggregate and mixed for approximately 1 min to obtain a well-coated, loose mixture. After mixing, the mixture was placed on a metal tray and conditioned for 2 hr at 150°C in the oven to facilitate the binder absorption of aggregates.

### Physicochemical Analysis

**Fourier Transform Infrared Spectroscopy (FTIR).** FTIR-Attenuated Total Reflectance (ATR) test was conducted using a Bruker Tensor 27 (Shimadzu, model IR Prestige-21). The test was conducted to identify the functional groups present in samples of WBO bio-oil and bio-asphalt. Generally, the FTIR test produced a spectrum with peaks illustrating transmittance against wavenumber. A prominent peak observed at a specific wave number indicated the existence of the specified functional group in the samples that were tested. For this analysis, approximately 0.1 to 0.4 mL of sample was placed over the surface of a diamond, and the analysis was performed approximately three times with 4 cm<sup>-1</sup> resolutions with a range of 4000 to 600 cm<sup>-1</sup>. The FTIR graph illustrates transmittance *vs.* wavenumber. The *x*-axis represents wave number ranging from 500 – 4000 cm<sup>-1</sup>, with >1500 cm<sup>-1</sup> representing the functional group region and <1500 cm<sup>-1</sup> representing the fingerprint region. The peak may be classified as strong, moderate, and weak (Hareru and Ghebrab, 2020). In addition, the *y*-axis displays transmittance ranging from 0%-100%. This test is performed according to ASTM D7414 (ASTM, 2009b).

**Gas Chromatography-Mass Spectrometry (GC-MS).** GC-MS was performed according to ASTM D8276 (ASTM, 2019a) to determine the nature and type of

TABLE 2. PHYSICAL PROPERTIES OF THE CONVENTIONAL BINDER

Properties	Units	Specification range	Results	References
Penetration (at 25°C, 100 g, 10 s)	0.1 mm	60-70	64	ASTM (2013)
Softening point	°C	48-56	49.5	ASTM (2014)
Ductility (at 25°C)	cm	>100	>100	ASTM (2017)
Density	g cm <sup>-3</sup>	1.01 to 1.06	1.03	ASTM (2009a)

organic compounds in WBO bio-oil and bio-asphalts. This analysis was conducted using Shimadzu GC2010 Plus equipped with a quadrupole detector (GCMS-QP2010) and a BPX5 capillary column (20 m film thickness x 0.25 mm). The temperature of the column oven was maintained at 50°C before being raised by 2°C min<sup>-1</sup> to 180°C withhold 3 min, then to 280°C at the same rate withhold 5 min, and finally to 300°C by 2°C min<sup>-1</sup> withhold 7 min. The injector temperature was kept at 290°C. At a constant flow rate of 1 mL min<sup>-1</sup>, helium was employed as the carrier gas. At the same time, the potential chemical components in WBO bio-oil and bio-asphalts were studied using the Mass Spectrometry (MS) search libraries.

### Binder's Physical Test

**Penetration test.** A penetration test measures consistency by determining the binder's grade via the penetration value. This test was conducted using a penetrometer with the standard loaded needle penetrating vertically in 5 s while the temperature of the asphalt binder sample was maintained at 25°C in accordance with ASTM D5 (ASTM, 2013).

**Softening point test.** Based on ASTM D36 (ASTM, 2014), the softening point (SP) of asphalt binders was determined through the ring and ball test. Asphalt binder is a viscoelastic material that becomes softer and less viscous as the temperature rises.

**Penetration index.** The penetration index (PI) is used to measure the temperature susceptibility of a binder based on the linear relationship between log penetration and temperature. Pfeiffer and Van Doormal (1940) have developed an equation to establish the relationship between the asphalt binder consistency and the temperature sensitivity at the temperature of 25°C based on the penetration value and softening point. The equation is shown as Equation (1). All asphalt binder samples exhibit thermoplastic properties, becoming softer when heated and hardening when cooled.

$$PI = \frac{1952 - 500 \log Pen - 20 \text{Softening Point}}{50 \log Pen - \text{Softening Point} - 120} \quad (1)$$

**Rotational viscosity (RV) test.** RV test was conducted to determine the viscosity of asphalt binders at high temperatures. The rotational viscometer measured the torque required to maintain the rotating speed of the spindle in the asphalt binder at a constant rate. In this test, four different temperatures (120°C, 140°C, 160°C and 180°C) were used to test the control and bio-asphalt binders. A #27 spindle was used, and a constant rotating speed of 20 rpm was set. The standard

procedures were in accordance with ASTM D4402 (ASTM, 2015).

**Ductility test.** Based on ASTM D113 (ASTM, 2017), the ductility test measures the asphalt binder's ability to stretch. It is measured by the distance, in centimetres, to which a sample will elongate before breaking at a specific temperature and speed. The pulling rate of the ductilometer was maintained at 50 mm min<sup>-1</sup>, while the water bath temperature was kept around 25°C. Insufficient ductility in the binder will lead to lower resistance to fatigue cracking under repeated traffic loads.

**RTFO mass change.** Based on ASTM D2872 (ASTM, 2019b), a rolling-thin film oven (RTFO) is conducted to mimic the short-term aging of the asphalt binder. The RTFO test studies the effect of heat and air on a moving film of asphalt binder under rotation. Asphalt binder experiences short-term aging during transportation, mixing, storage, and handling processes. The RTFO provides a quantitative measure of the volatile loss (mass change) during the aging process. The mass change of the asphalt binder can be determined by using Equation (2).

$$\text{Mass change} = \frac{m_2 - m_1}{m_1 - m_b} \quad (2)$$

where  $m_1$  is the mass of the RTFO bottle with the asphalt sample before aging, and  $m_2$  is the mass of the RTFO bottle with the sample after aging. Then,  $m_b$  is the mass of an empty RTFO bottle.

### Binder Coatability Tests

**Static water immersion test.** The static water immersion test was conducted to determine the percentage of asphalt binder that remained coated on aggregate after being immersed in water. Each sample was represented by 100.0 g of oven-dry aggregates, prepared and mixed with 5.5 g of asphalt binder. The loose asphalt-coated aggregates were conditioned for 2 hr in an oven. After conditioning, the sample was transferred to a glass container and immersed in water at 25°C for 16–18 hr. Water infiltration between the thin asphalt binder film and aggregates may occur due to weak adhesion between the asphalt binder and aggregates. It caused the asphalt binder films to strip from the aggregates. A visual evaluation of the asphalt binder's coatability is conducted by observing the remaining covered area. The test was conducted in accordance with AASHTO T182 (AASHTO, 1993).

**Water boiling test.** Based on ASTM D3625 (ASTM, 2020), a standard protocol was performed to evaluate

the adhesion loss in a loose mix of aggregate coated with asphalt binder due to the boiling water effect. The sample containing 250 g of the uncompacted aggregate-binder mixture was prepared and boiled for about 10 min in a beaker filled with water. Before placing the mixture into the boiling water, the temperature of the mixture was checked to ensure that it was below the boiling point of the water but over 85°C. After 10 min of the boiling process, the excessive asphalt binder floating on the water surface was skimmed off to avoid recoating. The sample was then transferred onto a piece of the white paper sheet and cooled at room temperature. The level of stripping of the sample after cooling was determined using eye inspection.

## RESULTS AND DISCUSSION

### Physicochemical Analysis

**FTIR analysis.** Figure 3 shows the FTIR spectra for WBO bio-oil, control asphalt binder, and bio-asphalts prepared with 5, 10 and 15 wt% WBO incorporation. From the spectra data, the functional groups of WBO bio-oil were mainly phenols, alcohols, ketones, aldehydes, and carboxylic acids (Sukiran *et al.*, 2009). The existence of O-H stretching vibrations at 3400 cm<sup>-1</sup> implied the presence of alcohols, phenol, polymeric O-H and water. The absorbance peaks between 1812 cm<sup>-1</sup> and 1650 cm<sup>-1</sup> represented the C=O stretching vibrations and indicated the presence of ketones, aldehydes, carboxylic acids and their derivatives. Carbonyl components such as phenol and ester were detected by C-O stretching vibrations at 1263 cm<sup>-1</sup>. The presence of C-H plane bending within 900 cm<sup>-1</sup> to 700 cm<sup>-1</sup> indicated the presence of aromatic compounds, which was further verified by C=C stretching vibrations at 1602 cm<sup>-1</sup>.

Additionally, the FTIR spectra analysis results in Table 3 are consistent with the previous research using the same bio-oil (Sukiran *et al.*, 2009; Sutrisno and Hidayat, 2018). The FTIR study showed that the bio-oil composition was found to include highly oxygenated species, which were contributed by the pyrolysis of cellulose and lignin (Sukiran *et al.*, 2009). However, the FTIR spectra of WBO bio-oil and WBO bio-asphalt were distinct. The main absorption peaks for WBO bio-oil and WBO bio-asphalts were located at 3040 to 2800 cm<sup>-1</sup> and 1480 to 1300 cm<sup>-1</sup>, respectively, which correspond to the C-H stretching of alkanes and aliphatic compounds.

The spectra peak of the control binder (CS) was comparable to the WBO bio-asphalt with 5, 10 and 15 wt% WBO. Similar results were obtained with control asphalt, poppy capsule pulp bio-asphalt, and molasses bio-asphalt (Gürer *et al.*, 2020; Hareru and Ghebrab, 2020) due to the low bio-oil incorporation in the asphalt binder. Furthermore, the similar spectra indicated no substantial differences across functional groups, even when the dehydration occurred after bio-oil mixing with the control binder (Hareru and Ghebrab, 2020). After combining the conventional asphalt binder with WBO bio-oil in the current study, the absorbance for the wavenumber ranges of 900 cm<sup>-1</sup> to 700 cm<sup>-1</sup> and 1260 cm<sup>-1</sup> was lowered in all bio-asphalt samples.

**GC-MS analysis.** The chemical compounds in the asphalt binder, WBO bio-oil and WBO bio-asphalts are listed in Table 4. Phenol was the most abundant chemical in the bio-oil, with a percentage area of 20.69%. The majority of the chemicals discovered in bio-oil were determined to be lightweight chemicals. The other chemicals, such as phenol, 2,6-dimethoxy-(CAS)2,6-dimethyl and 1,2-benzenediol, were also detected in the bio-oil with percentage areas of 9.11% and 4.67%, respectively. The bio-oil was mostly

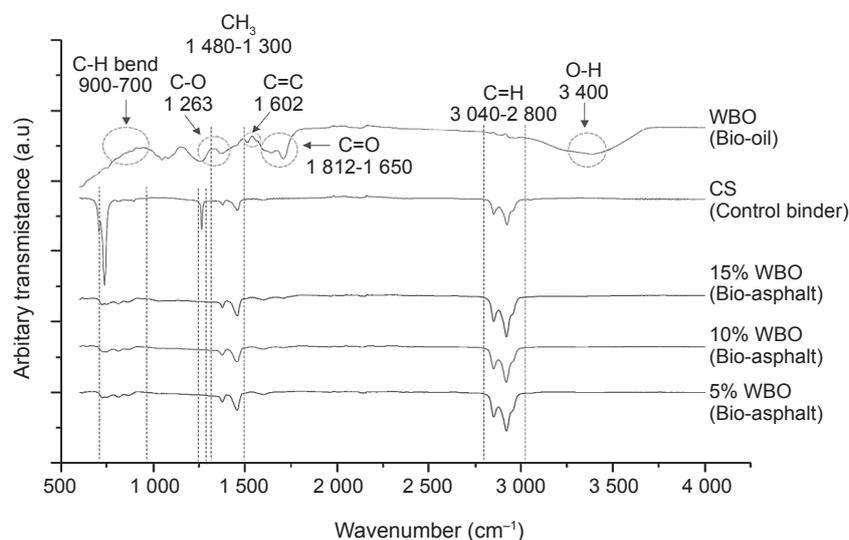


Figure 3. FTIR spectra analysis for bio-oil, asphalt and WBO.

TABLE 3. FTIR FUNCTIONAL GROUPS IDENTIFIED IN TESTED SAMPLES

Wavenumber (cm <sup>-1</sup> )	Functional group	Class of compounds
3 400	O-H stretching	Polymeric O-H, Water
3 040-2 800	C-H stretching	Alkanes
1 812-1 650	C=O stretching	Ketones, Aldehydes, Carboxylic acids
1 602	C=C ring stretch	Aromatic compounds
1 519	-NO <sub>2</sub> stretching	Nitrogenous compounds (usually do not appear in bio-oil spectra)
1 482-1 379	CH <sub>3</sub>	Aliphatic compounds
1 263	C-O stretching	Phenol, Ester
1 100-997	S=O stretching	Sulfoxide compounds (usually do not appear in bio-oil spectra)
900-700	C-H plane bending	Aromatic compounds

composed of aromatic and oxygenated compounds, according to the GC-MS data, which supported the FTIR study of WBO bio-oil.

Furthermore, the dominant compounds found in WBO bio-asphalts were dodecenoic acid, 1,2,3-propanetriyl ester, which consisted of 46.87% of the total sample. The other major compounds were Nonadecane (CAS) n-Nonadecane, myristic acid vinyl ester and fumaric acid, 3,3-dimethylbut-2-yl hexyl ester. Based on this analysis, the material characteristics and quality index of the bio-asphalt are dependent on the presence of different chemical compositions. Bio-asphalts that contain high levels of lightweight chemicals increase evaporation rate and mass loss. The lightweight chemical components in the 15% WBO bio-asphalt interact significantly with the stationary phase in the capillary column, leading to a longer retention duration. The substantial mass loss seen with 15% WBO bio-asphalt was proven and validated by the chemical profile from this research, which revealed a large number of lightweight elements.

### Binder's Physical Properties

**Penetration test results.** Figure 4 shows the penetration and softening point test results of the asphalt binders. As seen in Figure 4, the penetration value for the CS, 5% WBO bio-asphalt, 10% WBO bio-asphalt, and 15% WBO bio-asphalt was recorded as 64, 74, 82 and 90 dmm, respectively. The penetration values of bio-asphalt increased as the WBO content increased, thus indicating that WBO had the potential to reduce the stiffness of the binder.

**Softening point test results.** As shown in Figure 4, the higher bio-oil content has significantly reduced the softening point of the asphalt binder. Raman *et al.* (2015) also recorded a lower softening point after adding WBO as a partially replaced ingredient in a base binder. From the results, 15% WBO bio-asphalt had the lowest softening point (44.3°C), while the control binder had the greatest softening point (49.5°C). An appropriate dosage of WBO bio-oil

during the production of bio-asphalt is essential in ensuring the asphalt binder achieves an appropriate characteristic.

The penetration index ranges from -2 for a high-temperature susceptible binder to roughly +2 for a highly blown low-temperature susceptible binder. A binder with a penetration index within the range of -2 to +2 is suitable for use as a conventional paving asphalt binder (Read and Whiteoak, 2003). Theoretically, a lower softening point signifies a low viscosity asphalt binder, resulting in a higher penetration index, which is typically utilised in cold climates. Meanwhile, a binder with a lower penetration index is preferred in countries with warm climates due to its higher stiffness. According to the penetration index analysis findings in Table 5, WBO bio-asphalt has the potential to be used as a conventional paving asphalt binder in road construction. However, it would be premature to establish an optimal ratio for road construction at this stage. More research is needed to determine the long-term performance and durability of bio-asphalt mixtures in extreme conditions.

**Rotational viscosity test results.** Figure 5 illustrates that the viscosity of the bio-asphalts altered drastically independent of the WBO bio-oil dosages throughout the range of testing temperature. The addition of WBO bio-oil lowered the viscosity of the binder, implying that the bio-asphalt had higher workability than the CS due to its lower viscosity, which would consume less energy during the mixing process and throughout the pavement construction stage. The rotational viscosity value of all the samples was lower than 3 Pa·S at 135°C, which is the maximum permissible rotational viscosity value according to the Superpave standard to avoid excessively high mixing and compaction temperatures (AASHTO, 2016; ASTM, 2018). The laboratory mixing and compaction temperatures of the asphalt binders were determined based on the ideal viscosity ranges of 0.17 ± 0.02 Pa·S (mixing temperature range) and 0.28 ± 0.03 Pa·S, respectively (Asphalt Institute, 1989). Table 6 shows the optimum mixing and compaction temperatures for all asphalt binders. Theoretically,

TABLE 4. CHEMICAL COMPOUND DISTRIBUTION IN WBO BIO-OIL, CONTROL BINDER AND WBO BIO-ASPHALTS

Compounds	CS	WBO bio-oil	Bio-asphalt		
			5% WBO	10% WBO	15% WBO
1-Hydroxy-2-butanone		✓			
2-Cyclopenten-1-one		✓	✓		
Urea, N-[5-(ethylsulfonyl)-1,3,4-thiadiazol-2-yl		✓	✓		
1,2-Ethandiol, diacetate (CAS) Ethylene diacetate		✓	✓		
2(5H)-Furanone		✓	✓		
Phenol (CAS) Izal		✓			
4H-Pyran-4-one		✓	✓		
2,3-Dimethyl-2-cyclopenten-1-one		✓	✓		
Cresol <ortho->		✓			
Phenol, 4-methyl- (CAS) p-Cresol		✓			
Phenol, 2-methoxy-		✓			
Cyclohexane-1,2-dione <3-methyl->		✓	✓		
2-Cyclopenten-1-one, 3-ethyl-2-hydroxy-		✓			
Xylenol <2,5->		✓	✓		
Creosol		✓			
Benzoic acid (CAS) Retardex		✓	✓		
Phenol, 2-methoxy-4-methyl-		✓			
1,2-Benzenediol		✓	✓		
1,4:3,6-Dianhydro-.alpha.-d-glucopyranose		✓	✓		
1,2-Benzenediol, 3-methoxy-		✓	✓		
Ethanone, 1-(2,5-dihydroxyphenyl)-		✓	✓		
1,2-Benzenediol, 4-methyl-		✓			
Syringol <4-methyl->		✓			
Ethanone, 1-(4-hydroxy-3-methoxyphenyl)- (CAS) Acetovanillone		✓	✓		
Fumaric acid, cyclopentylmethyl isohexyl ester	✓		✓	✓	
Heptadecane, 8-methyl-	✓		✓	✓	
Dodecanoic acid, ethenyl ester	✓		✓	✓	
Fumaric acid, hexyl 2-methyl allyl ester	✓		✓	✓	
V Dodecanoic acid, 1-(hydroxymethyl)-1,2-ethanediyl ester	✓			✓	
Dodecanoic acid, ethenyl ester	✓			✓	
Butane, 1,1'-oxybis[4-iodo]-	✓				
Dodecanoic acid, 1,2,3-propanetriyl ester	✓		✓	✓	✓
Fumaric acid, 2-ethylhexyl isohexyl ester	✓		✓	✓	
Dodecanoic acid, ethenyl ester (CAS) Vinyl dodecanoate	✓				✓
Dodecanoic acid, 1,2,3-propanetriyl ester	✓				
Dodecanoic acid, ethenyl ester	✓				✓
Fumaric acid, 2-butyl hexyl ester	✓				✓
Dodecanoic acid, 1,2,3-propanetriyl ester	✓				✓
Dodecanoic acid, 1,2,3-propanetriyl ester	✓		✓	✓	
Dichloroacetic acid, undecyl ester					✓
Octadecanoic acid					✓
Hexadecane					✓
Oxalic acid			✓	✓	✓
Eicosanoic acid					✓
Aziridine					✓
4-Pentylcyclohexanone			✓		✓
Azacyclohexane			✓		✓
4-Nitrophenyl laurate	✓			✓	
Myristic acid vinyl ester	✓			✓	
Glyceryl tridodecanoate				✓	
Glyceryl tri-2,2-dideuterio dodecanoate				✓	✓
Oxalic acid, octadecyl propyl ester				✓	✓

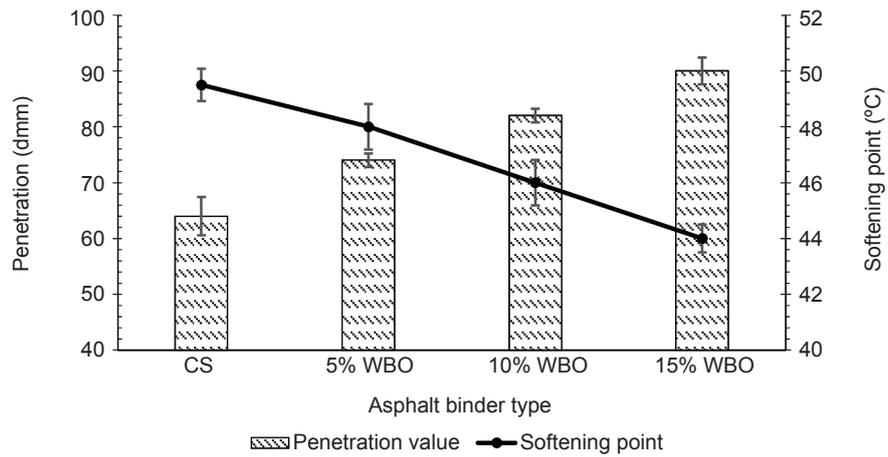


Figure 4. Penetration and softening point test results of asphalt binders.

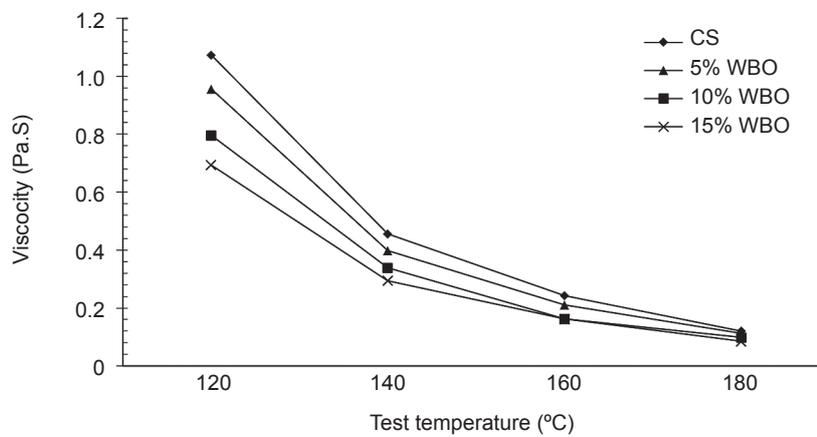


Figure 5. Rotational viscosity test results.

TABLE 5. THE PENETRATION INDICES OF THE CONTROL AND BIO-ASPHALT BINDERS

Designation	Binder type	Penetration index	Best possible use
CS	PEN 60/70	-0.7439	Conventional paving asphalt binder
5% WBO		-0.7699	Conventional paving asphalt binder
10% WBO	Bio-asphalt	-1.0680	Conventional paving asphalt binder
15% WBO		-1.3348	Conventional paving asphalt binder

Note: % is referring to wt%.

TABLE 6. RANGE OF MIXING AND COMPACTION TEMPERATURES FOR EACH SAMPLE

Binder	Temperature (°C)	
	Mixing	Compaction
CS	169-175	153-159
5% WBO	164-172	149-156
10% WBO	157-164	143-150
15% WBO	156-163	139-147

the WBO bio-asphalt has higher workability than the conventional binder, resulting in decreased energy consumption during the mixing and compaction process of the asphalt mixture.

**Ductility test results.** Figure 6a illustrates the ductility test results of the asphalt binders. In comparison to the CS, the use of WBO in partially replacing the asphalt binder significantly improved its ductility regardless of the amount of bio-oil added. The bio-oil significantly lowered the viscosity of the asphalt binder. The CS and bio-asphalts presented adequate ductility by meeting the JKR standard specification. This is essential to reduce the fatigue cracking potential typically induced by the continuous, repetitive traffic loadings.

**Mass loss after RTFO.** Figure 6b presents the mass loss for the control and bio-asphalt samples. From

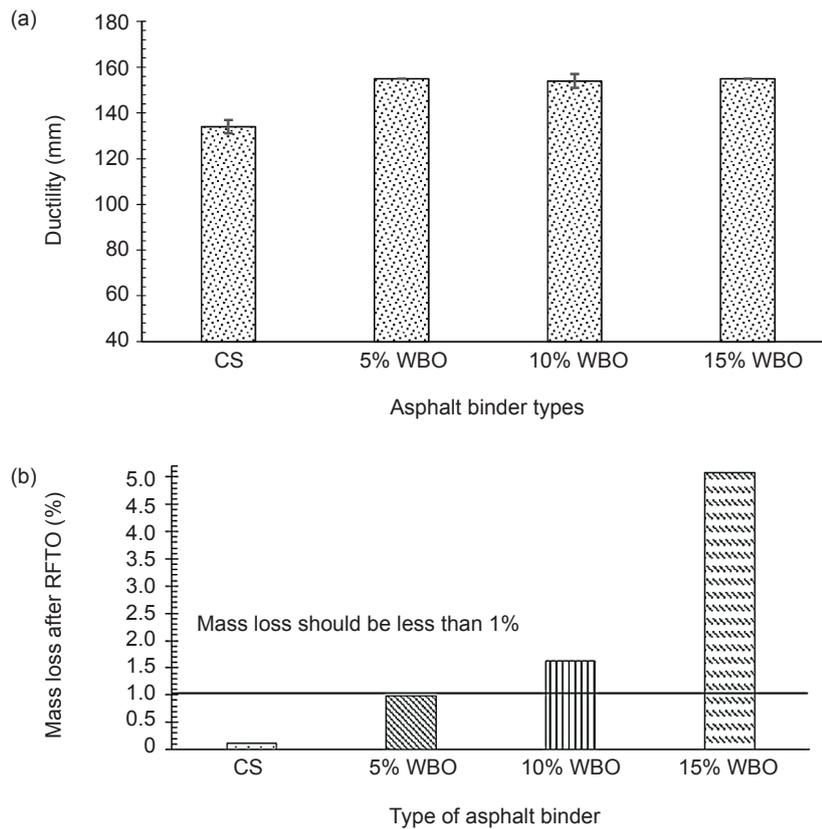


Figure 6. (a) Ductility values for the control and bio-asphalts, (b) RTFO mass loss test results.

the results, the mass loss of CS and 5% WBO bio-asphalt binders conforms to the Superpave requirement of less than 1.00%. In contrast, the 10% WBO and 15% WBO bio-asphalts had surpassed the requirement with mass losses of 1.62% and 5.09%, respectively. The higher the integration of WBO bio-oil, the greater the mass loss of bio-asphalts. The high mass loss is caused by its high water content of 18.74% and the presence of volatile elements, as reported by Sukiran *et al.* (2009). It is suggested that pre-treatment or pre-heating is required to eliminate the excess water content in WBO bio-oil. As a result, the 10% WBO bio-asphalt and 15% WBO bio-asphalt are less recommended for road construction without any pre-requisite treatment owing to their substantial mass loss that exceeds the standard specification.

#### Binder Coatability Index in Asphalt Mixtures

**Static water immersion test.** Table 7 displays the coatability index or the percentage of the coated region that remained after 16 hr in water at 25°C. From observation (Figure 7), all the asphalt binders demonstrated high retained coated aggregate areas of more than 95% after the static water immersion test. This indicated that the WBO bio-asphalts exhibited equivalent adhesion strength to the CS under the static effect of moisture. All

binder samples displayed good coatability and low stripping potential.

**Water boiling test.** Table 7 summarises the results of the water boiling tests for control and bio-asphalt binders. From visual observation (Figure 8), the addition of WBO bio-oil enhanced the coatability of the loose asphalt mixture when compared to CS. Furthermore, the higher bio-oil incorporation increased the aggregate areas that remained coated. Therefore, it is implied that WBO bio-asphalts help to strengthen the bonding between asphalt binder film and aggregates.

#### CONCLUSION

WBO bio-asphalt is being researched as an alternative resource to replace the conventional asphalt binder, as well as to solve the problem of empty fruit bunch disposal. The physical and chemical parameters of 5, 10 and 15 wt% WBO integrated bio-asphalt were established by laboratory testing and data analysis. WBO bio-oil decreases the softening point and viscosity of the binder while increasing the penetration and mass loss after RTFO when compared to the control binder. The ductility of the WBO bio-asphalt rises regardless of the quantity of WBO bio-oil supplied. According to the mass

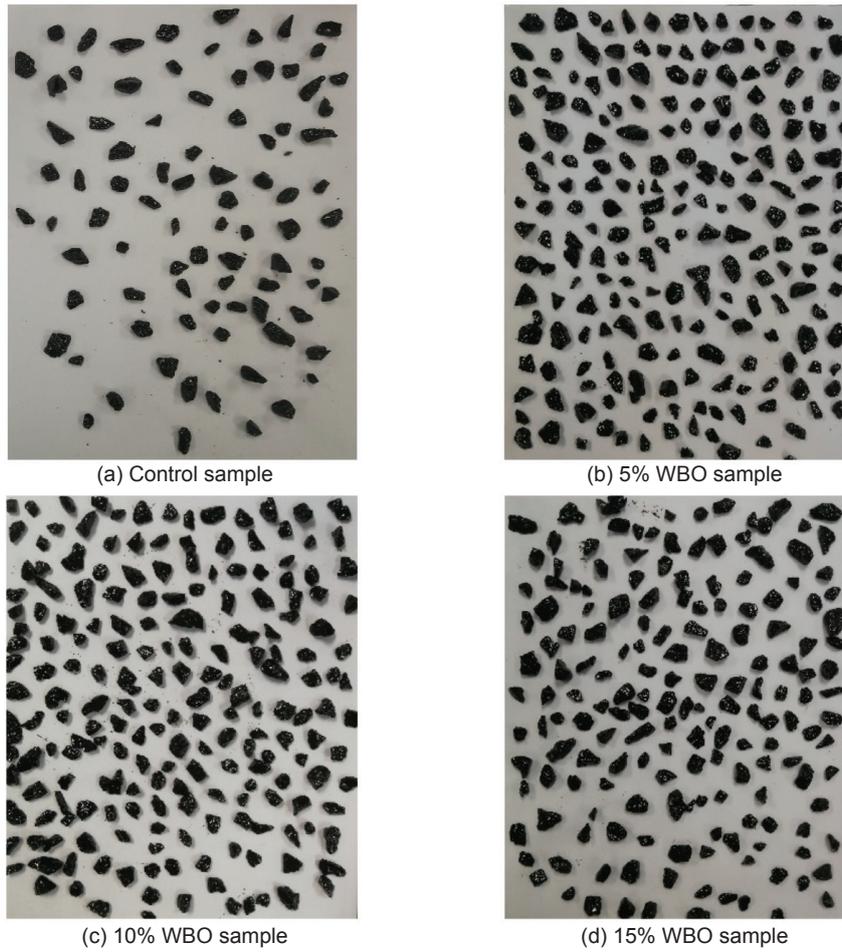


Figure 7. Observation of the aggregate coating in static water immersion test.

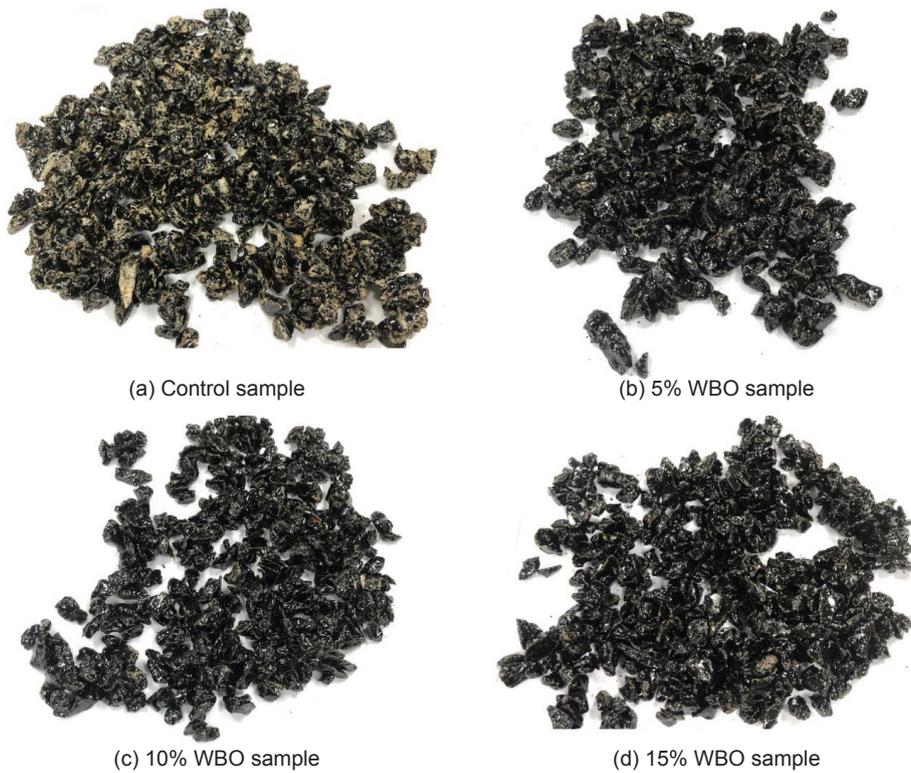


Figure 8. Binder coatability of loose mixes in water boiling test.

**TABLE 7. BINDER COATABILITY IN ASPHALT MIXTURES: STATIC WATER IMMERSION TEST AND WATER BOILING TEST**

Sample	Static water immersion test	Water boiling test
	Coatability index (%)	
CS	>95	47.5
5% WBO	>95	82.5
10% WBO	>95	90.0
15% WBO	>95	95.0

loss results, only the 5% WBO bio-asphalt achieved a mass loss of less than 1%, as required by the Superpave standard. The substantial mass loss is attributed to the material's high water content and the presence of volatile elements, according to physicochemical analyses. As a consequence, a pre-treatment of the WBO bio-oil to remove excess water should be considered. However, it would be premature to recommend an optimal proportion of bio-oil to be used in the production of bio-asphalt at this moment. More research should be done to assess the long-term performance of bio-asphalt in severe conditions. Besides, WBO bio-oil also contains phenol, alcohols, ketones, aldehydes, aromatic compounds, and carboxylic acids. The GC-MS analysis results showed that phenol and dodecenoic acid, 1,2,3-propanetriyl ester, are dominant in WBO bio-oil and bio-asphalt, respectively. In static water immersion and water boiling tests, all WBO bio-asphalts displayed increased coatability and strengthened adhesion strength between the asphalt binder film and aggregates. As a result, WBO bio-asphalt has a significant potential for application in the development of bio-asphalts as a partially substituted material.

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