

## Physical and Chemical Properties of Modified Bitumen with Oil Palm Decanter Cake

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### ABSTRACT

*The growing emphasis on sustainable development has led researchers to investigate alternative materials aimed at mitigating the environmental impact associated with conventional bitumen production. A promising strategy involves the use of waste materials from the palm oil industry, specifically Oil Palm Decanter Cake (OPDC), a biomass by-product produced in substantial quantities. OPDC is characterized by its high moisture content, organic composition, and nutrient richness, alongside its significant biodegradability, rendering it a viable candidate for bitumen modification. This study examines the feasibility of employing OPDC and polyurethane (PU) as modifiers in 60/70 penetration grade bitumen. Experimental blends were formulated with varying OPDC contents (0%, 5%, 10%, 15%, and 20% by weight of bitumen), maintaining a constant proportion of PU, to evaluate their impact on the physical and chemical properties of the modified bitumen. Physical performance was assessed through penetration, softening point, ductility, viscosity, and storage stability tests, while chemical analysis was conducted using Fourier Transform Infrared (FTIR) spectroscopy. The findings indicated that OPDC enhances the stiffness and thermal stability of bitumen, with the 10% OPDC blend exhibiting the most favourable balance of physical and chemical performance. These results suggest that OPDC, in conjunction with PU, can serve as an effective and sustainable bitumen modifier, contributing to the advancement of pavement materials and promoting the valorisation of agricultural waste.*

*Keywords:* OPDC; oil palm waste; modified bitumen; physical; chemical

### INTRODUCTION

From a life cycle assessment (LCA) perspective, the exploration of alternatives to bitumen or the enhancement of pavement service performance has gained considerable importance (Heidari et al. 2022). This represents a paradigm shift in pavement engineering, wherein the recycling of bituminous mixtures emerges as a viable

strategy to mitigate greenhouse gas emissions and protect the environment (Wu et al. 2021). In the quest to replace bitumen, both industry and academia are diligently seeking renewable materials that can satisfy the increasing demand for environmentally friendly and sustainable materials (Lin et al. 2022). Recent years have witnessed numerous studies focusing on the incorporation of biomass-derived materials as fillers in bituminous mixtures (Goulart et al. 2011). These

investigations have examined a diverse array of substances, including high-density polyethylene, marble quarry waste, building demolition debris, pulverized tire rubber, cooking oil, starch, plastic bottles, glass, brick, ceramic materials, and cigarette ends, for their potential use in asphalt concrete and bitumen (Rahman, Mohajerani, and Giustozzi 2020). It is evident that researchers in pavement engineering are increasingly considering the palm oil industry as a promising avenue for sustainability.

The palm oil industry's expansion has led to a significant increase in biomass waste production, particularly Oil Palm Decanter Cake (OPDC). This by-product, generated during the crude palm oil purification process, has garnered attention due to its unique properties and potential applications (Abdullah & Sulaiman, 2013). OPDC's high moisture content, biodegradability, and rich organic composition make it an attractive resource for various industries (Nasrin et al. 2008). The presence of cellulose, hemicellulose, and lignin in OPDC further enhances its value, as these components can be utilized in the production of biofuels, biochemicals, and other value-added products (Baharuddin et al. 2010).

The growing interest in OPDC stems from the need to address environmental concerns associated with palm oil production while simultaneously exploring sustainable alternatives for resource utilization (Mahlia et al. 2014). As the palm oil industry continues to expand, the efficient management and valorization of OPDC become increasingly crucial. Research efforts are focusing on developing innovative technologies and processes to harness the full potential of this biomass waste, aiming to create a more circular and sustainable palm oil production system (Chin et al. 2020). By transforming OPDC from a waste product into a valuable resource, the industry can potentially reduce its environmental footprint while generating additional economic benefits (Yusoff 2006).

Cellulose is the most critical component conferring structural integrity to plant sources. While both cellulose and hemicellulose contribute to the formation of natural fibers, they differ in strength due to their structural differences; cellulose possesses a crystalline chain, whereas hemicellulose is characterized by an amorphous chain (Kabir, Wang and Cardona 2012). This structural distinction renders cellulose stronger than hemicellulose. The primary constituent of cellulose and hemicellulose that contains the hydroxyl group (-OH), which imparts hydrophilic properties to natural fibers, is cellulose. Hydrophilic materials are those capable of absorbing water or moisture from their surroundings (Razak et al. 2012). Lignin, on the other hand, exhibits an amorphous structure and aromatic properties (Aziz et al. 2015). Within the natural fiber structure, lignin functions as an adhesive (Kabir, Wang and Cardona, 2012), thereby facilitating the binding of

hemicelluloses. The principal factor in the integration of cellulose, hemicellulose, and lignin is cellulose, as it imparts strength to the entire structure (Adam et al. 2019).

To further substantiate these findings, previous research has demonstrated that the OPDC (Adam et al. 2014) yields favorable outcomes when compared to other studies employing natural fibers such as jute (Siddika, Mansura, and Hasan 2013), palm fiber (Khalid et al. 2008), and EFB (Razak et al. 2012). Furthermore, Adam et al. (2015) explored various OPDC percentages in a composite material by developing an OPDC natural polymer composite (OPDC-NPC). Additionally, OPDC serves as a raw material for cellulose and polyose and is utilized as animal feed and fertilizer (Zanirun et al. 2014).

As previously noted, while prior research has explored the use of OPDC as biomass by-products, there is a lack of studies investigating its application in road pavement construction. This study designs and conducts a comprehensive experiment to assess the feasibility of utilizing OPDC and PU as bitumen modifiers. The polymerization of 2,4-diphenylmethane diisocyanate (MDI) and palm kernel oil-based polyol (PKO-p) was undertaken to synthesize PU. PKO-p is a polyurethane polyol derived from the esterification of palm kernel oil (PKO), a sustainable resource (Wong and Badri 2012). PU serves as an additive to bitumen. By employing appropriate formulations and components, bitumen can be modified to enhance its mechanical properties (Izquierdo et al. 2012).

This research primarily aims to assess the efficacy of bitumen modified with OPDC by examining its physical, chemical, storage stability, and thermal characteristics. The study involves blending bitumen with varying proportions of OPDC (0%, 5%, 10%, 15%, and 20%), alongside PKO-p and 2,4-MDI. This study employs a range of physical assessments, including tests for penetration, softening point, viscosity, ductility, and storage stability. Additionally, the chemical analysis utilises FTIR testing to determine the composition of chemical compounds.

## EXPERIMENTAL DESIGN

### MATERIALS AND SAMPLE PREPARATION

The primary binder employed in this investigation was 60/70 penetration grade bitumen. The oil palm decanter cake (OPDC) was procured from the Sime Darby Oil Palm Mill located in Sua Betong, Negeri Sembilan, Malaysia. Fresh OPDC underwent oven drying at 135°C for 24 hours to eliminate moisture, followed by grinding and sieving to attain a particle size of 300µm. The processed OPDC was integrated into the base binder at weight percentages of 5%, 10%, 15%, and 20%. To formulate the OPDC-modified

bitumen, 20% polyurethane (PU) was incorporated, consisting of a mixture of palm-based polyol (PKO-p) and a chemical hardener (2,4-Methylene diphenyl diisocyanate, 2,4-MDI), combined in a 100:60 ratio. Additionally, a separate blend of PEN 60/70 and PU was prepared to function as a secondary control binder, designated as OPDC 0 (0%), alongside the base binder. The base binder was heated to 165°C, after which OPDC was introduced into the bitumen. The bitumen mixture was agitated at 2000 rpm for 15 minutes utilizing an IKA T 25 digital ultra-TURRAX high shear mixer.

## OPDC PROPERTIES

FTIR test was done to identify the organic and inorganic compounds present in the OPDC sample. The FTIR spectra are shown in Figure 1. The FTIR test conducted on the OPDC sample provides valuable insights into its chemical composition by measuring the absorption of infrared radiation at different wavelengths. The resulting FTIR spectra reveal characteristic peaks and bands corresponding to specific functional groups and molecular structures, allowing for the identification of organic and inorganic compounds present in the sample.

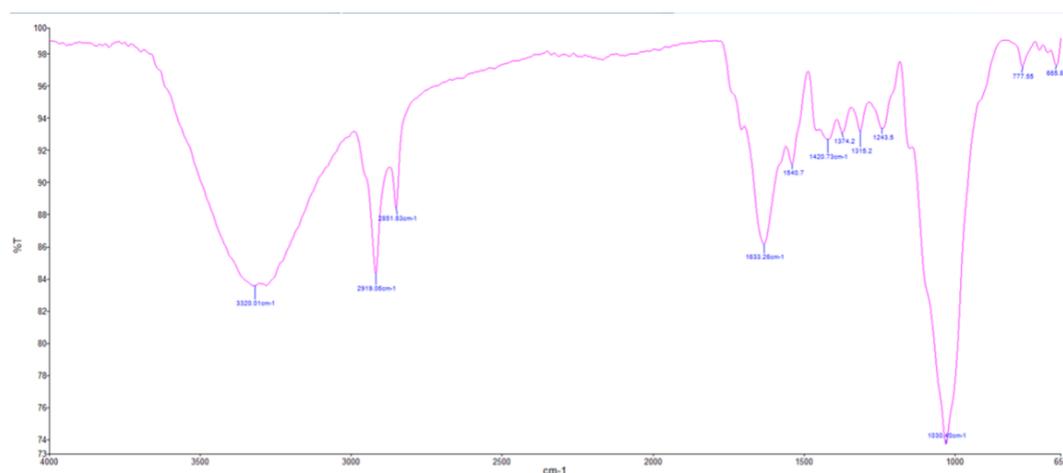


FIGURE 1. FTIR graph analysis on raw OPDC sample

According to the interpretative framework by Nandiyanto et al. (2017), significant alterations in the FTIR peaks and patterns of OPDC were detected at temperatures exceeding 180 °C. These spectral changes suggest structural modifications, indicating a thermal limit that impacts the stability of OPDC. By comparing FTIR spectra

with earlier research (Coates, 2000; Nandiyanto et al. 2017), the findings are validated, and distinctive functional groups are identified. This analysis is crucial for comprehending the molecular behavior of OPDC and enhancing its use across various industries.

TABLE 1. Breakdown of FTIR spectra of OPDC

Band Number	Wavenumber (cm-3)	Functional Group	Nutrient Types
1	3320.01	O-H	
2	2919.05	CH2 and CH3, and C=O	Cellulose
3	2851.63	ester	Hemicellulose
4	1633.26		Lignin
5	1540.70		Lipids
6	1420.73		Phospholipids
7	1374.20	-	-
8	1315.20		
9	1243.50		
10	777.55		
11	665.81	CH1 and CH3	Lipids

The structural components of oil palm decanter cake (OPDC)—cellulose, hemicellulose, and lignin—offer promising potential as bitumen binder modifiers due to their unique properties and interactions. Cellulose, with its crystalline chain structure, provides strength and stability (Rowell, 2012), while hemicellulose, characterized by an amorphous chain, offers flexibility (Kumar et al. 2008). Lignin, acting as a natural binding agent, not only connects the other components but also shares chemical similarities with bitumen, specifically aromatic carbon rings (Laurichesse & Avérous, 2014). This chemical resemblance suggests that lignin could potentially serve as a partial substitute for bitumen in certain applications (Khosravani & Tehrani, 2021). The combination of these three structural components is expected to enhance the adhesive properties between the bitumen binder and aggregates, potentially improving the overall performance of asphalt mixtures (Xiao et al. 2010).

The cementitious nature of lignin, coupled with its aromatic properties, may contribute to better cohesion within the binder and improved adhesion to aggregates (Liu et al. 2014). Additionally, the varying strengths and structural characteristics of cellulose and hemicellulose could provide a balanced reinforcement effect, potentially enhancing the durability and mechanical properties of the modified bitumen binder (Yusoff et al. 2014). This synergistic interaction between OPDC components and bitumen opens up new possibilities for developing more sustainable and effective road construction materials (Rahman et al. 2020).

## LABORATORY TESTINGS

### PHYSICAL PROPERTIES TEST

The assessment of the physical properties of OPDC-modified bitumen was conducted following the guidelines established by the American Society for Testing and Materials (ASTM). The suite of physical tests administered comprised the penetration point test (ASTM D5), the softening point test (ASTM D36), viscosity measurements at 135°C and 165°C (ASTM D113), the ductility test (ASTM D4402), and the storage stability test (ASTM D7173).

### STORAGE STABILITY TEST

The evaluation of the storage stability of modified bitumen was conducted by analysing the softening point values of its upper and lower segments. A cylindrical aluminium container, with dimensions of 25 mm in diameter and 300 mm in length, was utilized to hold approximately 50 g of the sample. This container was then subjected to a controlled temperature of  $135 \pm 5$  °C in an oven for a duration of 48 hours. After this heating process, the top and bottom sections of the aluminium cylinder were carefully removed. To ensure consistent mixing, it is crucial that the temperature difference between the top and bottom sections does not exceed 2.5 °C. This finding suggests that the modified bitumen demonstrates sufficient stability for storage at elevated temperatures.

### FOURIER TRANSFORM INFRARED (FTIR) TEST

For the chemical analysis of OPDC-modified bitumen, Fourier Transform Infrared (FTIR) spectroscopy is utilized due to its efficiency in identifying molecular structures and functional groups. The analysis is carried out using a Perkin Elmer-1650 spectrometer, which detects the presence of chemical functional groups within the wavenumber range of 600 to 4000  $\text{cm}^{-1}$ . A small, heated droplet of bitumen is deposited onto the sample holder to create a thin film suitable for spectral analysis. The spectrum is obtained via a Zinc Selenide (ZnSe) platform using the Attenuated Total Reflectance (ATR) technique, which facilitates direct surface measurement without complex sample preparation. This method is highly effective for assessing bitumen modification due to its sensitivity to molecular bonding changes (Smith, 2011; Griffiths & de Haseth, 2007). After preparation, samples are stored in a covered dish in a cool, dark location until analysis, in accordance with ASTM E1252-98 guidelines (ASTM, 1998).

## RESULTS AND DISCUSSION

### PHYSICAL PROPERTIES ANALYSIS

To assess the physical properties of OPDC-modified bitumen, all testing was conducted in accordance with the standards set by the American Society for Testing and Materials (ASTM). The findings related to the physical properties of the OPDC-modified bitumen are detailed in Table 2.

TABLE 2. Physical properties of OPDC modified bitumen

Properties	Unit	Value					Standard Test Method	Standard Test Method Requirement
		PEN 60/70	OPDC 1 (5%)	OPDC 2 (10%)	OPDC 3 (15%)	OPDC 4 (20%)		
Penetration at °C	0.1 mm	66.00	16.64	30.68	34.54	36.94	ASTM D5	60-70
Softening Point	°C	49.00	48.20	49.00	49.40	54.40	ASTM D36	47-52
Viscosity at 135 °C	mPa.s	0.55	0.88	1.38	1.54	1.93	ASTM D4402	3.0 Pa.s
Ductility at 25 °C	cm	120	115	105	101	100	ASTM D4402	>100

Table 2 illustrates that the OPDC-modified bitumen meets the requirements for viscosity, ductility, and softening point, with the exception of OPDC 1. This exception is deemed irrelevant as no OPDC is incorporated, serving instead as a secondary control bitumen. However, the penetration results, characterized by uniformly low values, suggest that the OPDC-modified bitumen exhibits excessive stiffness. This stiffness is attributed to the incorporation of 20% PU into the bitumen blend. The selection of a 20% PU concentration was determined through extensive trial and error, identifying it as the optimal percentage to address the high residual content in OPDC. Furthermore, the fibrous composition of OPDC—comprising cellulose, hemicellulose, and lignin—contributes to the reinforcement of the bitumen matrix, which is reflected in the enhanced softening and ductility results as the OPDC percentage increases from 5% to 20%.

#### STORAGE STABILITY ANALYSIS

A storage stability test was conducted to confirm the uniform blending and stability of the modified bitumen during storage. Figure 3 presents the differences in softening points between the top and bottom sections for 0%, 5%, 10%, 15%, and 20% OPDC. Evaluating the stability of the modified bitumen is a crucial part of this process.

As seen from Figure 2, the control binder along with OPDC 0, OPDC 1 and OPDC obtain the differences of less than 2.5 °C while OPDC 3 and OPDC 4 obtain more than 2.5 °C differences. OPDC 3 and OPDC 4 do not achieve the desired stability due to the overwhelming content of OPDC. Based on the OPDC's chemical properties, the OPDC has residual oil in it which makes it harder for the bitumen blending process to achieve homogeneity.

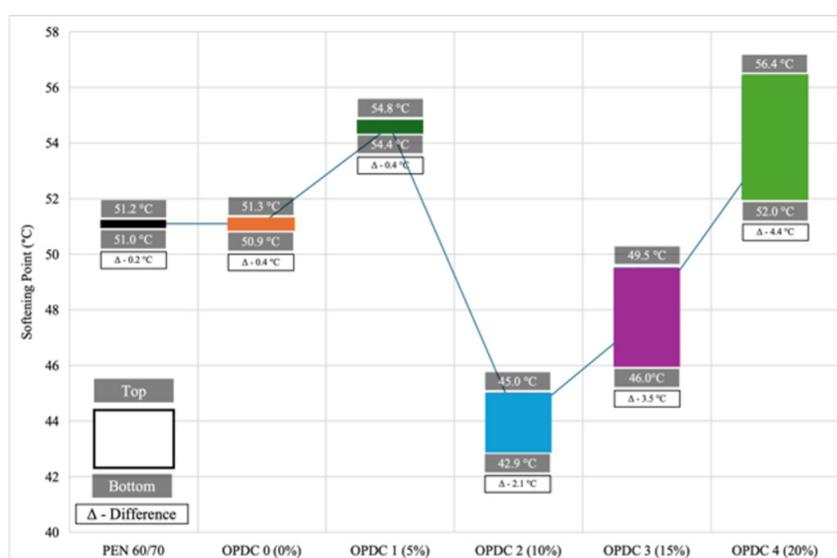


FIGURE 2. Storage stability of base binder and OPDC modified bitumen

The blue line depicts the trend as increasing amounts of OPDC are integrated into the modified bitumen. Initially, there is a slight 0.2 increase in the difference between PEN

60/70 and OPDC 0 (0%). With the introduction of OPDC, a notable rise in softening is evident at OPDC 1 (5%), which is followed by a pronounced decline at OPDC 2

(5%), and then a steady increase up to OPDC 4 (20%). This pattern suggests that OPDC significantly affects the storage stability of the modified bitumen. As a result, it is clear that only the blends OPDC 0, OPDC 1, and OPDC 2 maintain stability during storage. Thus, the ideal OPDC concentration for bitumen blending should be kept below 10%.

### CHEMICAL PROPERTIES ANALYSIS

The FTIR spectrum of OPDC-modified bitumen at concentrations of 0%, 5%, 10%, 15%, and 20% is depicted in Figure 4, with the corresponding functional groups detailed in Table 2. As indicated in Table 2, a similar pattern of stretching is observed in OPDC 1, OPDC 2, OPDC 3, and OPDC 4, in contrast to OPDC 0. The bending

vibrations of NH and the aromatic branched chain produce absorption peaks at specific wavelengths, indicating the presence of an aromatic secondary amine group within the material. The highest peaks for OPDC 0, OPDC 1, OPDC 2, OPDC 3, and OPDC 4 are recorded at 3699 cm<sup>-1</sup>, 3772 cm<sup>-1</sup>, 3919 cm<sup>-1</sup>, 3953 cm<sup>-1</sup>, and 3977 cm<sup>-1</sup>, respectively. From the individual graphs in Figure 5(a), (b), (c), (d), and (e), it is evident that OPDC 3 and OPDC 4 exhibit more pronounced bending vibrations compared to OPDC 1 and OPDC 2, whereas the shortest bending vibration observed in the fingerprint region is associated with OPDC 0.

Figure 2 shows the normalized spectra of OPDC modified bitumen with absolute baseline and integration of areas for index production in favour over peak based methods.

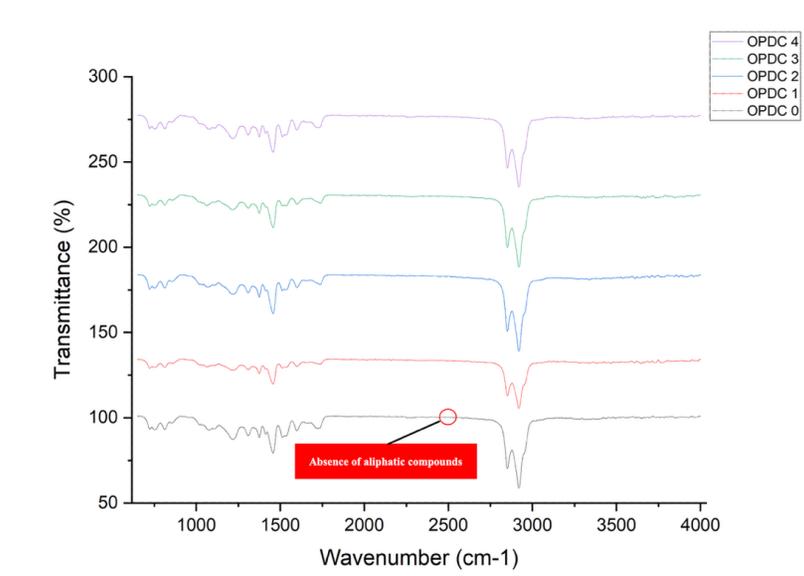


FIGURE 2. Storage stability of base binder and OPDC modified bitumen

TABLE 3. FTIR peaks identified in OPDC modified bitumen

Wavenumber (cm-1)	Functional Group (Coates 2010)	OPDC Modified Bitumen				
		OPDC 0	OPDC 1	OPDC 2	OPDC 3	OPDC 4
3600-3645	Non-bonded hydroxy group, OH stretch	✓	✓	✓	✓	✓
3300-3500	Aromatic secondary amine, N-H stretch	✓	✓	✓	✓	✓
2860-2935	Aliphatic compounds, -CHO stretching vibration	-	✓	✓	✓	✓
2845-2935	Methylene-CH <sub>2</sub> - asymmetric and symmetric stretch	✓	✓	✓	✓	✓
1300-1610	Carboxylate group, RCOO- stretch	✓	✓	✓	✓	✓
1630-1680	Amide group, -NH <sub>2</sub> conjugated with carbonyl group, C=O stretch	-	-	-	-	-
1310-1360	Aromatic tertiary amine, CN stretch	✓	✓	✓	✓	✓
670-900	Aromatic C-H out-of-plane bend	✓	✓	✓	✓	✓
700-735	Aromatic ring, C-H 1,2-Disubstitution (ortho)	✓	✓	✓	✓	✓
800-860	Aromatic ring, C-H 1,2-Disubstitution (para)	✓	✓	✓	✓	✓

The analysis reveals a slight distinction in the chemical functional groups between OPDC 0 and the other OPDC-modified bitumen samples (OPDC 1, OPDC 2, OPDC 3, OPDC 4). Specifically, OPDC 0 is unique among these samples in that it lacks aliphatic compounds, as evidenced by the absence of the  $-CHO$  stretching vibration. This vibration is present in OPDC 1-4, as illustrated in red in Figure 2. Thus, it can be concluded that OPDC exhibits strong compatibility with bitumen PEN 60/70, PKO-p, and '2,4-MDI.

## CONCLUSION

This study has investigated the potential of palm oil waste, specifically oil palm decanter cake (OPDC) and polyurethane (PU), in bitumen at varying percentages, focusing on both physical and chemical properties. The physical properties of OPDC-modified bitumen improve as the OPDC percentage increases from 5% to 20%, attributed to the natural chemical components in OPDC, namely cellulose, hemicellulose, and lignin, which provide structural support. Regarding storage stability, only the blends OPDC 0, OPDC 1, and OPDC 2 demonstrate stability. The optimal OPDC percentage for bitumen blending is determined to be 10%, corresponding to OPDC 2. In terms of chemical properties, OPDC-modified bitumen (5% - 20%) is considered stable due to the presence of numerous aromatic compounds identified through FTIR spectra. These aromatic compounds maintain stability through the presence of conjugated  $\pi$ -bonds. Based on the conclusions drawn from its physical and chemical performance, 10% OPDC is identified as the optimal percentage for blending, exhibiting superior performance in both physical and chemical aspects, thereby indicating that OPDC can serve as an effective modifier.

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## DECLARATION OF COMPETING INTEREST

None.

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