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## Adhesion Characterization of Palm Oil Mill Sludge Modified Asphalt Binder

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Abstract: The adhesion of mineral aggregates to asphalt binder is an essential point that attributes to the quality and performance of the asphalt mixture. Moreover, the considerable problem to the asphalt pavement could come from the lack of bonding between the materials. Generally, adhesion of mineral aggregates depends on the source or type of aggregate and asphalt binder. This study investigated the adherence coverage of palm oil mill sludge (POMS) modified binder with granite aggregate. Base asphalt binder of penetration grade 60/70 was blended with 1%,2%,3%,4% and 5% of POMS to produce the modified asphalt binders, respectively. Consequently, POMS modified binders were mixed with the granite aggregate to produce loose mix samples. In this current study, boiling test procedure was used to reduce the adhesive bonding of the loose mix sample. Then, Image J software was used to evaluate the stripping area of the samples. The results from image analysis revealed that different stripping areas due to different amounts of POMS incorporated in the modified asphalt binder. Up to 4% POMS modified asphalt showed an acceptable resistance toward moisture compared to un-modified asphalt. Image J helped to produce a clear stripping result compared to visual observation. In conclusion, POMS could be used as an asphalt modifier with satisfactory adhesion properties.

Keywords: Adhesion of asphalt binder, modified asphalt binder, palm oil waste

### 1. Introduction

The physical or chemical properties of the asphalt binders and aggregates, which are considered the two critical components of asphalt mixtures, directly influence the performance of pavement. In pavement mixtures, the asphalt binds the aggregates particles together and transfers the traffic loading stresses during its service life. Therefore, good adhesion or bonding between asphalt binder and aggregate is particularly important to ensure the durability of a pavement. However, this adhesion can be reduced if the moisture infiltrates the layer [1]. In general, adhesion between aggregates and asphalt binder depends on the chemical nature or the source of the asphalt binder and the physical properties of aggregate [1]. The appropriate modifiers are essential to ensure the strong adhesion (bonding) of asphalt binder to the aggregate surface [2]. Natural resources are rapidly depleted in recent years due to the massive amounts of total materials

consumed worldwide. The limited resources of crude oil, increasing cost of materials and environmental concerns have urged the researchers to find alternative materials in asphalt modification. There are various modifiers have been used in pavement materials such as engineered products, industrial by-products, naturally occurring materials and waste materials [3]. The increasing demand for new manufacturing materials, construction businesses and agriculture sector increased trash production, resulting in a considerable increase in the volume of waste in landfills.

In 2020, Malaysia supplied about 34.3% of the global palm oil exports and 25.8% of the world's palm oil production [4]. The manufacture of palm oil produces 10% of oil from fresh palm and kernels, leaving 90% as waste. Palm oil processing generates various waste materials and by-products, including empty fruit bunches, fibres, kernel shells and palm oil mill sludge (POMS) [5]. POMS was reported as the second highest waste from palm oil processing industry [5]. Palm oil sludge is normally disposed in palm plantations, which can bring to environmental pollution [6]. POMS consists of suspended solid and residual oil after the palm oil mill effluent discharge. Therefore, researchers are searching for alternatives to lessen the harm of the waste caused to the environment by employing this agricultural waste as modifier in construction such as pavement materials.

Based on the previous study conducted by Borhan et al. [7], the mixture contained palm oil fly ash (POFA) displayed more resistance to permanent deformation, when compared to control samples. Palm kernel shell, whereby at 10% can be applied as replacement for coarse aggregate for medium traffic design [8]. In asphalt binder modification, Raman et al. [9] and Poh et al. [10], bio-oil produced from pyrolysis process of palm oil empty fruit bunch softens the asphalt binder with comparable rutting resistance to un-modified binder. Bio-oil is a complex mixture of various organic compounds generated from biomass materials [10]. Raman et al. [9] reported that 2% to 8% of bio-oil from EFB was able to produce a range of penetration values, similar to the control sample, resulting in a penetration grade of 80/100. Poh et al. [10] observed that 5% of bio-oil derived from EFB displayed a slight penetration value, which still ranged in the penetration grade of 80/100. In a recent study, Uchoa et al. [11] studied the influence of palm vegetable oil which is derived from hydrogenated palm oil fat (HPF). It was found that the HPF reduces the stiffness by reducing the complex modulus, G\* and greater elasticity,  $\delta$ .

Most of the other studies using bio-oil focused on the physical and rheological investigation of asphalt binder. However, many other aspects need to be evaluated in order to ensure the quality of the performance of pavement mixture by using waste materials. Numerous research efforts have been conducted to understand the conception of the adhesion and to find the most appropriate test method to determine the affinity between asphalt binder and aggregate [12]. Thus, appropriate test methods for characterizing the asphalt binder-aggregate adhesion properties to mitigate stripping are imperative. The common method to determine the adhesion properties is boiling water test. This testing is simple, easy to perform and offers good potential in detecting the moisture susceptibility mixture [1]. However, it is often difficult to differentiate the level of stripping especially for the area of stripping less than 30% [13]. Image analysis was suggested as it provides more objective and reliable tests [14]. The objectives of this study are to investigate the loss of adhesion of POMS modified binder and to evaluate the stripping area by using Image J software.

#### 2. Materials and Methods

#### 2.1 Asphalt Binder

In this study, asphalt binder with penetration grade 60/70 was used as a base binder. The selection of this asphalt binder was based on the current practice of road construction in Malaysia. Table 1 shows the basic properties of this asphalt binder.

Table 1 - r hysical properties of r EN 00/70		
Parameter	Value	
Specific gravity	1.03	
Penetration at 25°C (dmm)	66.86	
Softening point (°C)	53.25	
Viscosity at 135°C (Pa.s)	0.64	

 Table 1 - Physical properties of PEN 60/70

#### **2.2 POMS**

The asphalt binder was modified by adding different percentages of POMS (1%, 2%, 3%, 4% and 5%). These percentages were selected based on the previous study [9,10]. Palm oil mill sludge (POMS) was obtained from a palm oil factory at Johor Bahru, Malaysia. Fig. 1 shows the POMS which is exhibited thick, dark brownish color, high solid and oil content. The density of POMS is measured by using pycnometer, was recorded as 1.26 g/cm<sup>3</sup>.



Fig. 1 - Palm oil mill sludge

#### 2.3 Aggregate

The current study employed crushed granite as the aggregates. The aggregates were sieved to obtain different aggregate sizes based on the mix design of AC 14. However, only coarse aggregate mixture retained at 2.36 mm sieve and above was used to produce the loose mixture [15]. About 1100 g of clean oven-dried aggregates were fully coated with 61.6 g of asphalt with 0%, 1%, 2%, 3%, 4% and 5% of POMS, respectively. This weight of asphalt binder was based on optimum asphalt binder content that has been conducted in other studies [16].

#### 2.4 Preparation of Sample

In the preparation of the modified binder, 300g of asphalt binder was heated at 110°C until it was melted. Then, POMS was added gradually and blended using high shear mixer at 160°C, 800 rpm, for 30 minutes. The temperature of 160° is consider as a suitable high temperature to avoid excessive aging of asphalt binder [17]. The POMS modified asphalt binder was mixed with coarse aggregate by using heavy-duty mixer at mixing temperature of 170°C.

#### 2.5 Boiling Water Test

The boiling water test was performed in accordance with ASTM D3625 [18]. A total of six types of mixtures were used in this study. Approximately 500 ml of water was filled in a 1000 ml beaker and heated to a boiling water point of 100°C. Then, about 250 g of loose hot mixture was poured into the beaker with boiling water and left for 10 minutes. After 10 minutes, the mixture was filtered, placed on dry paper, and left at room temperature for 30 minutes to dry. The before and after images of the samples were captured with a smartphone with a resolution of 8000  $\times$  6000 pixels.

#### 2.6 Image Analysis

Image J, an open-source image processing application, was used to further analyze the sample picture. The picture was taken with indirect light to avoid excessive sparkle but sufficient to produce a clear image [14]. The evaluated photo was subjected to a method in which the pixels were identified based on their HSB value in order to determine the degree area of asphalt stripping. The H component refers to Hue of the colour, the S refer to saturation and B refers to brightness. The ability to classify the pixel in the function of their colour was possible through a specific plugin inside Image J. This plugin allows interaction with individual components of several colour spaces and emphasises one type of colour over others. The HSB colour space was chosen for classification because of its superior ability to categorise pixel areas and ease of use.

Fig. 2 shows the steps to analyze the picture using Image J. The process starts with the binary image generated. After loading the original image into Image J, the first step is to reproduce the image in a specific size that fits the picture as shown in Fig. 2(a). Then, the scale was set to millimetre, and the scaling size is shown on the image as a reference once the scale was established. The next step is to use image-adjust-color-threshold to assess the region of mixture on the image, which brings up to HSB colour space settings. By selecting original-filtered, the mixture's covered area was displayed in white, as displayed in Fig. 2(b). In such conditions, for setting component B brightness value has been set from range the 123 to 130. For caution, during capturing picture process before and after, the background colour was set to green to avoid colour illumination as green colour can be adjusted with any variant type of colour. Once the area cover was determined, after the filtered setting is selected, a yellow line surrounded the covered area as a sign of the area to be measured. The total area was provided by clicking the analyze-measure. Lastly, for measuring the area of stripping, component S saturation was adjusted at value of 45/45 and the area of stripping appeared on the screen as shown in Fig.

2(c). This process was repeated on another sample. According to Amelian et al. [14], suitable threshold value is important to distinguish the pixels in the image. As a result, it is vital to utilise the same saturation and brightness threshold values within all samples for a consistent comparison [14]. As was mentioned earlier, after applying the specific range of threshold value, the number of black and white pixels was counted and finally the amount of sample surface and stripping area of the mixture was determined. Finally, the adhesion coverage was estimated using the equation (1), which was referred to as final asphalt binder coverage after the boiling test [19]:

Adhesion coverage = 
$$\frac{\text{Area of asphalt binder}}{\text{Area of asphalt binder} + \text{Aggregate}} \times 100$$
(1)

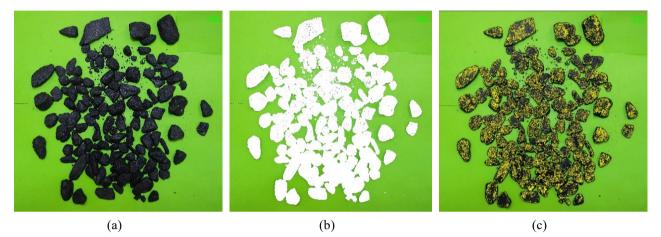


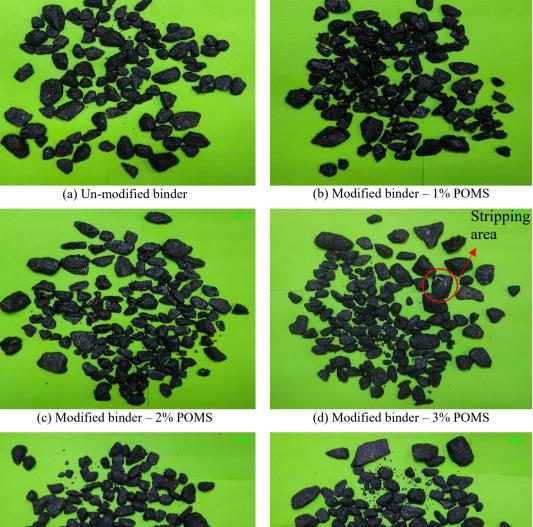
Fig. 2 - Image J analysis step consists of (a) Binary image (After capture); (b) Determination of sample area; (c) Determination of stripping area

#### 3. Results and Discussion

Boiling water test is very simple to perform and has potential to evaluate the anti-stripping additives [20]. In this study, POMS modified asphalt mixtures were tested in order to observe the loss of adhesion due to the action of boiling water and compared to un-modified asphalt mixture. As described in the methods section, the same setting of threshold, saturation, pixels and brightness was applied during image analysis. Fig. 3 shows the picture of loose samples after the boiling test. It can be observed that the stripped area is scattered, and it is difficult to quantify the actual area. Additionally, the visual observation of the stripped area is mainly depending on the judgement of the operator [14]. In a study conducted by Lie et al. [12], the coating area above 95% for static immersion test was used as an indicator for bonding or adhesion of asphalt binder with aggregate. However, this study uses boiling water test which is faster to conduct and able to show the small changes in the adhesion because of water [12]. In order to obtain better results for stripped area, the captured pictures were further analyzed by using Image J software.

Table 2 shows the total area mixture and total area of stripping of the asphalt mixture obtained from the analysis of Image J software. As shown in Table 1, the area of stripping is depended on the total area. It should be mentioned that the weight of samples has been standardized in order to ensure the consistency of comparison. It can be seen that the total area of each sample displayed the variation value. 1% POMS and 5% POMS exhibited the lowest and highest value of stripping area with 261.182 mm<sup>2</sup> and 376.967 mm<sup>2</sup>, respectively.

In order to produce a meaningful analysis since the total area is different, the calculation of adhesion coverage was applied by using Equation 1. Fig. 4 shows the percentage of adhesion coverage. The adhesion coverage value was found more than 95%. It also noted that the adhesion properties of POMS modified binder do not show much difference compared to un-modified mixture. The adhesion coverage for 1%, 2% and 4% of POMS modified samples are slightly higher compared to un-modified mixture. The modified asphalt mixture with 1% POMS has the highest amount of adhesion coverage with a total percentage of 99.02%. The higher value of adhesion coverage indicated better stripping resistance. Meanwhile, the modified samples exhibit a lower value compared to other samples. In view of the results obtained, the modified asphalt binder with POMS shows the acceptable resistance to stripping compared to un-modified binder with POMS shows the acceptable resistance to stripping compared to un-modified binder. This finding agrees with other findings reported by Poh et al. [10] which suggest that bio-oil from palm oil empty fruit bunch is able to be used as an asphalt modifier. As the POMS contained the high solid, oil and grease [21] and soften the asphalt binder, there is a high concern in terms of mixture performance. There are good adhesion properties found by the POMS modified binder with granite aggregate. This adhesion is important to ensure the durability of the mixture.





(e) Modified binder – 4% POMS



(f) Modified binder – 5% POMS

Fig. 3 - Loose samples after boiling test

Table 2 - The total area of sa	amples and total area	of stripping on asphalt mixture
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POMS content (%)	Total area (mm <sup>2</sup> )	Stripped area (mm <sup>2</sup> )
0	17616.331	261.182
1	23496.521	231.413
2	23873.910	326.387
3	18486.650	348.613
4	18786.446	266.165
5	20154.895	376.967

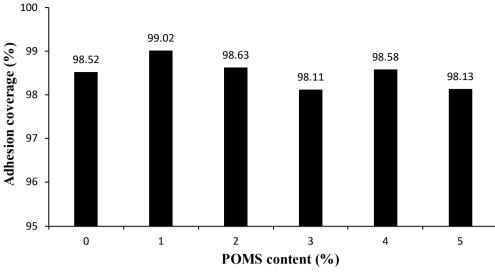


Fig. 4 - Adhesion coverage of un-modified and POMS modified sample

#### 4. Conclusion

In this study, image analysis was applied to study the adhesion properties of POMS modified asphalt mixture. In order to induce the adhesion effect, a boiling water test was performed on the loose mixtures. After the test, the stripping area by visual observation is difficult to differentiate since the area is small. Then, the image analysis was used to distinguish the stripping and coated area. Image J software has provided a quantitative analysis of the stripping area. Based on the analysis, the modified asphalt binder with up to 4% (POMS) shows adequate resistance to stripping compared to the un-modified asphalt binder. It is therefore the addition of POMS does not jeopardize in terms of adhesion with the aggregate at certain percentage.

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