Effect of different sizes of palm oil fuel ash (POFA) towards physical properties of modified bitumen

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Abstract. In the past decades, numerous numbers of studies have been carried out to find ways enhancing properties of bitumen. Other than using polymer, agricultural waste such as palm oil fuel ash (POFA) is one of the waste products that can be used to modify bitumen. In this study, the physical and rheological properties of POFA modified bitumen were examined based on different grinding hour and different percentage of POFA. The bitumen were mixed with different percentages of POFA (0, 5 and 7%) which passed through 0.075 mm sieve and grinded at different period (1 and 4 hour). The samples were then tested and compared to conventional bitumen. From TEM results, POFA grinded at 1 hour have sizes between 3-7 µm while POFA grinded for 4 hours have finer sizes between 500 nm to 3 µm. The results showed that fineness of POFA affect properties of bitumen significantly. Decreasing in penetration value and decreasing in softening temperature indicates that the modified bitumen becomes harder than conventional bitumen. Modified bitumen gives best results when added with 7% POFA sizes of 500 nm to 3 µm compared to 3 to 7 µm.

1. Introduction
Malaysia is one of the largest producers and exporters of palm oil around the world. Production of palm oil itself produces tonnes of waste product abandoned to cause environmental pollution. Therefore, this issue getting more attention among researchers whom are working on many studies over past decades to explore the potential of wastes as a possible construction material [1-5]. In road construction, binder properties play one of the crucial roles to ensure the road pavement performing well and have longer life span. Other than that, most of the road in Malaysia are constructed using flexible pavement as it is durable and would provide a smooth riding experience to the user. However, increasing in traffic load, temperature and volumes causing distress to the road and improvement is required in order to overcome road deformation. Thus, modification of conventional bitumen is the best practice to improve bitumen adhesive and cohesive properties.

Modification of bitumen is no longer alien towards pavement researchers. Modification can be done by adding polymer such as SBR, latexes, and EVA or waste material such as palm oil fuel ash (POFA) [6-12]. Usually, addition of polymer such as styrene-butadiene-styrene (SBS) could help overcome common pavement defects such as rutting and aging but the price of the modified bitumen could hike between 60-100% [13, 14]. In road construction industries, material cost will always be the
main concern and limitation to the contractors. Hence, utilizing industrial waste product will help minimize modifier’s cost and in extra promotes sustainability.

Modification of conventional bitumen using waste material previously shows that it improves physical properties such as penetration, softening point and also viscosity significantly [8, 15-17]. In 2013, Rusbintardjo et al. [8] studies the fundamental and rheological properties of oil palm fruit ash modified bitumen. POFA is a waste from oil palm that are burned to generate electricity at temperature of 700-1000°C in palm oil mill [18, 19]. He found that POFA is feasible to be used as modifier and that POFA modified bitumen met the requirements for bitumen grade 60/70 PEN. He was also suggested the appropriate amount of POFA to be added into bitumen were within 5-7% by weight of bitumen. Later in 2014, Hainin et al. [16] investigated the influence of POFA at different percentage with different aging condition. The results suggested that addition of POFA improves the resistance of the binder by decreasing the penetration value and increasing softening point temperature for both unaged and aged samples. They were also found out that POFA modified bitumen have higher resistant to permanent deformation at high temperature compared to conventional bitumen.

In term of fineness, it is believed that fineness of the material affects the binder properties. Abdullah et al. [20] use coconut shell (CS) as a modifier at different fineness (< 75 μm, 75–150 μm, and 150–300 μm). They replaced CS at 10%, 15% and 20% by weight of bitumen. As the results, bitumen with CS size less than 75 μm shows better results in all physical properties testing. A noticeable increment of softening point temperature and penetration were also found by Magar [21]. According to his study, modifying bitumen with finer crumb rubber size has significantly increased the physical properties of bitumen. Thus, results improved significantly with decreasing of the particles size.

2. Material and methodology

2.1 Palm oil fuel ash

Originally, POFA were actually has a coarser particle size. To obtain a uniform size, POFA was sieved by using sieve with opening of 0.075 mm and passed through it. POFA was grounded using a grinding ball mill with different grinding hours of 1 and 4 hours. The average size of POFA obtained were examined using transmission electron microscopy (TEM). After that, POFA were added into bitumen with different percentages by weight of bitumen and by different POFA sizes labelled according to grinding size. The samples were labelled as 5%-1hr, 5%-4hr, 7%-1hr and 7%-4hr.

2.2 Sample preparation

The physical properties of control bitumen were carried out to confirm that the binder met the standard specification including viscosity test [22]. Before mixing, 400 g of 80/100 PEN bitumen were prepared in a steel container and heated in the oven at approximately 130°C to allow it turns into liquid state. POFA were added and mixed with bitumen by using high shear mixer at speed of 800 rpm, at temperature of 160°C for 60 minutes. Prepared samples were then tested on its physical properties such as penetration test [23] and softening point [24] test as well as storage stability test. Plus, the penetration index was also calculated based on the results obtained from softening point tests and penetration tests.

| Test Properties                  | Standard Specification | Test Results |
|----------------------------------|------------------------|--------------|
| Penetration at 25°C (d-mm)       | ASTM D5                | 82           |
| Softening point (°C )            | ASTM D36               | 40           |
| Viscosity at 135°C (Pa · s)      | ASTM D4402             | 0.3          |
2.3 Storage stability test
The storage stability for control and modified bitumen were tested to determine the stability and homogeneity of the sample. A 50 g of heated sample was first poured into an aluminium can and stored vertically in an oven for 48 hours at 163 ± 5°C. After that, the cans containing sample were taken out and placed in refrigerator at -6.7 ± 5°C for at least 4 hours to solidify the sample. The solid samples were cut into three equal parts and the value was then determined by the difference of softening point temperature between bottom and top section of the sample.

3. Results and discussion

3.1 Transmission Electron Microscopy (TEM)
Transmission Electron Microscopy is used to obtained a quantitative measures of particles such as size distribution, average particle size and morphological characteristic of POFA. Figure 1 shows the TEM images of POFA after grinding for 1 and 4 hours under same magnification size of 2000 and the scale is 5µm. From the images, POFA grounded for 1 hour has approximate size between 3-7 µm and POFA grounded for 4 hours has approximate size between 500 nm to 3 µm. It can be seen that POFA have edgy particle shape. By grinding POFA for more hours, POFA was crushed into smaller size than original, reduced in porosity and has more irregular shape.

![TEM images of POFA after grinding](image)

(a) POFA after 1 hour grinding  (b) POFA after 4 hours grinding

**Figure 1.** TEM images of POFA after grinding.

3.2 Storage stability of POFA modified bitumen
The sample is considered as storage stable if the temperature’s difference is less than 2.2°C. Some POFA particles which dispersed might accumulated and settled at the bottom of the can after 3 days storage in the oven. Thus, the softening point temperature for bottom section was higher than top section. Table 2 shows the differences of softening point temperature for storage stability test of all samples. The difference between those top and bottom sections was not more than 2.2°C thus all samples was categorized as stable and both POFA and bitumen were compatible to each other.

| Sample       | Difference between top & bottom section (°C) | Condition     |
|--------------|----------------------------------------------|---------------|
| 5%-1 HR      | 0.5                                          | < 2.2 °C      |
| 5%-4 HR      | 1.0                                          | < 2.2 °C      |
| 7%-1 HR      | 1.5                                          | < 2.2 °C      |
| 7%-4 HR      | 1.5                                          | < 2.2 °C      |

Table 2. Differences of softening point temperature for storage stability test
3.2 Penetration test
Figure 2 shows penetration value for unaged and aged (RTFO) samples. As shown in Figure (penetration), the penetration value for both unaged and aged samples improved when POFA was added into bitumen. For unaged samples, penetration values decreases from 82 d-mm (control sample) to ranging between 62-68 d-mm.

The decreasing of penetration values are ranges between 60-70 d-mm where it were within range of bitumen grade 60/70 PEN. After underwent short-term aging, the bitumen hardened above 30 d-mm which indicates that POFA modified bitumen have high resistance to cracking. The best results goes to bitumen contains 7%-4hours of POFA. This condition shows that addition of POFA into bitumen has significantly affects the penetration value. However, inconsistent results occurs when fineness of POFA were increased. POFA modified bitumen shows better results when mixed with finer POFA at 7% than 5% by weight of bitumen.

3.3 Softening point
Figure 3 shows the softening point values for all samples. According to JKR specification [17], softening point for bitumen 80/100 PEN must be within 42-52°C. However, the softening point for control bitumen does not meet the specification where the temperature obtained was 40°C. Nevertheless, the softening points of POFA-modified bitumen were higher than control bitumen for both conditions. Generally, softening points were increased as the bitumen undergoes aging process results in hardening of the bitumen. For bitumen with 5% POFA, softening point was higher when bitumen was modified with POFA grounded for 1 hour compared to 4 hour. Contrary, bitumen contain 7% POFA grounded for 4 hour have higher temperature compared to 7%-1hr. Thus, more and finer POFA content gives higher results than the rest.
3.4 Penetration Index

The penetration index (PI) is normally used to measure the susceptibility of bitumen on temperature. Usually, bitumen with PI value ranges between -1 to +1 is favourable for pavement construction [25]. As shown in Table 3, low PI values were obtained which indicates higher temperature susceptibility. PI values for unaged samples 7%-1hr and 7%-4hr modified bitumen lies between acceptable values. Higher PI values indicate that bitumen has higher resistance to permanent deformation and low temperature cracking. Also as presented in Table 3, the lowest PI value were obtained from conventional bitumen followed by sample 5%-4hr, 5%-1hr, 7%-4hr and 7%-1hr at were -1.64, -1.51, -0.92 and -0.86 respectively. After aging, the value of PI decreases towards negative side marks that the bitumen becomes more brittle at low temperature compared to unaged samples.

Table 3. Penetration Index before and after aging

| Sample     | Condition | Unaged | RTFO |
|------------|-----------|--------|------|
| Control    |           | -3.06  | -3.02|
| 5%-1 HR    |           | -1.51  | -1.93|
| 5%-4 HR    |           | -1.64  | -2.35|
| 7%-1 HR    |           | -0.86  | -2.26|
| 7%-4HR     |           | -0.92  | -1.75|

4. Conclusion

Based on the results obtained, the following conclusion can be drawn:

a) Adding POFA in conventional bitumen with different percentage and different POFA sizes has significant effects to the penetration value and softening point temperature.

b) Introducing POFA in bitumen decreased the penetration value. Penetration value of POFA modified bitumen is within specification for 60/70 PEN bitumen.

c) Bitumen modified with 7% POFA with finer size (500 nm to 3 μm) exhibits better results in all test compared to control bitumen.

d) From PI values, increased in PI value shows that POFA modified bitumen are less susceptible to temperature.

Figure 3. Graph of softening point at different size of POFA and grinding hour for unaged and after RTFO.
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