Effect of grinding period on physical properties of modified bitumen using palm oil fuel ash (POFA)

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Abstract. Malaysia is having rapid economic development in recent years, and this has placed high performance demand on its highway infrastructure. The increased in number of traffic has led to severe pavement deterioration. Due to this, better and stronger pavement materials are needed in order to cater the increasing number of axle loading. Palm oil fuel ash (POFA) which comes from the ash of burning mesocarp of the palm oil fruitlets has been used as a modifier to improve the properties of bitumen. POFA is a biomass that had pozzolanic properties (siliceous material). By using POFA in bitumen, it will promote sustainability and lesser landfill to dump this waste. The objective of this research is to evaluate the effect of grinding period of POFA and different percentage of POFA towards physical properties of modified bitumen. A few sets of samples of POFA modified bitumen at different grinding time and POFA content have been prepared and tested with penetration test and softening point test. The microstructural characteristics of POFA were also investigated by using Field Emission Scanning Electron Microscope (FESEM). From the result, it shows that grinding time and POFA content does not influence the physical performance of the bitumen.

1.  Introduction

Deterioration of road structure happened more these days due to the increased of traffic volume and low efficiency on the road maintenance process. To increase on the long term durability of the pavement, the bituminous layers should be improved with regard to performance properties, such as resistance to permanent deformation, fatigue, and aging. Study on the application and usage of bitumen modifier using waste product in order to increase the quality of the bitumen has become current focus in bitumen modification [1-4].

The American Society for Testing and Materials (ASTM) defines bitumen as a class of black color (solid, semisolid or viscous) cementitious substance which occurs in nature or obtains in petroleum processing, composed principally of high molecular weight hydrocarbons of which bitumen. Bitumen has certain weaknesses whereby it can ‘flow’ at high temperature, leading to pavement rutting, and it can fracture at low temperature, leading to pavement cracking, the adhesion between bitumen and aggregate breaks down under a combination of ageing and water attack. The inherent weaknesses of
bitumen cause highway maintenance works quite difficult and expensive. Bitumen is shear-sensitive and hence making it prone to shove and rut at higher temperatures. On the other hand, at low temperatures it becomes brittle and tends to undergo fracture cracking and potholing. Cracking is generally reduced by making the asphalt binder either less viscous or less temperature susceptible. Reducing low temperature viscosity leads to soft bitumen and the mechanical properties of mixture deteriorate. According to McGennis [5], modified bitumen is produced for the purpose of altering and improving the properties of the bitumen to enhance the long term performance of pavements. While the modifier may affect many properties, the majority of modifiers are used in an attempt to reduce temperature dependency, oxidative hardening of bitumen, and the moisture susceptibility of asphalt mixtures [5, 6].

Malaysia is the world’s leading producer and exporter of palm oil and palm oil products with a reputation for quality and reliability. In a report published by the Department of Environmental, Ministry of Science, Technology and the Environment Malaysia [7], Malaysia Palm Oil production in the year 2009 was 17.56 million tons. From 1kg of oil palm fruitlets can result in 0.34kg of crude palm oil (CPO) and 0.1kg Kernel Oil or in total results in 0.44kg of oil. This means that 1kg of oil palm fruitlets results in 0.44kg of the mesocarp waste. If the product of palm oil in year 2009 was 17.56 million tons (44% of the oil palm fruitlets), it is means that there are 7.73 million tons of the waste of mesocarp in year 2009. Salihuddin [8] is the pioneer researcher to mark this as Palm Oil Fuel Ash or POFA. Palm Oil Fuel Ash (POFA) is a by-product produced after burning of both palm oil husk and palm oil shell as fuel in the boiler at palm oil mill. Generally after combustion about 5% POFA by weight of solid wastes is produced. In other words, the physical characteristics of POFA are very much influenced by the operating system in palm oil factory. In practice, POFA produced in Malaysia Palm Oil Mill is dumped as a waste without any profitable return [9].

Previous research had also proved that bitumen modification can soften binders and mixtures at high temperature to minimize rutting and reduce the detrimental effects of load induced moisture damages, improve fatigue resistance, particularly in environments where higher strains are imposed on the asphalt concrete mixture [10-12]. By using modification bitumen will also improve bitumen-aggregate bonding to reduce stripping, improve pavement durability with an accompanying net reduction in life cycle costs, and improve overall performance as viewed by the highway user. POFA content is most feasible to be used as bitumen modifier and based on the penetration test result of 5% OPFA-MB (Oil Palm Fuel Ash bitumen modifier) can be graded as binder penetration 60/70 PEN [13] because it is resistant to high temperature rutting and resistant to low temperature thermal cracking. The addition of POFA was also found to increase the Penetration Index (PI) of the bitumen even when subjected to aging [14]. POFA was also found to improve the performance of modified asphalt concrete in terms of elasticity modulus, stiffness, indirect tensile strength and creep resistance [15, 16].

2. Material and method
The purpose of this study was to observe the effect of adding palm oil fuel ash in bitumen and act as bitumen modifier by using different percentages of POFA. The testing procedures were based on The American Society for Testing and Material (ASTM) standard guide for laboratory works. POFA had been grinded by using Los Angeles Abrasion machine at different grinding hours (1, 2, 3, and 4 hours). POFA modified bitumen were prepared by mixing different percentage of POFA (at different grinding hour) into 80/100 penetration bitumen. Physical tests, namely penetration tests (ASTM D5) and softening point test (ASTM D36) had been conducted on each samples.

2.1 Sieve Analysis
Sieve analysis had been carried out in order to get the POFA that passing 75µm sieving size. These POFA were then grinded at different grinding hours (1, 2, 3, and 4 hours).

2.2 Modification process of bitumen
Four different percentages of POFA has been chosen based on previous findings by Rusbintardjo G. (2011), namely 0% (control), 5%, 6%, and 7% by the total weight of the bitumen content. POFA has been mixed with bitumen 80/100 penetration at a mixing temperature of 160°C, for about of 60 minutes and stirring speed of 800rpm. POFA was poured into 400g of bitumen at room temperature before mixing. For every 100g of bitumen, an equal amount of POFA was added then the mixtures of bitumen-POFA were heated in the oven just to make it as liquid later then place it on a hot plate under the drive shaft rotor propeller of the mixer at the speed of 800rpm. This procedure was conducted so that POFA was thoroughly mixed with bitumen.

2.3 Field Emission Scanning Electron Microscope (FESEM)
After grinding process of POFA, samples were examined using Field Emission Scanning Electron Microscope (FESEM) to check its morphological structure. FESEM which is available at University Lab Management Unit, Universiti Teknologi Malaysia are able to visualize a very small object and give topography information to its user.

3. Result and discussion

3.1 Morphological structure of POFA
The morphology of POFA with grinding hour of 0, 2 and 4 has been observed under FESEM and shown in Figure 1. Under the magnification of 1000, it can be seen that POFA is generally has irregular shape with porous and spongy texture for all samples. It was expected that, further grinding of POFA for 4 hours crushed the particle into smaller size, reduce porosity texture and it has more irregular shape compared to ungrounded POFA. The finding obtained is similar to previous study by other researchers [17, 18].

![Microstructural images of POFA](image1)

**Figure 1.** Microstructural images of POFA (a) Microstructure of POFA passing 75µm (0hour) (b) Microstructure of POFA after grinding for 2 hours (c) Microstructure of POFA after grinding for 4 hours
3.2 Penetration test

Figure 2 shows the results of penetration test for different percentages of POFA which are 5%, 6% and 7%. On each percentage, the effect of grinding time has been further looked. Generally from this Figure 2, it can be seen that by grinding the POFA from 1-4 hour, the penetration values has decreased slightly showing that the specimens has become harder. However for 5% and 6% of POFA, with the increased of grinding period from 1 to 3 hour the penetration has shown an increment from 7.24 to 8.20 and 7.01 to 8.37 respectively. When the grinding time increased to 4 hours, the penetration has reduced back to 7.21 and 7.43 respectively. In addition, analysis of variance (ANOVA) test has been run in order to determine the significant of effect of grinding time towards penetration value.

Table 1 shows ANOVA of Penetration Value by different percentage of POFA to grinding hour, the P value are higher than the P critical which is 0.05. The hypothesis on the different hour of grinding will give smaller penetration value was rejected because based on this ANOVA, it shows it has no significant difference between each of the grinding time.

| Variables       | P_{value} | P_{crit} | Condition | Significant | Remarks         |
|-----------------|-----------|----------|------------|-------------|-----------------|
| 0 hour of grinding | 0.78      | 0.05     | P > P_{crit} | No          | Reject the hypothesis |
| 1 hour of grinding | 0.40      | 0.05     | P > P_{crit} | No          | Reject the hypothesis |
| 2 hour of grinding | 0.76      | 0.05     | P > P_{crit} | No          | Reject the hypothesis |
| 3 hour of grinding | 0.74      | 0.05     | P > P_{crit} | No          | Reject the hypothesis |
| 4 hour of grinding | 0.40      | 0.05     | P > P_{crit} | No          | Reject the hypothesis |

Table 2. Graph of penetration value based on different percentage of POFA
Figure 3 shows the results of the penetration test for different grinding times which are 0 hour, 1 hour, 2 hours, 3 hours and 4 hours. At each grinding time, the effect of different percentage of POFA has been further looked and compared with control sample (without POFA). In general, the graphs show inconsistent result with some samples of POFA modified bitumen having higher penetration value compared to the control sample.

![Graphs showing penetration value for different grinding times](image)

**Figure 3.** Graph of penetration value based on different grinding time on POFA

Further analysis has been done by using POFA, where from Table 2 shows that ANOVA results of each percentages of POFA at certain grinding hour are not having the significant change with each other. The hypothesis is rejected because the samples with different percentages of POFA modified bitumen are not having significant effect with the controlled sample.
Table 2. ANOVA of Penetration Value by grinding period to different percentage of POFA

| Variables     | P_value | P_crit | Condition | Significant | Remarks           |
|---------------|---------|--------|-----------|-------------|-------------------|
| 5% of POFA    | 0.36    | 0.05   | P > P_crit| No          | Reject the hypothesis |
| 6% of POFA    | 0.68    | 0.05   | P > P_crit| No          | Reject the hypothesis |
| 7% of POFA    | 0.19    | 0.05   | P > P_crit| No          | Reject the hypothesis |

3.3 Softening Point Test

Figure 4 shows the results of softening point for different percentages of POFA which are 5%, 6% and 7%. For each percentage of POFA the effect of grinding time (0, 1, 2, 3 and 4 hours) towards softening point value has been further looked. At 5% POFA, it was recorded that softening point value increased from 39°C to 41.5°C with an increase of grinding time from 0 to 3 hours. When the grinding time increased to 4 hours, the softening point has reduced back to 40°C. However, for 6% of POFA, the temperature maintain at 40°C except at 3 hour grinding time, the value increased to 42°C and reduced back to 40°C at 4 hours grinding time. For 7% POFA, inconsistent value has been recorded. Further investigation has been done by conducting the ANOVA test analysis.

Based on Table 3 ANOVA of softening point value by different percentage of POFA to grinding hour it shows all of the grinding times are not have the significant change. The hypothesis on the different grinding time will give higher temperature on softening point test is rejected because it has no significance value at every temperature.

Figure 4. Graph of softening point value based on different percentage of POFA

Figure 3 shows the results of the penetration test for different grinding times which are 0 hour, 1 hour, 2 hours, 3 hours and 4 hours. At each grinding time, the effect of different percentage of POFA has been further looked and compared with control sample (without POFA). In general, the graphs show inconsistent result with some samples of POFA modified bitumen having higher penetration value compared to the control sample.
Figure 5 shows the results of the softening point test at different percentage of POFA. For 1, 3 and 4 hours grinding time, there is a slight increase of softening point values with an increase of POFA content, compared to control sample. However, similar to penetration test, inconsistent results were found in softening point values in certain cases. Therefore ANOVA test has been done in order to summarize the significant of POFA towards softening point value.

| Variables            | P_value | P_crit | Condition | Significant | Remarks               |
|----------------------|---------|--------|-----------|-------------|-----------------------|
| 0 hour of grinding   | 0.89    | 0.05   | P > P_crit| No          | Reject the hypothesis |
| 1 hour of grinding   | 0.62    | 0.05   | P > P_crit| No          | Reject the hypothesis |
| 2 hour of grinding   | 0.61    | 0.05   | P > P_crit| No          | Reject the hypothesis |
| 3 hour of grinding   | 0.61    | 0.05   | P > P_crit| No          | Reject the hypothesis |
| 4 hour of grinding   | 0.46    | 0.05   | P > P_crit| No          | Reject the hypothesis |

Figure 5. Graph of softening point value based on different grinding time of POFA
From Table 4 ANOVA of Softening Point Value by grinding period to different percentage of it can be seen that all of the percentages have P value that are bigger than the P critical. Therefore, the hypothesis was rejected because each of the percentages of POFA was not significance to controlled sample.

| Variables | P_value | P_crit | Condition | Significant | Remarks            |
|-----------|---------|--------|-----------|-------------|--------------------|
| 5% of POFA| 0.24    | 0.05   | P > P_crit| No          | Reject the hypothesis |
| 6% of POFA| 0.43    | 0.05   | P > P_crit| No          | Reject the hypothesis |
| 7% of POFA| 0.70    | 0.05   | P > P_crit| No          | Reject the hypothesis |

3.4 Penetration Index
Based on Table 5, the penetration index value was found to be within the range of -3.1 to -2. Penetration index less than -2, means that the bitumen has high temperature susceptibility. Asphalt mixture with this high temperature susceptibility bitumen will face with permanent deformation at high temperature, and it will easily crack at low temperature.

| Penetration Index | Control sample | 0 | 1 | 2 | 3 | 4 |
|-------------------|----------------|---|---|---|---|---|
| 5%                | -2.6           | -3.1 | -3 | -2.5 | -2.20 | -3 |
| 6%                | -2.97          | -3.00 | -2.97 | -2.50 | -2.00 | -3.05 |
| 7%                | -2.50          | -3.00 | -2.8 | -3.00 | -3.00 | -3.00 |

4. Conclusion
From the results obtained, the following conclusions can be drawn:

(i) From softening point and penetration test results, addition of POFA into bitumen have either increased or decreased the value respectively for certain samples.
(ii) However, ANOVA analysis shows that for all conditions, the p value was found to be more than 0.05. It can be conclude here that the grinding time and percentage of POFA does not contribute to any significant effect towards penetration and softening point result.

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