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Engineering Properties of Palm Oil Clinker Fine-Modified Asphaltic Concrete Mixtures

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Highlights:

- The feasibility of using POCF as alternative modifier was evaluated.
- POCF as modifier improved conventional properties of the base bitumen.
- The POCF modified mixtures showed improved engineering properties.

Abstract. Palm oil clinker (POC) is a non-biodegradable palm mill by-product typically discarded in dumpsites. This study analyzed the performance of POC powder (POCF) as bitumen modifier in terms of conventional and engineering properties of bitumen and asphalt mixture. For the study, base bitumen of 60/70 penetration grade was utilized and different POCF dosages (0, 2, 4, 6, and 8% by weight of bitumen) were added. The base bitumen was effectively modified with POCF and then characterized. The conventional and engineering properties of the modified bitumen and asphalt mixtures were assessed. From the characterization results, the formation of Si-O crystalline structure and a new Si-OH functional group was identified. Furthermore, a meandering pattern was observed due to the modification of the base bitumen with POCF. Based on the conventional test results it was revealed that the addition of POCF to the base bitumen resulted in a stiffer blend compared to unmodified bitumen. The addition of POCF improved the modified mixtures' Marshall stability relative to the unmodified mixtures. Analysis of variance (ANOVA) and regression modeling showed the influence and significance of POCF-MB, with R^2 values of (95-99%). Finally, the 4-6% POCF dosage was found to be the optimum dosage, yielding the best performance in terms of the engineering properties evaluated.

Keywords: *asphalt mixture; bitumen; engineering properties; modifier; fine palm oil clinker.*

1 Introduction

Conventional asphalt mixtures are unable to cope with the rise in traffic loads, resulting in pavement distresses such as rutting and fatigue cracking. It is

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necessary to improve and modify conventional bitumen to overcome these distresses [1]. Bitumen plays a major role in evaluating asphaltic concrete viscous behavior as it is used as a binder in road paving to provide an adhesive and protective coating for aggregates [2]. However, with the rise in traffic volume and low reliability of the road life cycle process, road structure degradation occurs more frequently these days, as conventional materials can no longer cater to the increase in axle wheel loads, which leads to deterioration of the asphaltic concrete pavement. Thus, modification is required to improve the flexible material layer.

Research on the application of bitumen modifiers from waste has sparked a renewed interest in bitumen modification studies [3,4] aimed at finding alternative waste materials to minimize bitumen dependency [4]. Also, bitumen is modified to improve its properties and enhance long-term pavement performance. Most modifiers are used to minimize temperature dependence, bitumen oxidative hardening, and asphalt mixture moisture susceptibility and rutting [5]. To decrease life cycle costs and provide environmental advantages, the use of industrial and biomass waste as modifiers to improve asphalt mixture characteristics has recently been considered [1,6]. Also, industrial wastes such as steel slags for the surface of asphalt mixtures such as stone mastic asphalt (SMA) have been utilized to improve their performance [6,7]. The problem of biomass waste accumulation exists worldwide, but especially in Southeast Asia. Malaysia is one of the largest palm oil producers, generating large quantities of palm oil waste, such as palm oil clinker (Jagaba, *et al*. [8]).

The palm oil industry is a significant contributor to the country's contamination crisis, producing around 2.6 million tonnes of agricultural waste yearly [9]. With high conventional modifier costs, it will be beneficial to use alternative (waste) materials as bitumen modifiers. The use of biomass waste in the pavement industry is of keen interest to highway engineers to meet the growing challenges and harness the waste potential. Waste materials have been shown to be capable of improving bitumen blends and mixtures properties [10-13]. POC is a major by-product in Malaysia, dumped by palm oil mills and with limited usage in other sectors [13-15]. In the concrete industry, POC has been utilized as a replacement for aggregate material [16] and as cement substitute [17,18]. These studies' results indicated improved concrete properties. To mitigate the environmental threats of POC waste and the excessive traffic on roads, POC has been employed as bitumen modifier. From the literature review, there are limited studies on the use of POCF as alternative bitumen modifier.

The fundamental objective of this study was to analyze the influence of modifying bitumen with POCF and to evaluate the Marshall properties of asphalt mixtures produced with different dosages of POCF incorporated into the bitumen. Also, ANOVA and regression analysis were performed to evaluate significant effects and the reliability of POCF on the engineering properties of the asphalt mixtures.

2 Materials and Methodology

2.1 Materials

For this research, base bitumen of penetration grade, 60/70 was used for sample preparation, as suggested for tropical environments by the Malaysian Standard Specification for Road Works (JKR) [19]. Petronas Refinery Malacca supplied the bitumen. Based on preliminary standard tests conducted in the laboratory, the bitumen conventional properties are shown in Table 1.

Table 1 Base bitumen conventional properties.

Properties	Units	Specification	Standard Limits	Results
Specific gravity	-	ASTMD70-18	1.0-1.06	1.03
Penetration	dmm	ASTMD5-13	60-70	67
Softening point	°C	ASTMD36-12	47-52	48.9
Ductility at 25 °C	cm	ASTM D113-07	>100	132
Mass loss	%	ASTM D2872	–	0.02

Granite aggregate obtained from the Sunway Quarry in Perak, Malaysia was utilized. A gradation plot of the asphalt mixtures with nominal maximum aggregate size 14 (AC14) based on JKR is shown in Figure 1.

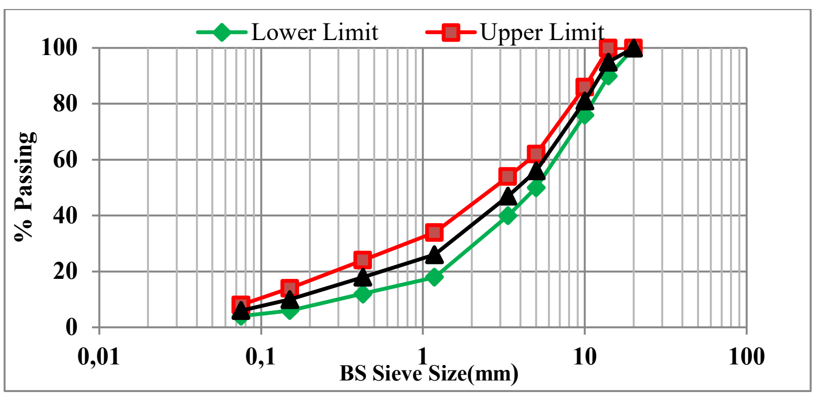

Figure 1 Gradation plot as per JKR-specified limits [19].

The POC was collected in the vicinity of the Kayutah palm mill located in Perak, Malaysia. The POC was dried and then grounded using Los Angeles abrasion machines based on ASTM C131-06 at 150 RPM for 8 hours and then sieved through standard 75 µm British standard sieve to qualify as a modifier. The filtrate, called palm oil clicker fine (POCF), was utilized for this research. To mitigate inconsistency, the POC, the bitumen, and the mineral aggregates were obtained from the same supplier throughout the whole study.

2.2 Methodology

2.2.1 Preparation of Modified Bitumen

For this study, POCF-modified bitumen (POCF-MB) was prepared with 2, 4, 6, and 8% dosages of POCF by weight of base bitumen. The bitumen was heated at a temperature of 120 °C in an oven to let it turn into the required viscous state for easy mixing. Before mixing, a known weight of the base bitumen was measured and poured into a stainless-steel vessel flask for mixing. Using dry blending, the POCF was then added progressively to prevent aggregation according to the base bitumen's weight and mixed using a multi-mixer at a shear rate of 1,000 rpm and a mixing temperature of 140 °C for 52 minutes to obtain a homogeneous bitumen blend.

2.2.2 Bitumen Conventional Properties Tests

The conventional properties of the prepared bitumen samples, i.e. penetration test, softening point test, penetration index, and storage stability tests, were evaluated. The penetration test was carried out as stipulated in ASTM D5 to assess the consistency of the base bitumen and modified bitumen at a temperature of 25 °C. The softening point as the highest temperature beyond which a standard steel ball can be sustained by bitumen [20] was conducted according to ASTM D36. For studies on bitumen's temperature sensitivity, the penetration index (PI) value was assessed using nomographs of Van Der Poel, as shown in Eq. (1)

$$PI = \frac{1952-500logPen_{25}-20S}{50logPen_{25}-sp-1} \tag{1}$$

where Pen_{25} represents the penetration value and SP is the bitumen softening point. Finally, to evaluate the storage stability of the POCF-MB blends and verify their compatibility as per ASTM D5892 standard storage stability tests were performed.

2.2.3 Marshall Sample Preparation

Marshall samples were prepared and compacted using a gyratory compactor. The test was conducted as stipulated by ASTM D5581-07. The optimum bitumen content (OBC) of the control mixtures (without POCF) was evaluated and found to be 5.1% of the weight of the total mix, based on JKR's specification [19]. This control mixture OBC was utilized for the preparation of the modified asphalt mixtures at the different dosages of POCF (2, 4, 6, and 8%) by weight of the control OBC to facilitate the analogy of the control. The POCF modified mixes were analyzed without considering bitumen as separate variable and their properties.

The volumetric and Marshall stability test results were analyzed to examine the engineering properties of the asphalt mixtures compacted with a standard laboratory gyratory compactor at 100 gyrations for heavy traffic loading. Analysis of variance (ANOVA) and regression analyses were used to statistically investigate if the modifier (independent variable) had a significant effect on the engineering properties (dependent variables) of the modified asphaltic mixtures at a 95% confidence interval. Also, regression analysis was done to assess the reliability and significance of the regression model with an R^2 range of (0 to 1).

3 Results and Discussion

3.1 Bitumen Conventional Test and Characterization

3.1.1 Conventional Properties

The POCF-MB influenced primary consistency test results presented in Table 2 and shown in Figure 2 are explained below. For the penetration, the decline in value can be explained by the increased stiffness of the modified bitumen, which is most likely related to the resistance to particle movement within the bitumen matrix Also, the rise in softening point attributed to the POCF particles' surfaces makes it simpler for them to interact with the bitumen, which increases the viscosity of the modified bitumen, thus making the blend more difficult to overcome frictional resistance as the POCF dosage increases, thus making the blend stiffer.

Table 2 Conventional testing for POCF-MB.

Samples	Penetration (dmm)	Softening point (°C)	Penetration Index (PI)	Storage stability (°C)
Base bitumen	67	48.0	-1.03	0.2
2%POCF-MB	60	49.0	-1.03	0.9
4% POCF-MB	58	49.6	-0.96	1.4
6% POCF-MB	57	50.0	-0.90	1.7
8% POCF-MB	55	51.8	-0.54	2.1

The addition of POCF to the base bitumen indicated its significant influence on bitumen stiffness. It also improved its temperature vulnerability and viscoelasticity by decreasing the particle motion as the light components of bitumen were absorbed by the POCF particles. The susceptibility to bitumen temperature is defined as the dependencies of its rheological parameters on temperature [21]. All POCF-MB exhibits reduced temperature susceptibility compared to the base bitumen.

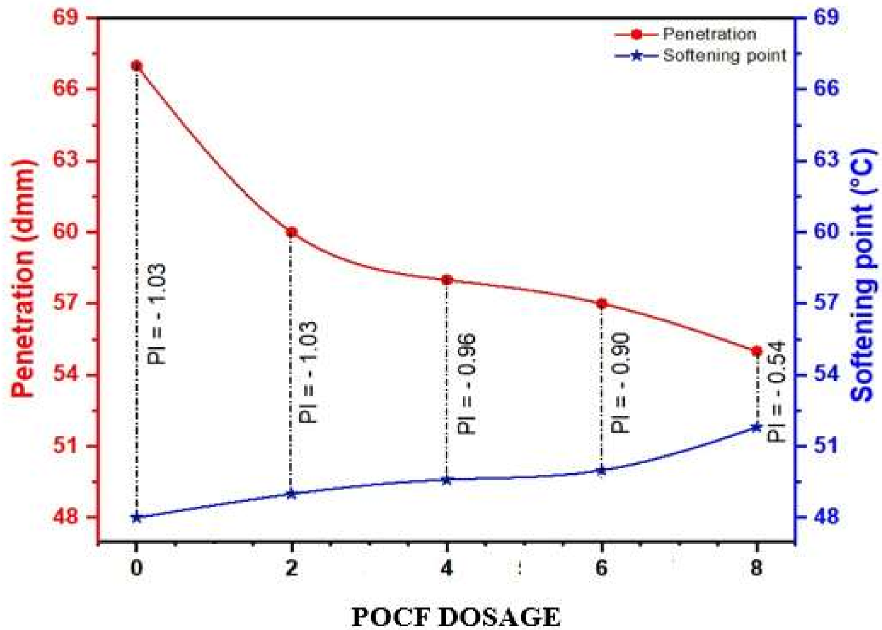

Figure 2 Effect of POCF dosage on penetration, softening point, and PI.

The samples' stability under high-temperature storage conditions was evaluated. It was observed that POCF-MB had good storage stability, with a softening point disparity between the top and bottom tubes of 2.2 °C, which means that POCF-MB storage segregation was low, which indicates that the bitumen blends were mixed homogeneously.

3.1.2 POCF-MB XRD Patterns

Figure 3(a) presents the XRD pattern of the POCF-MB. The crystalline structures of the POCF-MB were assigned to the appearance of two important XRD peaks of 2θ around 20.95° (1 0 0) and 26.65° (0 1 1) in the POCF's XRD. Besides that, with an increase in POCF dosage from 0 to 8%, the peaks' amplitude showed an incremental enhancement. The modification subsequently led to the obvious reaction of SiO_2 or crystalline POCF particles with the bitumen as new asphaltene sheets detected at 2θ = 23.9° for the modified bitumen. These findings are consistent with the literature [22,23].

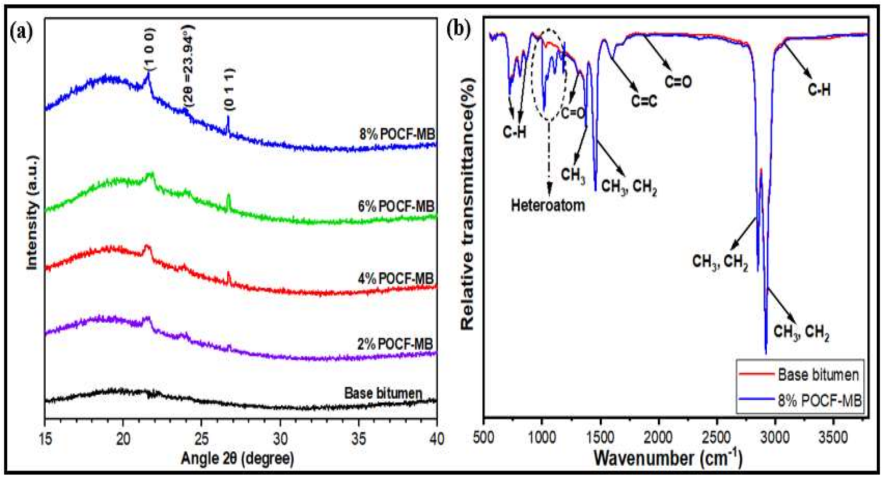

Figure 3 (a) Combined XRD pattern of base and various POCF-MB mixtures, (b) FT-IR spectra of base bitumen, and 8% POCF-MB, indicating the functional group's bonding vibrations.

3.1.3 POCF-MB FTIR Spectra

The base bitumen and 8% POCF-MB are shown in Figure 3(b). It was clear that significant changes occurred. At wavenumber 1030 cm^{-1}, the emergence of an absorbance region can be seen. The appearance of Si $(OH)_4$ is seen in the 1,102 cm^{-1} absorption wave number, indicating that the Si-O–Si bond was formed by Si-O vibration deformation. As shown in Figure 3(b), the addition of POCF led to a peak in chemical bond shifting of the bitumen bonds, as observed around 1,010 cm^{-1} of absorbance, showing that there was $SiOH_4$ present, which corresponds to stretch vibrations caused by the displacement of the SiO bonds, indicating that silica has a wide range of bitumen reactivity. This may be deduced from the proportional quantity of silica and carbon in the POCF, generated by a greater percentage of improperly bonded regions between the functional groups of bitumen hydrophobicity and the POCF surface. Thus, the bitumen particle's motion is increased, which produces a stiffer bitumen with decreased penetration value and an increased softening point by exhibiting bond modification for the modified bitumen. The chemical bond analysis showed that the base bitumen underwent slight alterations between its bonds with the incorporation of POCF.

3.1.4 POCF-MB SEM

Surface images of the base bitumen and the modified bitumen with 8% POCF were obtained from the SEM analysis. The microstructure of the modified bitumen had a rather different characteristic compared to the base bitumen, as shown in Figure 4(a). The SEM for 8% POCF-MB was substantially modified relative to the base bitumen, as shown in Figure 4(b), which indicates different irregular wandering patterns, most likely due to the asphaltene structure's arrangement, which is caused by the addition of POCF to the base bitumen and shows the presence of SiO_2 in the POCF-MB, as observed from the EDX. These findings are in line with the previous literature where wastes were utilized as bitumen modifier [22,23].

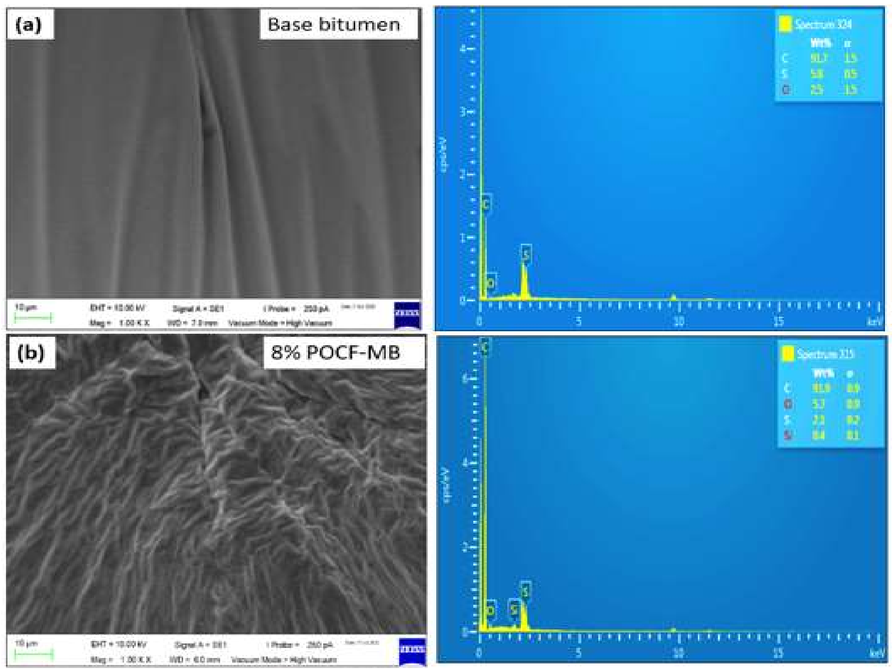

Figure 4 SEM image and EDX of (a) base bitumen and (b) 8% POCF-MB.

3.2 Engineering Properties of POCF-MB Asphalt Mixtures

3.2.1 Bulk Specific Density

Figure 5(a) shows the POCF-MB asphalt mixtures' bulk specific gravity. The bulk density has a linear trend as the POCF dosage increases. The modified samples had higher values of density compared to the control mixture. This may be due to the increased viscosity of the POCF-MB, which tends to penetrate the aggregate and a proper coating is formed. The lowest was 2.398 g/cm^3, recorded for the control sample, while the highest was 2.478 g/cm^3 obtained for the 8% POCF-MB sample.

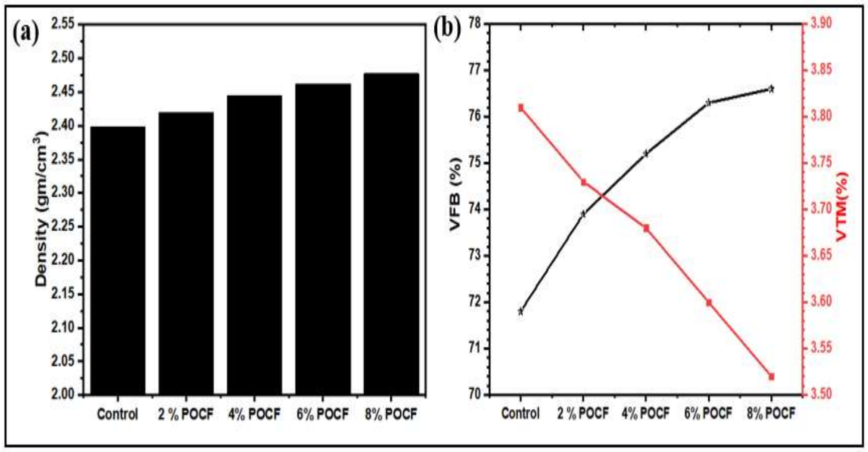

Figure 5 Effect of POCF-MB on mixtures: (a) density and (b) void analysis.

3.2.2 Void Analysis

Figure 5(b) shows the effect of POCF dosage on the asphalt mixtures' voids filled with bitumen (VFB) and the void in total mix (VTM) values. A significant increase in VFB was also seen with an increase in POCF dosage in the bitumen. This is because the POCF-MB improved the viscosity, because of which more gaps in the asphalt mixture fill up, which helps resist low-temperature cracking and creates higher durability. Meanwhile, the VTM was seen to decrease from 3.82% to 3.53% as the POCF dosage increased from 0% to 8%. This trend can be related to the fact that as the POCF-MB content increases, the percentage of voids in the mixture decreases. This is because the enhanced physical properties of POCF-MB require a greater amount of bitumen, which will ultimately lead to improved workability while mixing and improved bulk densities, which in turn leads to a decrease in mixture voids. For all mixtures, the void parameters were within the range defined by JKR [19].

3.2.3 Marshall stability and Flow

Figure 6(a) indicates Marshall stability versus flow for the 0 to 8 % POCF-MB asphalt mixtures. Both the stability and flow of the POCF-MB modified mixtures gradually increased with an increase in POCF content up to the point beyond which a decrease occurs. The improvement in Marshall stability of the POCF-MB asphalt mixtures can be attributed to better bond formation between the components in the mixture, while the declining trend is due to the hydrostatic pressure from the bitumen and the inter-granular interaction of the aggregate. These cause the bitumen to fill the voids, thereby causing a drop in stability value.

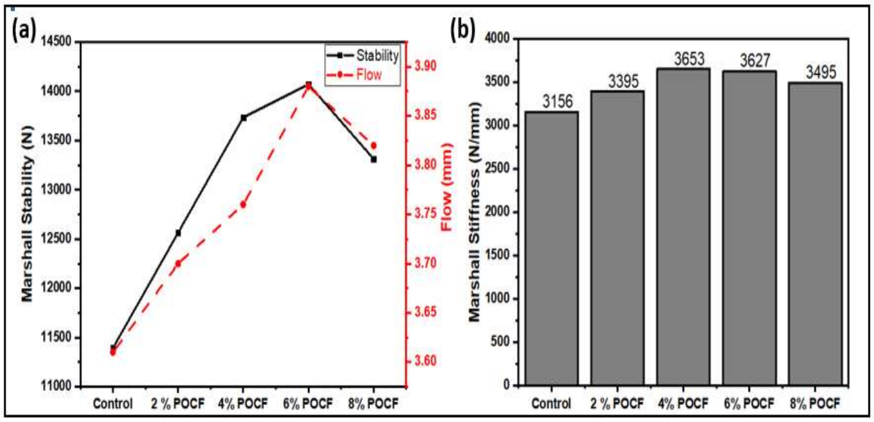

Figure 6 Effect of POCF-MB on mixture (a) stability and flow, (b) Marshall stiffness.

Meanwhile, the trend of flow may be due to an improvement in mixture flexibility as the POCF dosage increased from 0 to 6% and then began to diminish marginally at 8%. As the dosage of POCF increased, the stability and flow value increased. In mixes with a POCF dosage, the bonding between the POCF and the bitumen may be the cause of the increase in flow, which can be ascribed to improved bonding between the POCF and bitumen blends, which improves the mixture's flexibility, resulting in more mixture displacement. The decline may be because of reduced bonding between the POCF and the bitumen in mixes that received a higher dosage. This indicates that the presence of POCF significantly influenced the engineering properties of the asphalt mixtures. The Marshall stability and flow values for all mixes were within the stipulated standards of JKR [19].

3.2.4 Marshall Stiffness

Figure 6(b) shows the stiffness values of the mixes. An improvement in the Marshall stability and stiffness values indicates that the POCF-MB asphalt mixtures have more potential to endure heavy traffic loads and thus minimize the risk of rutting. The POCF-MB asphaltic mixtures between 4 and 6% showed the optimum increase in stiffness, i.e. 13.7% and 13.0% higher than control. The improvement in stiffness can be attributed to improved coating and cohesion of the modified mixtures.

3.3 Statistical Analysis

3.3.1 Analysis of Variance (ANOVA)

Table 3 displays the ANOVA on the effect of POCF addition on the engineering properties of the POCF-MB asphalt mixtures. The analysis revealed that all POCF-MB doses had a measurable impact on the enhanced mixtures' volumetric performance and Marshall properties. Therefore, this study indicates that asphaltic mixtures of POCF-MB tend to enhance the mechanical properties of asphaltic concrete.

Table 3 ANOVA on the properties of asphalt mixture modified by POCF-MB.

POCF	Density (g/cm^3)	VFB (%)	VTM (%)	Flow (mm)	Stability (kN)	Stiffness (kN/mm)
0%	2.39	74.30	3.50	3.61	11.39	3.15
2%	2.42	75.10	3.53	3.70	12.56	3.40
4%	2.46	75.50	3.60	3.76	13.73	3.65
6%	2.52	76.80	3.90	3.88	14.07	3.62
8%	2.59	78.60	4.10	3.82	13.31	3.48
F-cal.	4194	10195	972.14	5757	737	1411
F-cri	5.32	5.32	5.32	5.32	5.32	5.32
P- value	3.6 E-12	1.0E-13	1.2E-09	1.0E-12	3.6E-09	2.8E-10
P-cri	< 0.05	< 0.05	> 0.05	>0.05	< 0.05	< 0.05
Remark	Significant	Significant	Significant	Significant	Significant	Significant

3.3.2 Regression Analysis

The regression analysis of the properties of the POCF modified asphalt mixtures at different dosages is shown in Table 4. The regression model has R^2 values between 0.95 and 0.99, which indicates that the models validate that a significant percentage (95-99%) of the variation in the POCF was directly related to or can be explained by the variation in the engineering properties of the POCF-MB asphalt mixtures. For all properties, POCF-MB addition showed a significant contribution to the enhancement of its engineering properties during its service life.

Table 4 The coefficient of determination of POCF-MB asphalt mixtures properties.

Mixture properties (units)	Density (g/cm^3)	VFB (%)	VTM (%)	Flow (mm)	Stability (kN)	Stiffness (kN/mm)
R^2 values	0.956	0.996	0.979	0.965	0.973	0.975
Best fit graph function	Linear	Cubic	Quadratic	Cubic	Quadratic	Quadratic

4 Conclusions

The incorporation of POCF tends to improve the conventional properties of base bitumen. From the analytical techniques, the POCF-MB sample's XRD pattern suggested that the structural transition is due to the addition of crystalline POCF to the amorphous bitumen matrix. In contrast, the FTIR spectra indicate extreme peaks of SI-OH asymmetric stretching vibration due to the incorporation of POCF. From the SEM, meandering patterns on the bitumen surface may result from new network structure formation after the chemical reaction between the POCF and the bitumen. Based on the conventional test results it was revealed that the addition of POCF to the base bitumen resulted in a stiffer blend with a reduced penetration value, an improved softening point and temperature susceptibility. In terms of engineering properties, it was observed that 4-6% POCF as bitumen modifier tended to improve engineering properties in terms of Marshall stability compared to conventional samples.

The statistical analysis (ANOVA and regression model) verified and showed that POCF had a significant effect on all engineering properties of the asphaltic concrete and the regression model showed the impact and significance of POCF-MB with R^2 values in the range of 95-99%. This study recommends the use of POC as bitumen modifier, which harnesses sustainability and improves the mixture's engineering properties in terms of Marshall stability.

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