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Chemical characterization of asphalt binder containing palm oil mill sludge

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Abstract. Modification of asphalt binder is continuously explored due to its escalating cost and increasing demand for this non-renewable material. As an alternative, the potential of waste materials was assessed for use as a modifier in asphalt binder. This study focuses on investigating the physical properties of unmodified and modified asphalt binders, with a specific emphasis on the chemical properties of palm oil mill sludge (POMS) modified asphalt binder. In this investigation, the control sample employed was PEN 60/70, while the POMS content ranged from 0% to 5% with an increment of 1%. Penetration and softening point tests were conducted on the POMS-modified binder, and Fourier Transform Infrared Spectroscopy (FTIR) tests were conducted to assess the chemical properties of both un-aged and short-term aged asphalt binders. The results revealed that the addition of POMS modified the asphalt binder by inducing a softening effect proportional to the percentage of POMS. The aging process was found to be significantly delayed in the POMS-modified binder with increasing POMS content.

1. Introduction

Malaysia has been one of the main producers and exporters of palm oil products in recent decades [1]. Annually, the global production volume of palm oil continues to rise, resulting in an escalation in both effluent discharge and palm oil mill waste to the environment. The palm oil mill sludge (POMS) is a highly polluting material that contributes significantly to environmental contamination in Malaysia as a result of the oil extraction process [2]. Before being released into the streams and rivers, POMS must undergo a treatment to neutralize pollutants such as heavy metals and organic compounds. Unfortunately, because a large amount of palm oil sludge is produced at a time, treatment of this wastewater is expensive and difficult to manage [3]. The present binder materials, on the other hand, have a problem with bitumen aging. The impact of aging is that it diminishes the pavement's performance and durability while also affecting its cracking resistance [4]. Due to changes in the viscoelastic behavior of the materials over time, the problem of asphalt binder aging will also have a significant impact on stiffness and brittleness [5].

The economic and environmental benefits have led to an increase in the use of recycled materials in asphalt binders. Most research uses biomass materials, which can improve the low-temperature



performance as well as its resistance to deformation at high temperatures [6]. According to Zhang et al [6], biomass-based materials exist in various forms such as ash, oil or char form and when blended with asphalt, it is known as bio-asphalt. The performance of bio-asphalt will depend on the source or type of biomass materials. Previous investigations of asphalt binder modification using palm oil fly ash (POFA) have shown that it improves the performance of the asphalt binder over its service life [7]. The proportion employed in the alteration will also influence its performance. Based on the findings, adding POFA to the binder improves the binder's resistance to oxidative aging [7]. Another study by Xin et al. [8], found that the palm bio-oil derived from empty fruit bunch softened the asphalt binder due to increasing penetration value, reduced softening point and increased the ductility of asphalt binder.

POMS modified binder is a superior approach for POMS treatment. POMS can be used as an excellent raw material for bioconversion employing a variety of biotechnology methods. As a result, POMS is a substance that can be employed as a recycling binder in an asphalt binder modification to address aging, deformation and environmental concerns in asphalt pavement construction [6]. The POMS-modified asphalt binder is expected to enhance the asphalt pavement performance while also helping to solve the environmental challenge. Thus, this study aims to investigate the effects of POMS on the chemical properties of asphalt binder.

2. Experimental Procedure

2.1. Materials

In this study, there were two main materials employed: asphalt binder with a penetration grade of 60/70, as well as palm oil mill sludge (POMS) as a modifier. Figure 1 presents the raw material of POMS. Asphalt binder with penetration grade of 60/70 used in this study was supplied by Kemaman Bitumen Company (KBC). Meanwhile, POMS with different proportions (0%, 1%, 2%, 3%, 4% and 5%) by total weight of the asphalt binder were used for this study.



Figure 1. The raw material of palm oil mill sludge

2.2. Preparation of Modified Asphalt Binder

The modified asphalt binder was created by blending different POMS (0%, 1%, 2%, 3%, 4% and 5%) with an asphalt binder penetration grade of 60/70 using a Silverson mixer at a mixing temperature of 160°C, 30 minutes mixing time and 800 rpm [8]. Initially, the asphalt binder was heated until it reached 160°C then the POMS were added gradually. The modified asphalt binder was allowed to mix thoroughly for 30 minutes until a homogenous blend was achieved.

The short-term aging of POMS-modified asphalt binder in this study was simulated by rolling thin-film oven (RTFO). The oxidative aging time chosen for the rolling thin-film oven (RTFO) test is 75 minutes at a temperature of 163°C to bring the asphalt binder into a short-term-aging state [9]. An

open cylindrical glass bottle with a diameter of 64 mm and a height of 140 mm was filled with 35 grams of asphalt samples. The samples are then promptly introduced into the RTFO, which has been preheated to 163°C and then rotated at 15 revolutions per minute. At the same time, 4000 mL/minute of hot air was continuously blasted in [10].

2.3. Binder testing

Further testing on its properties was conducted after all six different proportions of POMS-modified asphalt binder were made. The physical properties of unmodified and modified asphalt binder were tested by performing a penetration and softening point test. The penetration testing followed the American Society for Testing and Materials (ASTM) D5 procedure [11]. Before testing, the asphalt binder sample was conditioned in a water bath for 1.5 hours. The penetration was carried out using a specific needle under standard temperature, time, and loading parameters of 25°C, 5 seconds, and 100 g, respectively. The penetration needle is positioned at the tip of the surface and the loading is released to observe the penetration value. Three determination at points was made on the surface of samples at least 10 mm from the container's side and at least 10 mm apart [11].

The ring and ball test is another name for the softening point test was carried out following ASTM D36 [12] guidelines. The asphalt binder was heated in the oven at 110°C for not more than 30 minutes until it became suitably pourable. The asphalt binder then was poured into the rings and left to cool for at least 30 minutes. The top of the specimen was cut off after cooling, and it was then set on a flat smooth brass plate with ball-centering guides and a thermometer in place. After all were set in place, the beaker was filled with distilled water to a depth of not less than 102 mm and not more than 108 mm and held at 5°C for 15 minutes. The heat was applied until the temperature was gradually increased by 5°C per minute until the ball could penetrate through the asphalt and sag downward at a distance of 25 mm at which the temperature point was recorded as the softening point.

Fourier Transform Infrared Spectroscopy (FTIR) was used to determine the chemical functional and structural changes in a medium [13]. In this study, FTIR-ATR (Attenuated Total Reflectance) was used to conduct the chemical investigation of unmodified and POMS modified binder under unaged and short-term aged. The Fourier Infrared Spectrometer was preheated for at least 30 minutes in advance before starting the testing. A background scan was performed before each measurement was conducted. The acquisition parameters were set to 32 scans, a resolution of 4 cm⁻¹ and wavenumber range or reflective mode of test between 600 and 4000 cm⁻¹ [14].

3. Results and discussion

3.1 Penetration and Softening Point

Figure 2 presents the relationship between penetration and softening point test for POMS-modified asphalt binder. This test was conducted to get an overview physically about the physical changes in asphalt binder by the addition of POMS. It can be seen that, with the increasing amount of POMS, the penetration value increased and the softening point decreased. Penetration reflects the consistency of the asphalt binder. The increasing penetration value indicated the softening effects of the addition POMS. Penetration increased by 2.45% for 1%, 8.88% for 2%, 12.87% for 3%, 15.06% for 4% and 22.89% for 5% POMS compared to base binder 0% POMS. Then, the softening point slightly decreased for 3.6% for 1%, 3.96% for 2%, 5.05% for 3%, 6.49% for 4% and 7.03% for 5% POMS. This result showed that POMS caused the dilution effect like bio-oil [10,15,16]. Xin et al., [16] suggest that bio-oil needs to be selected carefully to ensure the asphalt binder achieves the appropriate characteristic. The soft asphalt binder can enhance the workability of asphalt mixture [17] but needs to be limited to reduce the rutting susceptibility [10].

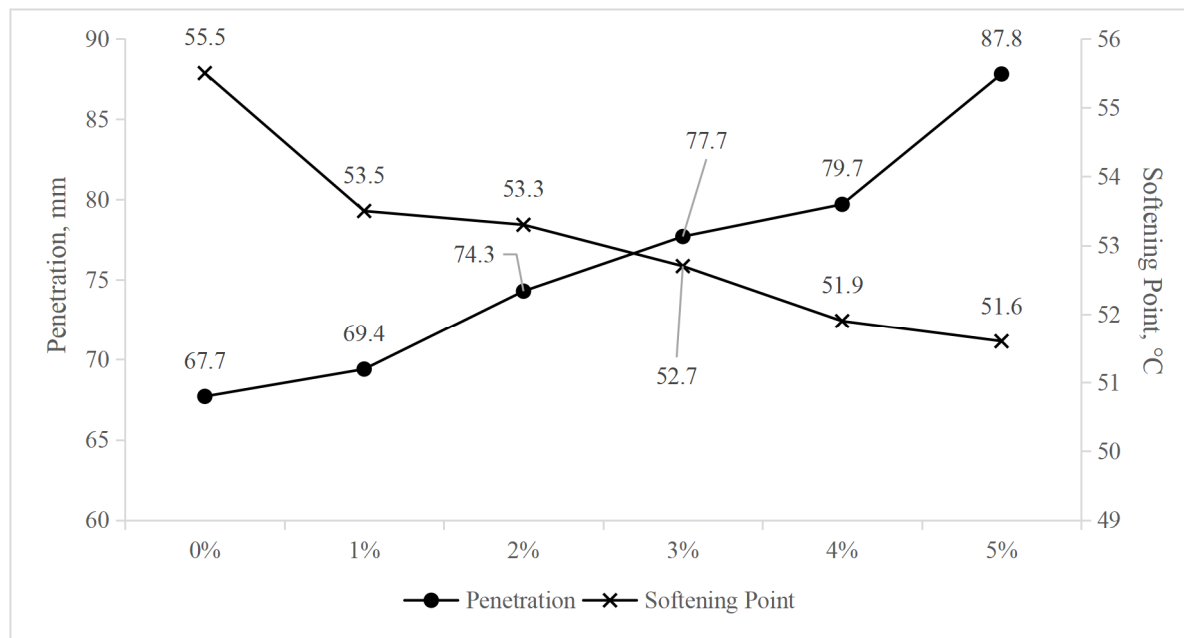


Figure 2. Penetration and softening point value of POMS-modified asphalt binder.

3.2 Chemical Properties

The structural alterations and chemical functions of carbonyl (C=O) and sulfoxide (S=O) groups are studied using infrared vibration modes [18]. Figure 3 and figure 4 shows the FTIR absorbance spectra for carbonyl and sulfoxide group for unmodified and POMS-modified asphalt binder under unaged and short-term age respectively. The graph presented for wavelength 600 to 2000 cm^{-1} only. The chemical bonding for the POMS-modified asphalt binder in varied percentages under simulation of unaged and STA has no significant variation, according to the findings. Most of the chemical bonds that were present at the peak in the original asphalt binder are also present in the modified asphalt binder, although their absorbance is slightly altered. The chemical bond group at peak in the original and modified asphalt binder under unaged and STA conditions are hydrocarbon CH (stretching vibration) 2851 – 2921 cm^{-1} , carbonyl group C=O (stretching vibrations) 1598 – 1600 cm^{-1} , Alkene C=C (stretching vibrations) 1598 – 1600 cm^{-1} , hydrocarbon (bending vibration) 1455 – 1458 cm^{-1} , Ether C-O-C, alcohols C-OH (stretching vibration) 1032 – 1057 cm^{-1} . At wavenumbers of 1030 cm^{-1} and 1700 cm^{-1} , the absorbance spectra of unaged and STA-treated asphalt binder. In comparison to the carbonyl group, both unaged and STA-treated asphalt binder gained a high amount in the sulfoxide group, as seen in the spectra. This is attributed to the elevated sulfur content present in the asphalt binder [5].

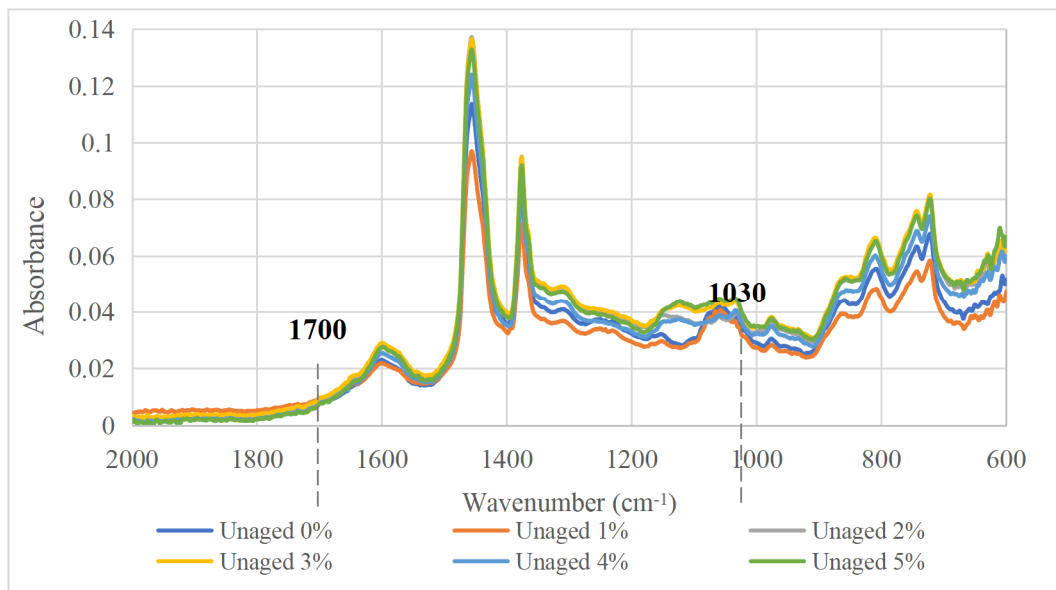


Figure 3. FTIR absorbance spectra for carbonyl and sulfoxide group for unmodified and POMS-modified asphalt binder (unaged)

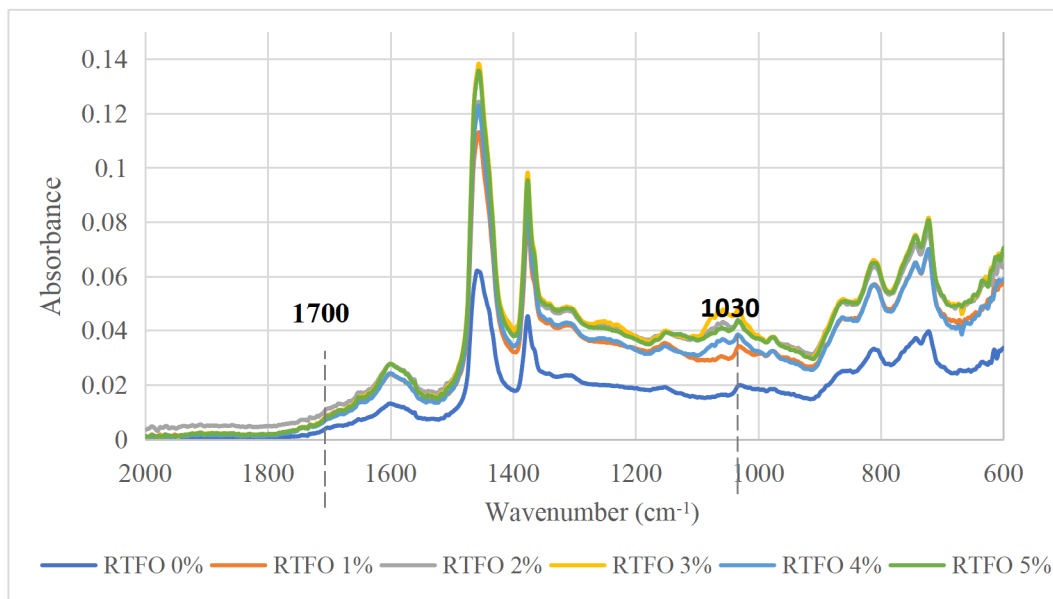


Figure 4. FTIR absorbance spectra for carbonyl and sulfoxide group for unmodified and POMS-modified asphalt binder (STA)

The presence of C=O absorption at 1700 cm^{-1} indicates the presence of the carbonyl group, while the presence of S=O absorption at 1030 cm^{-1} indicates the presence of the sulfoxide group. This region is investigated to determine the carbonyl and sulfoxide groups as indicators of asphalt aging performance before and after the aging process. The carbonyl function group is used to determine the degree of oxidation during the aging of a binder [18]. A clear individual graph is illustrated in figure 5 and figure 6. The absorbance amount of carbonyl and sulfoxide group in unmodified and various percentages of POMS-modified asphalt binder were compared between unaged and STA conditions. The detection of the carbonyl group in un-aged asphalt binder is consistently decreasing except at 3% POMS, whereas in short-term aged asphalt binder, it is spotted to be increasing until 2% POMS, decreasing through the additional percent of POMS, and increasing back at 5% addition of POMS. According to figure 5, the sulfoxide group was observed to have a steady absorbance value throughout

the addition of POMS, except for 4%, which increased somewhat for un-aged. Except for the POMS at 4%, the sulfoxide group in STA is growing. Figure 6 shows that the carbonyl group in the un-aged condition decreases as POMS is added, whereas the carbonyl group in the STA condition increases until POMS is added at 2% and then decreases. The addition of POMS to asphalt binder has been found to have an impact on chemical structures.

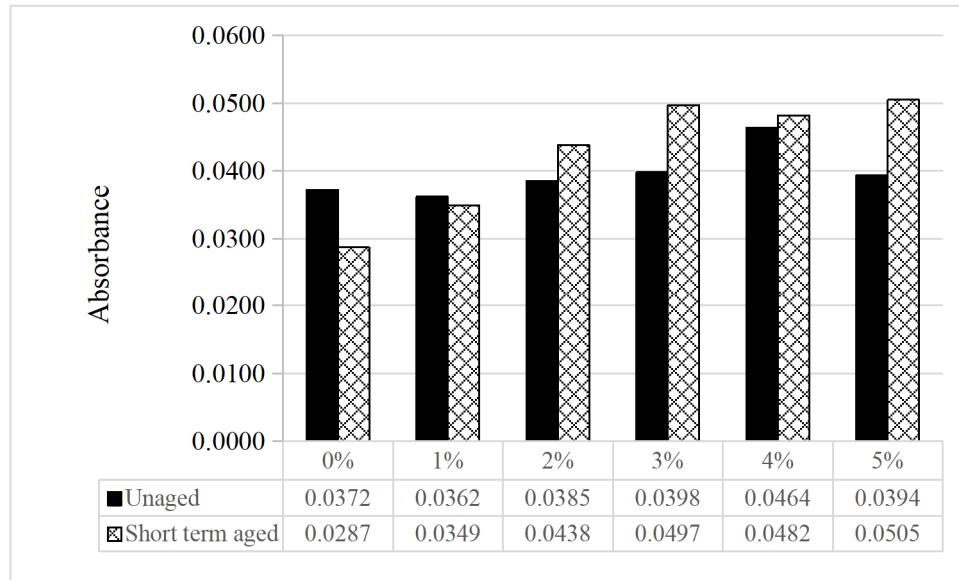


Figure 5. Comparison of sulfoxide absorbance for unaged and STA POMS-modified asphalt binder.

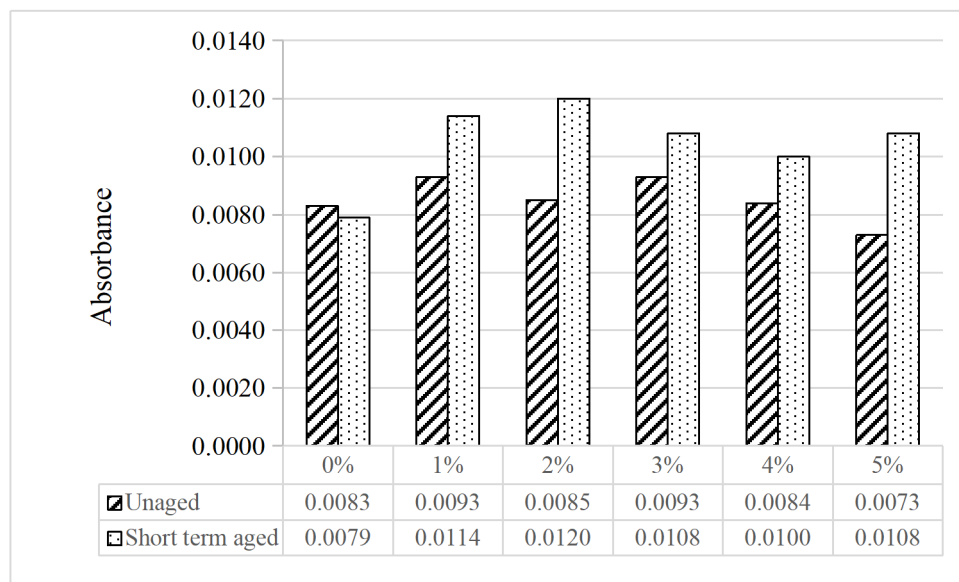


Figure 6. Comparison of carbonyl absorbance for unaged and STA POMS-modified asphalt binder.

Carbonyl absorbance increases as the aging process progresses, as shown in both graphs. This indicates that the POMS-modified asphalt binder undergoes an oxidation reaction during the manufacturing process. The oxidation process is detected in this study after the simulation under STA conditions utilizing the RTFO method. Because of the decreased carbonyl absorbance, adding a fraction of POMS additive to the asphalt binder modifies its chemical composition, reducing the aging

process of the changed asphalt binder. It was discovered that adding the POMS additive to the asphalt binder changed the absorbance spectra and softened the asphalt binder.

4.0 Conclusion

In conclusion, the addition of POMS caused the penetration value and softening point of POMS-modified asphalt binder changes. The penetration value increased progressively with the addition of POMS and the softening point decreased as the amounts of POMS in the asphalt binder increased. The modified asphalt binder softened with the addition of the POMS ingredient and had a high-temperature susceptibility, indicating that it is not suited for use in high-temperature conditions. The effect of adding POMS on the chemical characteristics of modified asphalt binder was examined. The addition of POMS to modified asphalt binder slows the aging process.

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References

- [1] Shahbandeh M 2023 *Production Volume of Palm Oil Worldwide from 2012/13 to 2022/23* Retrieved on August 27, 2023 from <https://www.statista.com/statistics/613471/palm-oil-production-volume-worldwide/>
- [2] Mosunmola A G and Olatunde, S. K. 2020 Palm oil mill effluent (POME) and its pollution potentials: A biodegradable prevalence, *Pollut. Eff. Control.* **8** 1 - 5
- [3] Osman N A, Ujang F A, Roslan A, Ibrahim M F and Hassan M A 2020 The effect of palm oil mill effluent final discharge on the characteristics of *Pennisetum purpureum*. *Sci Rep.* **10** 1-10
- [4] Camargo I G D N, Hofko B, Mirwald J. and Grothe H 2020 Effect of thermal and oxidative aging on asphalt binders rheology and chemical composition, *Materials*, **13** 1–21
- [5] Hofko B, Porot L, Cannone A F, Paolikakos L, Huber L, Lu X, Mollehauer K and Grothe H 2018 FTIR spectral analysis of bituminous binders: reproducibility and impact of ageing temperature, *Mater. Struct* **51** 1-16
- [6] Mahssin Z Y, Abdul Hassan N, Yaacob H, Puteh M H, Ismail C R, Putra Jaya R, Mohammad Zainol M and Mahmud M Z H 2021 Converting Biomass into Bio-Asphalt - A Review. *IOP Conf Series: Earth and Environmental Science* **682** 012066
- [7] Hainin M R, Jaya R P, Ali Akbar N A, Jayanti D S and Yusoff N I M 2014 Influence of palm oil fuel ash as a modifier on bitumen to improve aging resistance, *J. Eng. Res.*, **2** 34–46
- [8] Xin T W L, Han M T, Tai G, Hasan M R M, Kassim M A, Sukiran M A. 2022 Characterizations of Bio-Asphalt Incorporating Palm Bio-Oil From Waste Empty Fruit Bunches. *J Oil Palm Res.* **35** 45-57
- [9] Hofko B, Alavi M Z, Grothe H, Jones D and Harvey J 2017 Repeatability and sensitivity of FTIR ATR spectral analysis methods for bituminous binders, *Mater. Struct. Constr.* **50** 1–15
- [10] Yan Y Y, Yang M, Ran X, Zhou L. Zou, and M. Guo, 2020 Application of infrared spectroscopy in prediction of asphalt aging time history and fatigue life, *Coatings*, **10** 1–18
- [11] ASTM D5 2013 *Standard test method for penetration of bituminous materials* (West Conshohocken, PA United State: ASTM International) pp 1-4
- [12] ASTM D36 2014 *Standard test method for softening point of bitumen (ring-and-ball apparatus)* (West Conshohocken, PA United State: ASTM International)
- [13] Bowers B F, Huang B, Shu X and Miller B C 2014 Investigation of Reclaimed Asphalt Pavement blending efficiency through GPC and FTIR, *Constr. Build. Mater.*, **50** 517–523
- [14] Rasman M, Hassan N A, Hainin M R, Putra Jaya R, Haryati Y, Shukry N A M, Abdullah M E and Kamaruddin N H M 2018 Engineering properties of bitumen modified with bio-oil. *MATEC Web of Conferences* **250** 02003

- [15] Alattieh S A, Al-Khateeb G G, Zeiada W and Shanableh A (2020). Performance assessment of bio-modified asphalt binder using extracted bio oil from date seeds waste. *Int. J Syst. Assur. Eng Man* **11** 1260–1270
- [16] Xin T W L, Han M T, Tai G, Hasan M R M, Kassim M A and Sukiran M A 2022 Characterisations of Bio-Asphalt Incorporating Palm Bio-Oil From Waste Empty Fruit Bunches. *J of Oil Palm Res* **35** 45–57
- [17] Yu, J., Guo, Y., Peng, L., Guo, F., & Yu, H. (2020). Rejuvenating effect of soft bitumen, liquid surfactant, and bio-rejuvenator on artificial aged asphalt. *Constr. Build. Mater.* **254** 1-11
- 118] Hamzah M O and Shahadan Z 2011 Effects of Aging on the Physical, Rheological and Chemical Properties of Virgin Bitumen Incorporating Recovered Reclaimed Asphalt Pavement Binder, *Aust. J. Basic Appl. Sci* **5** 1323–1331