Evaluation of the Impact of Short-Term Aging on Volumetric and Marshall Properties of Palm Oil Clinker Fine Modified Asphalt Concrete (POCF-MAC)

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Abstract. Disposal of palm oil mill waste material has become a significant factor impacting the environmental situation. Among the palm oil waste the Palm Oil clinker (POC) is usually discarded in the landfill with no economic use. The use of waste materials as modifiers for bitumen mixtures is one of the better alternatives. For this study, the impact of short-term aging on Palm oil clinker fine (POCF) modified asphaltic mixtures was investigated. The POC chunks were initially grounded and sieved through the British standard (BS) 0.075 mm sieve to obtain the POCF that can be utilized as a bitumen modifier at 4%, 6%, and 8% by weight of bitumen. In this investigation, 4.9% optimum bitumen content was selected with a 60/70 penetration grade bitumen to investigate the properties of the POCF on the ACW 14 modified asphalt mixture before and after aging. The study implemented a laboratory simulation of short-time aging (S-TA). Volumetric and Marshall properties were evaluated and analysed to compare the unaged and aged samples. Furthermore, the regression model validates significantly (R² value 0.89-0.99) that variation in the aging condition and POCF content is directly related to the variation in the Marshall and volumetric properties. The investigation shows that the aging condition and POCF content had a significant effect on voids, Marshall stability, and Marshall quotient, as the S-TA sample showed better results compared to unaged this finding was also verified by ANOVA analysis. The research finding indicates that mixtures with POCF-MB have better performance as compared to conventional mix even under S-TA.

1. Introduction
The surge in the number of heavy traffic axle loading has made conventional mixtures not able to withstand the stress and loading. Similarly, aging that occurs before and during compaction of asphalt mixture referred to as short-term aging (S-TA) [1] also affect the pavement as the aggregate tends to absorb more bitumen at this stage[1]. This aging (oxidation) usually occurs at a higher rate in places with a hot climate where the temperatures are high like Malaysia which causes the mixtures to become stiffer due to reaction with oxygen before compaction and laying of the pavement. Hence modification can be to enhance some of its properties. Bitumen modification could help to improve pavement performance and decrease pavement distresses such as fatigue cracking and permanent deformation by enhancing the service life [2]. Polymer modification of bitumen was the commonly used modification in the last few decades as there was a substantial enhancement from literature in terms of rheological and mechanical properties of the bitumen[3, 4]. Also, the utilization of modifiers...
in asphalt binders tends to reduce the optimum bitumen content, improve Marshall stability, enhances the cohesion between bitumen and mineral aggregates[5]. Furthermore, the use of bitumen modifiers can reduce the environmental effect on the roads like traffic noise, maintenance, and rehabilitation cost[2]. With the paradigm shift towards sustainability, a lot of researchers have explored the use of waste materials to enhance the properties of the bitumen. Previous research have showed the effect of utilizing waste like Palm oil fuel ash(POFA) [6, 7], cylinder oil[8], waste oil[9, 10] cup lump rubber[11, 12] as bitumen modifiers. Malaysia been one of world’s principal supplier of crude palm oil and palm oil is the country's largest agricultural industry. Malaysia reportedly has a palm oil plantation of some 4 million acres[13]. The estimated yield of world’s principal supplier of crude palm oil and palm oil is about 75 million tons per year using a 20 per cent average oil extraction ratio (OER). [13].

However, a large amount of waste material is produced during the processing of crude palm oil, which include palm oil fiber (POF), shells, and empty bunches of fruits (EFB). Many of the high-fuel-value waste materials, such as POF and shells, can be recycled to generate energy to produce the electricity needed for the extraction process of palm oil in the mill[13]. During the combustion period, a large amount of palm oil clinker (POC) and palm oil fuel ash(POFA) are produced [13] separately. This process of pyrolysis to produce power in the oil mills ended up creating POC a porous, bulky solid which are dumped near the palm oil mills. Gradually, the quantity of this residue is growing as there is growth in the production of palm oil in Malaysia [14] alone POFA and POC production is over 10 million tons/year[15, 16]. These biomass wastes are considered environment friendly and geopolymer material [17, 18]. In current practice, this residue is normally neglected on landfill that causes contamination of the environment[19, 20] and has now become a burden[21]. These residues are just deposited in Malaysia's landfills with minimal utilization of this material in other industries[22]. POFA and POC are one of Malaysia's most important biomass residues that can be used in the construction industry[19]. However, researched work has shown that POFA-modified bitumen gives additional susceptibility to mineral aggregate coating and adhesion failure compared to unmodified samples. It also reduces mixing temperature [7, 23, 24] that is prone to bitumen, rutting, fatigue, and thermal cracking.

With a lot of pressure on conventional resources like bitumen and aggregate, which are unable to cater for the increase in traffic loading and pavement distresses like rutting and fatigue cracking, there is a need to modify the bitumen or mixtures to overcome those loading and distresses. Therefore, as major by-product discarded in Malaysia from the palm oil mills is POC with limited use in other industries [19] has been utilized as a substitute for aggregate material in concrete as evaluated by researchers like [25-28] and for cement substitute by [29-31], the results of the studies have shown concrete properties enhancement. POC is ideal in the concrete mixture to replace standard gravel aggregate and also to match pavement design[32]. It can be seen, based on past studies, POC was most frequently used as aggregate or cement substitute for construction materials However, there are limited studies on the use of POC as bitumen modifier under of short term aging effect of asphalt mixture which occur during the pre-compaction process of the pavement construction.

2. Materials

2.1 The Mineral Aggregate

The aggregate selected for the research activities were granite stone collected from Sunway Quarry Kampar, Ipoh, Perak primarily used for the construction of highways. The mineral aggregate were kept for 24 hours at the temperature of 120°C in the oven to dry. The aggregate gradation ACW(AC14) was selected according to the stipulated gradation of the Malaysian Ministry of Public Works (PWD) for a wearing course for heavy traffic[33]. Sieve analyses were carried out, and the available grading for the aggregate used in the study is shown in Table 1.
### Table 1: Ministry of Public Works of Malaysia (PWD) specified ACW14 gradation limits

| Sieve size (mm) | Specified percent passing range | Percent passing used (%) |
|-----------------|---------------------------------|--------------------------|
| 20              | 100 - 100                       | 100                      |
| 14              | 90 - 100                        | 95                       |
| 10              | 76 - 86                         | 81                       |
| 5               | 50 - 62                         | 56                       |
| 3.35            | 40 - 54                         | 47                       |
| 1.18            | 18 - 34                         | 26                       |
| 0.425           | 12 - 24                         | 18                       |
| 0.15            | 14 - 16                         | 10                       |
| 0.075           | 4 - 8                           | 5                        |
| Pan             |                                 | 5                        |

2.2 Bitumen

The bitumen used was PEN 60/70 grade obtained from PETRONAS refinery Malacca, Malaysia. It was utilized for all ACW 14 mixtures (control and modified samples). An investigation was conducted to evaluate the characteristics of pristine bitumen. Physical characteristics of the pristine bitumen utilized met all Malaysian public works department (PWD) standards specifications [33].

### Table 2: Pristine bitumen physical properties

| Bitumen properties | Bitumen Grade 60/70 | Specifications |
|--------------------|---------------------|----------------|
| Penetration (dmm)  | 68                  | ASTM D5 -06    |
| Softening point (ºC)| 48                  | ASTM D36M – 09 |
| Ductility at 25ºC(cm) | >130                | ASTM D113-07   |
| Specific gravity   | 1.02                | ASTMD70-03     |

2.3 Palm Oil Clinker Fine (POCF)

The POC was retrieved from a KATUTAH biomass oil mill in CHEMOR, PERAK, the POC, was collected as waste from the palm oil shell combustion also fiber which was discarded within the vicinity of the company. Fig 1 displays the image of POC chunks, a solid substance of blackish-grey colour. The Bulk POC’s free water was eliminated by heating it in the oven for 3 hours at 100ºC. The moisture-free POC was grounded at 150 revolutions per minute (RPM) for 8 h using the Los Angeles abrasion machine. It then became a powder material. POCF was then sieved with the 0.075mm BS sieve and the filtrate was used as the research material and referred to as “POCF”. Different percentages (4%, 6%, and 8%) by weight of the bitumen were used as modifier.

![Figure 1: The process of producing POCF](image)
2.3.1 Chemical compositions of POCF

X-ray fluorescence (XRF) was carried out to indicate the chemical composition of POCF as shown in Table 1. It was observed that Silica oxides, alumina, potassium oxide, and calcium oxide account for the major percentage. Thus, POCF possesses some pozzolanic properties of class C since the sum of SiO2, Al2O3, and Fe2O3 is over to 50 percent but less than 70 percent as specified by ASTM C 618 [34].

| Chemical Composition (weight %) | POCF  |
|---------------------------------|-------|
| SiO3                            | 53.70 |
| Al₂O₃                           | 1.46  |
| Fe₂O₃                           | 3.87  |
| SiO₃ + Al₂O₃ + Fe₂O₃ (SAF)     | 59.03 |
| K₂O                             | 13.90 |
| MgO                             | 2.37  |
| P₂O₅                            | 5.29  |
| SO₃                             | 0.92  |
| CaO                             | 17.0  |
| Loss on ignition (LOI)          | 4.95  |
| Moisture content                | 0.96  |
| Specific gravity                | 2.58  |
| Colour                          | Blackish grey |

2.3.2 Mineralogy Phase of POCF

X-Ray Diffraction (XRD) is a method, which is widely used by researchers to classify material amorphous nature[35]. Differences in the formation of amorphous or crystalline silica can be detected by the XRD technique; however, amorphous material exhibits high reactivity [36]. The POCF XRD pattern is described in Fig. 2. A large hump of amorphicity halo with angular 2 spectrum of 20–35 ° from the inset of figure 2 below, whereas the remaining are crystalline phases.

![Figure 2: XRD pattern of POCF](image)

The POCF materials show a central halo at the 2θ around 26.7° which is linked to the overlap of amorphous forms of silica. Thus, this amorphsity halo indicates the material reactivity. The reactivity of SiO2 also depends on its crystalline nature [37]. Prior studies have shown that amorphous silica plays a major part in pozzolanic behaviour of a material [38, 39].
3. Methodology

The test procedures were based on the ASTM and AASHTO laboratory works standard guide. An aging test was used to assess specimen degradation. The aging technique adopted for this research was specified by AASHTO R30-02[1]. At a mixing temperature of 140ºC, POCF was mixed with 60/70 bitumen penetration for about 52 minutes at 1000 rpm stirring speed. The unaged mix samples and aged mixes were prepared in compliance with ASTM D1559, based on the Marshall mix design process AC14 aggregate gradation was used in mix design according to the road work requirements of the Department of Public Works of Malaysia (PWD Malaysia) [33]. The optimum bitumen content (OBC) for the control mix used for the study is 4.9%. Samples for unaged (U-A) and short-term aging(S-TA) conditions were prepared using the OBC in the laboratory under the same conditions as a control mix until the mixing phase. Then, evenly, the loose mix was placed in trays and set for 4 hours at temperature of 135±3 °C in a force draft oven. To simulate the aging and hardening of bitumen and mixtures during the construction and pre-compaction phase using the draft oven according to AASHTO R30 2002 [34]. The aging process ended by removing mixtures from the oven; afterwards the loose mixtures were heated to the compaction temperature and compacted using the gyratory compactor.

Statistical analysis like regression and ANOVA were carried out to investigate if the modifier (Independent variable) has a substantial effect on the aged and unaged asphaltic mixtures(dependant) properties. The analysis was carried out at a significance level (0.05). This is done by using F-distribution to compare the means of the two groups, in this case, volumetric properties of the asphaltic concrete (dependant variables) and percentage of the POCF (independent variable) in terms of S-TA. Regression analysis was also used to examine the relationship between two or more variables like the POCF (independent variable), volumetric and Marshall properties(dependent). The coefficient of determination $R^2$ (Range from 0 - 1) was used to measure the correctness and significance of the regression model in question (linear or polynomial) that best described the significance of variance in the dependent variables which is predictable from the independent variables.

4. Results and Discussion

4.1 Voids in Total Mix (VTM)

Figure 3 illustrate the relationship between the VTM and POCF material of asphalt mixtures under different aging conditions. The findings show that the VTM of POCF modified asphaltic concrete mixture after aging is higher when compared with the VTM of unaged mixtures. It was also observed that the unaged Asphalt concrete VTM of the improve from 3.5% to 4.1% as the POCF material increases from 0% to 8% when the equivalent VTM rises from 3.9% to 4.7% in the S-AT conditioned samples. The values of VTM of all mixes were within the range specified by the Malaysian public works department(PWD) for wearing course[33]. Higher content of air void enhances the aging of asphalt bitumen within the aggregate mass. While lower air void reduced aging and makes mixtures less penetrable[40, 41]. It is also noticed that very low air void can result in increased bitumen bleeding and plastic flow(rutting). It was observed that amount of air voids in asphaltic concrete mixture is of paramount importance and is closely related to stability, permeability, and durability.
4.2 Voids Filled with Bitumen (VFB)
As depicted in the figure 4, the modified mixtures have greater VFB values than the unmodified mixture. This is because the modified bitumen tends to fill up more spaces in asphalt concrete. Also, it was observed the VFB increased substantially with the rise in POCF material for the S-TA conditions. This can also be due to the aging effect which makes the bitumen to lubricate the aggregate and the matrix rearrange to a more densely packed orientation thus leading to a reduction in the voids until it gets to a minimum. As for S-TA, the decrease in VFB of the aged samples Shows a decline in the effective bitumen film thickness between the mineral aggregate resulting in higher low-temperature cracking and could result in lower asphalt concrete strength as the bitumen has a filling and healing effect to enhance the mixture ‘s flexibility. The maximum VFB was achieved within the percentage of POCF from 4% to 6%. Also, it was inferred that the increase in the amount of POCF enhanced the VFB, resulting in denser mixtures as reported by [40, 41] .The values of VFB of all blends were within the range stipulated by Malaysian public works department(PWD) for wearing course [33].

4.3 Marshall Stability (MS)
The stability is related to the resistance of asphalt concrete to displacement, shear stresses and permanent deformation which is mainly derived from cohesion and internal friction. Because as the
asphalt concrete is subjected to continuous heavy traffic loads regularly, it is essential to adopt asphalt concrete with good stability and flow. As shown in figure 5 for 0% (Control), 4%, 6% and 8% POCF modified mixtures showed stability values of 11.39kN, 13.73N, 14.07N and 13.31kN respectively, at OBC. As the viscosity increases the MS of the mixes also increases. For all mixes, asphaltic concrete stability improves with aging, an improvement in stability is partly attributed to bitumen hardening with values of 11.81N, 14.55kN, 14.65kN, and 14.14N. As stated by Romastrika, et al. [42] stated that beyond a certain time of exposure and aging bitumen becomes harder. The findings also show that the stability improves as the POCF content incorporation rises to a maximum peak and then reduces with further modifications. Furthermore, the values of MS of all mixes were within the range specified by Malaysian public works department(PWD) for wearing course[33] Comparable observation were reported by Jaya, et al. [41] and Ramadhansyah, et al. [40] when black rice husk ash (BRHA) and coconut shell ash effect on the short term aging were investigated. Thus, from this research optimal addition POCF rate was 6 percent with increase in MS of 4% of the aged samples in comparison with the unaged mixtures.

4.4 Flow
The flow value is an indicator of the capacity for the asphalt mixture to deform[40]. High flow suggested low resistance to the displacement of asphalt concrete. The figure 6 depicts the relationship between flow and POCF content at the different aging conditions. It was observed that asphalt concrete subjected aging conditions did not have much variation in the flow. The flow increases continuously with an increase with increase in POCF content up to the maximum beyond which its experiences a decline. This could be ascribed to an increase in flexibility of the mixes as the POCF dosage increases making the asphaltic concrete more flexible leading to more displacement of the mixtures. Nonetheless, the values of the flow of all mixes were within the range specified by the Malaysian public works department(PWD) for wearing course[33]. Volumetric properties have an important impact on asphalt pavement performance [43] as the stability and flow values reflect this effect from the experiment.

4.5 Marshall Quotient (MQ)
MQ is defined as the stability to flow ratio, it serves as an indicator to a measure material's resistance to shear stress which is attributed to resistance of the mixture to rutting[44]. Asphalt concrete with high MQ values are related to increased stiffness and improve asphalt concrete resistance against creep deformation[44]. MQ values are illustrated in figure 7, it is noted that the MQ values shows a similar trend to that obtained for MS. An increase in the MS and the MQ values suggest that even
after short term aging, the POCF modified asphalt mixtures have immense promise to bear heavy traffic loads and thus resist failure in permanent deformation. Because of the impact of aging, the mixture is stiffer than expected. Research has shown that aging induces oxidation and speed up the bitumen rate of hardening thus, leading to increased stiffness[43]. From the research, at 4% and 6% POCF unaged and aged samples a substantial increase in the MQ value was observed before it is reduced at 8% but still higher than the conventional. Thus, it can be suggested that the optimal additional POCF percentage was between 4 and 6 % with the POCF-MAC having enhanced stiffness of 13.7 and 13.0% higher than control mixtures which is a predictor of higher resistance against rutting, premature oxidative and pre-compaction aging.

Figure 7. MQ versus POCF-MAC under S-TA

4.6 Statistical Analysis

4.6.1 Analysis of Variance (ANOVA)
The results of the ANOVA analysis of the volumetric and Marshall properties of the unaged and S-TA asphalt concrete are shown in Table 3. The analysis showed that the asphalt concrete modified with POCF have a significant effect and boosted performance in term of Voids, MS, and MQ. Thus, the analysis suggested that the POCF even under aging conditions can improve asphaltic concrete mechanical properties. Furthermore, from the analysis, the modifier has displayed a considerable measure of improvement on the premature resistances to oxidative aging and rutting resistance as it improves the stiffness of the asphalt concrete with percentage variation in modifier and aging condition using the significant level of 5%.

Table 4: ANOVA results on the properties of POCF modified asphalt mixtures properties.

| POCF | VFB (%) | VTM (%) | Flow (mm) | MS (kN) | MQ (kN/mm) |
|------|---------|---------|-----------|---------|------------|
|      | U-A | S-TA | U-A | S-TA | U-A | S-TA | U-A | S-TA | U-A | S-TA |
| 0%   | 74.3 | 72.1 | 3.5 | 3.9 | 3.61 | 3.65 | 11.39 | 11.81 | 3.15 | 3.23 |
| 4%   | 75.5 | 72.6 | 3.6 | 4.3 | 3.76 | 3.81 | 13.73 | 14.55 | 3.65 | 3.82 |
| 6%   | 76.8 | 74.4 | 3.9 | 4.4 | 3.88 | 3.92 | 14.07 | 14.65 | 3.62 | 3.74 |
| 8%   | 78.6 | 76.4 | 4.1 | 4.7 | 3.82 | 3.86 | 13.31 | 14.14 | 3.48 | 3.66 |
| F cal | 1369.3 | 1243.9 | 0.179 | 0.01 | 0.19 | 0.16 | 445.59 | 416.43 | 838.19 | 760.76 |
| F crit | 5.98 | 5.98 | 5.98 | 5.98 | 5.98 | 5.98 |
| P value | 2.6E-8 | 3.5E-8 | 0.696 | 0.92 | 0.68 | 0.70 | 7.4E-7 | 9.0E-7 | 1.1E-7 | 1.5E-7 |
| P crit | < 0.05 | > 0.05 | > 0.05 | < 0.05 | < 0.05 |
| Remark | Significant effect | Insignificant effect | Insignificant effect | Significant effect | Significant effect |

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|------|---------|---------|-----------|---------|------------|
|      | U-A | S-TA | U-A | S-TA | U-A | S-TA | U-A | S-TA | U-A | S-TA |
| 0%   | 74.3 | 72.1 | 3.5 | 3.9 | 3.61 | 3.65 | 11.39 | 11.81 | 3.15 | 3.23 |
| 4%   | 75.5 | 72.6 | 3.6 | 4.3 | 3.76 | 3.81 | 13.73 | 14.55 | 3.65 | 3.82 |
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|------|---------|---------|-----------|---------|------------|
|      | U-A | S-TA | U-A | S-TA | U-A | S-TA | U-A | S-TA | U-A | S-TA |
| 0%   | 74.3 | 72.1 | 3.5 | 3.9 | 3.61 | 3.65 | 11.39 | 11.81 | 3.15 | 3.23 |
| 4%   | 75.5 | 72.6 | 3.6 | 4.3 | 3.76 | 3.81 | 13.73 | 14.55 | 3.65 | 3.82 |
| 6%   | 76.8 | 74.4 | 3.9 | 4.4 | 3.88 | 3.92 | 14.07 | 14.65 | 3.62 | 3.74 |
| 8%   | 78.6 | 76.4 | 4.1 | 4.7 | 3.82 | 3.86 | 13.31 | 14.14 | 3.48 | 3.66 |
| F cal | 1369.3 | 1243.9 | 0.179 | 0.01 | 0.19 | 0.16 | 445.59 | 416.43 | 838.19 | 760.76 |
| F crit | 5.98 | 5.98 | 5.98 | 5.98 | 5.98 | 5.98 |
| P value | 2.6E-8 | 3.5E-8 | 0.696 | 0.92 | 0.68 | 0.70 | 7.4E-7 | 9.0E-7 | 1.1E-7 | 1.5E-7 |
| P crit | < 0.05 | > 0.05 | > 0.05 | < 0.05 | < 0.05 |
| Remark | Significant effect | Insignificant effect | Insignificant effect | Significant effect | Significant effect |
4.6.2 Regression Analysis

The regression modelling of the properties of POCF modified asphalt concrete at various aging condition is depicted in table 4 and the values of $R^2$ is within 89% to 99%; which is an indicator that the model can validate that a significant percentage of variation in the modifier content at different aging condition is directly related to or can be explained by the variation in the properties of the modified asphalt concrete with POCF. Furthermore, the $R^2$ demonstrates the importance of POCF content's contribution to improving the properties during the S-TA of the asphalt concrete. The findings go to buttress the fact stated earlier that the use of POCF as a modifier also helps in improving the stability, stiffness and also helps increases the voids filled with bitumen which is essential in lubricating the aggregate to aid in better binding action to form a denser asphalt concrete.

| Table 5: Coefficient of determination of the properties of U-A and S-TA POCF-MAC |
|----------------------------------|-------|-------|--------|--------|--------|
| Mixture properties  | VFB     | VTM    | FLOW   | MS     | MQ     |
| Graph Function        | Quadratic | Quadratic | Quadratic | Quadratic | Quadratic |
| $R^2$ for U-A         | 0.992   | 0.961  | 0.982  | 0.962  | 0.894  |
| $R^2$ for S-TA        | 0.998   | 0.980  | 0.972  | 0.989  | 0.948  |

5. Conclusion

The use of agricultural by-products in asphalt construction is strongly recommended to promote sustainability either as replacement or modifier as this research study demonstrated that POCF has significant potential to be used as modifier. Based on the asphalt concrete results POCF-MAC has better performance as it produces mixtures that are comparatively denser blend of mixture when compared to the control mixtures. The outcome further demonstrates that the POCF-MAC at various dosages shows improved performance compared to the unmodified mixtures at both aging conditions. The Statistical analysis demonstrated that POCF-modified asphalt concrete has a major impact and improved performance in terms of Voids, MS and MQ(Stiffness) which are good indicators for dense and stiff asphalt mixtures and predictor for resistance against rutting, premature oxidative aging, and structural weakening furthermore the $R^2$ range from (0.89-0.99) validate the model and effectiveness of the application of the POCF content to enhancing the asphalt concrete properties during the aging process.

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