INTERNATIONAL JOURNAL OF INTEGRATED ENGINEERING
ISSN: 2229-838X e-ISSN: 2600-7916

IJIE

Vol. 17 No. 3 (2025) 29-37
https://publisher.uthm.edu.my/ojs/index.php/ijie

Investigating the Impact of Ageing on the Morphological Characteristics of Rejuvenated Asphalt Binder

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DOI: https://doi.org/10.30880/ijie.2025.17.03.004

Article Info

Received: 21 January 2025
Accepted: 30 June 2025
Available online: 29 August 2025

Keywords

Asphalt ageing, waste cooking oil, atomic force microscope, contact angle, morphological, reclaimed asphalt pavement, rejuvenators

Abstract

The performance and longevity of asphalt pavement are greatly impacted by ageing. However, after several years of service, asphalt pavement becomes harder and loses its flexibility, impact comfort and safety for road users. To overcome these problems, asphalt pavement requires effective maintenance or repair. The utilisation of rejuvenators in aged asphalt is one of the popular rehabilitation techniques. The aim of this study is to discover the impact of ageing and rejuvenation, waste cooking oil (WCO), on the asphalt binder's morphological changes by utilising contact angle and Atomic Force Microscopy (AFM) analysis. To evaluate the morphological changes in asphalt binder, this study employed a Sessile drop method and tapping mode. Asphalt binders were also subjected to short-term and long-term ageing simulations. The result shows WCO effectively increase the Surface Free Energy (SFE) value and reduces the surface roughness of asphalt binder. The result also illustrates that as the ageing duration increases, the contact angle tangent slope between asphalt binder and test liquids increases, indicating a reduction in SFE value of asphalt binder. From AFM images, the size of the bee structure or the catana phase increases as the ageing duration increases, indicating a morphological change and an increase in surface roughness. This phenomenon can be attributed to changes in the asphaltenes content of asphalt binder due to the ageing and rejuvenation process. These findings will provide important information about the effect of ageing and the rejuvenation process on the asphalt binder's surface morphology.

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1. Introduction

One of the important materials in asphalt pavement construction is asphalt binder. After a long time in service, asphalt binder undergoes an ageing process, leading to hardening and loss of its flexibility. The aged asphalt mixture is usually dumped in a landfill, which has raised environmental concerns. The latest studies have proved that aged asphalt can be recycled by using a rejuvenation technique. The rejuvenation technique and ageing process not only affect the basic properties of asphalt binder, but also affect the morphological structure of asphalt binder. This property is closely related to the asphalt binder‘s moisture resistance. Therefore, understanding the alteration in morphological structure in asphalt binder due to rejuvenation and ageing processes is important for developing an effective maintenance solution. The morphological structure in asphalt binder can be evaluated using contact angle and Atomic Force Microscopy (AFM).

The sum of external work done or energy between two substances to create a unit surface area and intermolecular forces is known as the SFE value [1]. It can be attributed to the cohesion and adhesion properties between two materials that hold them together. The SFE value can be calculated from the contact angle test. Generally, there are three methods for measuring the contact angle, which are the Wilhelmy balance or plate, the Washburn capillary rise method, and the Sessile Drop test [2]. The Sessile Drop test is the easiest and most common method used to determine the SFE value of asphalt binder. Aguiar-Moya et al. conducted a Sessile drop test on aged asphalt binder and found that the adhesion properties of the aggregate and asphalt binder increased after ageing simulation. However, Firmansyah & Tamalkhani [3] found that the asphalt binder’s adhesion property was reduced after 6 months and showed an increasing trend after 1 year. Kakar et al. [4] conducted a contact angle test using the Wilhelmy plate method and found that the integration of asphalt binder and limestone had greater moisture resistance compared to the integration of asphalt binder and granite. Furthermore, it is worth noting that the temperature has a notable impact on the contact angle of the asphalt binder between aggregates. This is because the temperature supplies the energy in the asphalt binder, allowing it to spread over the aggregates [5]. Shu et al. [6] found that the binder with a higher SFE value is more capable of self-healing. This is because the healing agent or rejuvenator is easier to adhere to the asphalt surface.

Over the years, AFM has proven to be a promising tool for evaluating the asphalt binder’s microscopic characteristics. It can give additional information such as the microstructural phase distribution and the material homogeneity. Recently, several studies have been carried out to study the influence of ageing on the surface morphology of asphalt binder. A study conducted by Samsudin et al. [7] revealed that the modified asphalt binder’s surface becomes rougher and the asphalt binder becomes more solid after ageing. The same finding was found by Abdelaziz et al. [8]. It was found that the binder with a longer ageing duration exhibits a more bee-like structure. This is because during ageing simulation, the oxidation reaction happens and increases the asphalt binder’s asphaltene content [9]. However, a recent research carried out by Koyun et al. [10] found that after short-term ageing simulation, the asphalt binder’s surface roughness illustrates no linear dependency with the ageing level. The asphalt binder’s rejuvenation process illustrates the opposite trend [11]. A research conducted by Ahmed et al. [12] found that as the percentage of WCO increased, the number of bee structures in the asphalt binder was reduced, and the size of the bee structures was enhanced. A similar finding was found by Rafiq et al. [13], where the bee structure was affected due to rejuvenation using Crude Palm Oil (CPO) and after ageing. It also needs to be mentioned that the rejuvenation of asphalt binder does not always reproduce the microstructure of the asphalt binder [14]. Furthermore, research conducted by Azahar et al. [15] illustrates that the asphalt binder containing treated Waste Cooking Oil (WCO) has the lowest surface roughness, which enhances the asphalt binder’s adhesion properties as well. This can be concluded that the transesterification of WCO can enhance the rejuvenation process of aged asphalt binder.

In this research, contact angle and AFM were utilised to evaluate the effect of ageing on rejuvenated reclaimed asphalt binder in cooperation with WCO. The aims of this research are to further explore the relationship between the effect of ageing and rejuvenation on asphalt binder surface morphology and characteristics. Understanding these mechanisms helps to improve the performance of rejuvenated asphalt pavement, incorporating WCO, and aids in the development of sustainable road construction

2. Materials and Methods

2.1 Materials

Virgin asphalt binder with penetration grade 60/70 (PEN 60/7) was employed as the control asphalt binder in this study. The binder was supplied by Asphalt Viking Company. The control asphalt binder showed a penetration of 66.5 dmm at ambient temperature and a softening point value of 52 °C. The Reclaimed Asphalt Pavement (RAP) was taken during the milling process at Johor Bahru. To separate the aged binder and aggregate, methylene chloride was used as a solvent in this study. At ambient temperature, RAP’s binder had a penetration of 19 dmm and a softening point value equal to 57.5 °C. Treated WCO from the transesterification process were used as a rejuvenator in this study. From the earlier stage of the study, the optimum WCO dosage for rejuvenation was found 3% by mass of the binder.

2.2 Binder Rejuvenation

RAP binder and virgin asphalt binder were initially heated up to 130°C in the oven to make the asphalt binder liquid and pourable. Then, virgin asphalt binder was mixed with RAP binder at a proportion of 30% RAP and 70% virgin binder. The aged binder was then mixed with 3 % of treated WCO based on the weight of the asphalt binder. The hot binder was then poured into a cylindrical container and positioned on a hot plate. During the mixing, the temperature of the hot plate was adjusted to 130°C to sustain the asphalt binder's viscosity. The temperature was carefully controlled to ensure it did not exceed 160°C, aiming to minimise the ageing effect on asphalt binder. During mixing of the virgin asphalt and RAP binder, treated WCO was gradually added to the hot asphalt binder to ensure homogeneity. The binder was mixed at a 200 rpm rotational speed for a continuous 20 min. In this study, three types of binder were undergoing ageing simulation and referred to as VA (Virgin asphalt), R30 (30% RAP and 70% VA), and WCO3 (30% RAP, 70% VA and 3% treated WCO).

2.3 Binder Ageing

The rejuvenated asphalt binders were initially simulated under short-term ageing before undergoing a long-term ageing simulation. A Rolling Thin Film Oven (RTFO) was used to simulate a short-term ageing as per BS EN 12607 [16]. The binders were poured on the RTFO bottles and were spun for 75 minutes at a temperature of 163°C continuously with an airflow of 4000 millilitres per minute. Then, 50 g of aged binder was poured into the cylindrical pan. Pressure Ageing Vessel (PAV) was utilised to simulate long-term ageing as per ASTM D 6521 [17]. The asphalt binder was simulated for 20, 40, and 60 hours at 100°C with 2.1 MPa air pressure. According to Luo et al. [18], a 20-hour PAV period is equivalent to 5-6 years of asphalt binder in service.

2.4 Sample Preparation

Before testing the asphalt binder's surface morphology, its surface must be ensured smooth and glossy. The asphalt binders were initially heated at 130°C in the oven until melted and poured onto a microscope slide in a 1 cm droplet formation. The microscope slide was heated in the oven at 130°C for 2 minutes. The sample was stored and cooled at ambient temperature for a day in a sealed case to avoid any dust and kept in a horizontal position.

2.5 Contact Angle Test

The contact angle test was employed to assess the SFE value and wettability of liquid on the asphalt binder. To evaluate these properties, the Sessile drop method was utilised in this study. This method determines the asphalt binder's contact angle by measuring the drop shape of liquid on the flat surface of the asphalt binder by using an optical camera (see Fig. 1). The liquids used in this test were distilled water and toluene. Table 1 illustrates the surface tension values of both test liquids [19], [12]. The test was conducted by dropping 1 μL of liquid at ambient temperature. The tangent slope of the asphalt surface and the water droplet was taken after 2s. Five pictures were saved from each group to maintain the test accuracy.

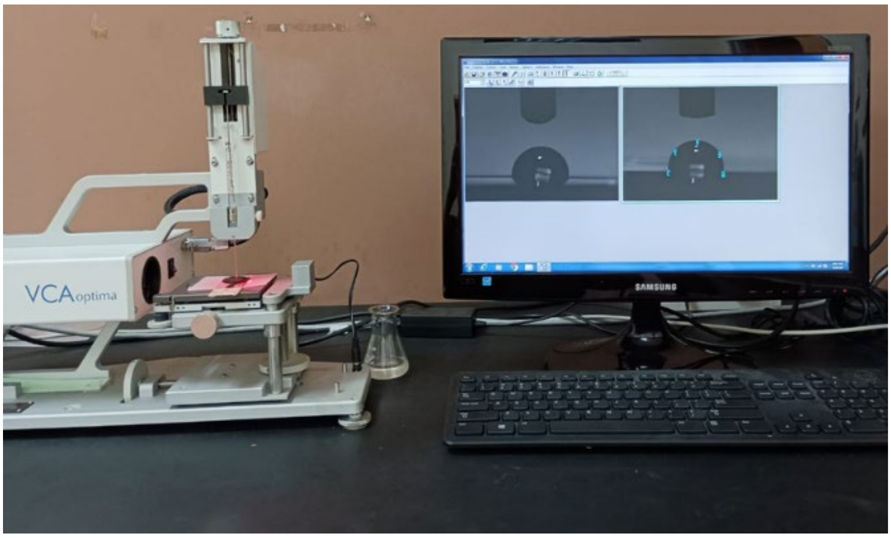


Fig. 1 *Contact angle device*

Table 1 *Surface tension value of both liquids*

Liquid	Surface Tension/ (mJm^{-2})		
	γ	γ^d	γ^P
Distilled Water	72.8	21.8	51.0
Toluene	28.5	28.5	0

The asphalt binder SFE value was determined by using the Owens, Wendt, Rabel, and Kaelble (OWRK) method. When the test liquid is dropped on the asphalt surface, the contact angle (θ) depends on three SFE values, which are the SFE value of the solid (γ_{sv}), the SFE value of the liquid (γ_{lv}) and the interfacial SFE value between the liquid and the solid (γ_{sl}). The Young's equation [20] can help to describe the interfacial free energy (γ_{sl}) at equilibrium as follows:

$$\gamma_{sv} = \gamma_{sl} + \gamma_{lv}\cos(\theta) \tag{1}$$

Based on Fowkes theory, the SFE value of solid and liquid can be express in two component which is dispersion a Lifshitz-van der Waals force (γ^d) and Polar (γ^p) which is acid-base force. Owens & Wendt [21] expand Fowkes's by including the geometric mean expression of SFE and establish the following equation [22]:

$$\gamma_{sv} = \gamma_{sl} + \gamma_{lv} - 2\sqrt{\gamma_{sv}{}^{d}\gamma_{lv}{}^{d}} - 2\sqrt{\gamma_{sv}{}^{p}\gamma_{lv}{}^{p}} \tag{2}$$

By combining Eq. (1) and Eq. (2), the SFE value of the asphalt binder can be calculated as below:

$$\frac{\gamma_{lv(1-\cos(\theta))}}{2} = \sqrt{\gamma_{sv}{}^{d}\gamma_{lv}{}^{d}} - \sqrt{\gamma_{sv}{}^{p}\gamma_{lv}{}^{p}} \tag{3}$$

2.6 Atomic Force Microscope (AFM)

AFM is a powerful device that can be utilised to assess the surface morphology, microstructure, and microchemical properties and characteristics of the asphalt binder. In this study, the intermittent-contact mode, or tapping mode, was selected because the binder used in the experiment was adhesive, and using this mode helped prevent the tip from sticking to the asphalt binder surface. Additionally, the intermittent-contact mode was chosen because it provided better image quality compared to the contact mode. In this test, the AFM employed a 300-kHz operating frequency and a scan rate of 0.5 Hz under regular conditions of room temperature and atmospheric pressure to evaluate the surface morphology of the asphalt. In order to examine the microstructure of asphalt, the AFM image was acquired at a scale of 20 × 20 µm. The quantitative microstructures of asphalt binder were identified through surface roughness analysis using the JPK data processing software in this study. Surface roughness was assessed using the arithmetic average height (Ra) parameter, which can be described by the following equation [8]:

$$R_a = \frac{1}{t}\int_0^l \left|y(x)\right| dx \tag{4}$$

where *y(x)* is the function of the asphalt binder's profile height, and *l* is the length evaluation.

3. Results and Discussion

3.1 Ageing Effect on Contact Angle and SFE Value

Fig. 2 illustrates the average tangent slope for the asphalt sample using distilled water. Fig. 3 illustrates the average tangent slope for the asphalt sample using toluene. It is also known that the contact angle for all asphalt binders is more than 90° for distilled water. This indicates that the asphalt binder is hydrophobic, which means the asphalt binder cannot be wet. This property is critical in the road construction industry, as moisture damage stands out as one of the main factors contributing to asphalt degradation. The average contact angle for the asphalt binder sample using toluene is between 30.97° and 12.97°. This result illustrates that toluene is partially wetting on the asphalt binder surface. Notably, the asphalt binder's contact angle was enhanced as the ageing duration increased for both liquid tests. According to Hossain et al. [23], this phenomenon is due to the complex molecular changes and interaction of the asphalt binder and the test liquid. It can also be included that the hydrophobicity of the asphalt binder is increased after ageing. It was noted that R30-unaged had the highest contact angle

(103.20°), followed by WCO3-unaged (101.94°) and VA-unaged (97.18°) when using distilled water and toluene. However, after a 60-hour ageing simulation, it was noted that WCO3-60 had the highest average contact angle (112.12°) when using distilled water, followed by R30-60 (110.74°) and VA-60 (110.44°). This illustrates that the average contact angle for rejuvenated aged asphalt binder is more affected than that of the virgin asphalt binder.

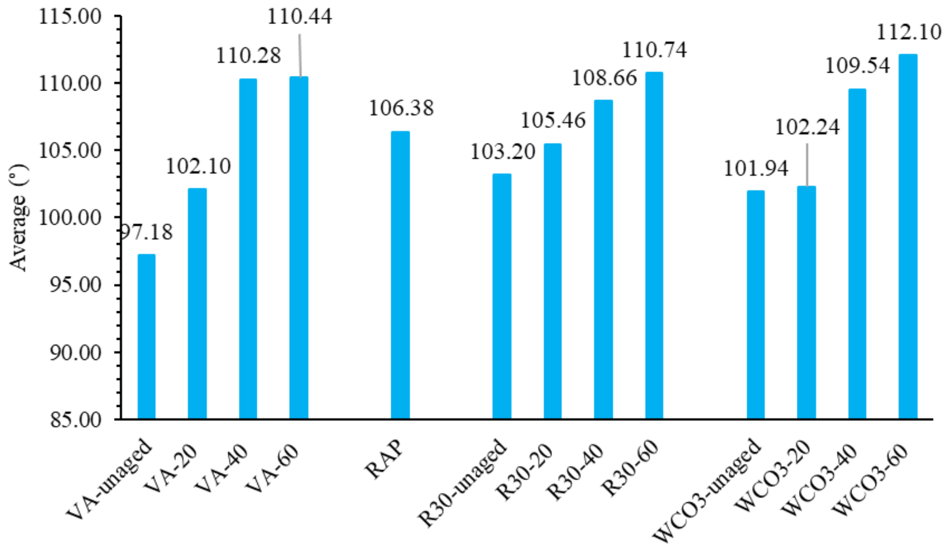


Fig. 2 *Average contact angle for distilled water*

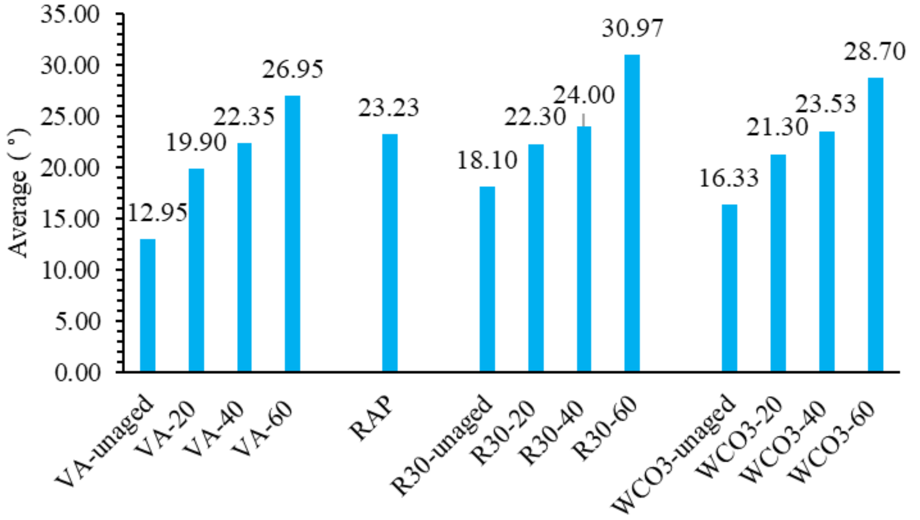


Fig. 3 *Average contact angle for Toluene*

The SFE value is essential in determining the adhesion characteristics between an asphalt binder and aggregate [20]. When the SFE value of asphalt binder is high and the surface tension of the liquid is low, the liquid will efficiently adhere to the asphalt binder's surface. Fig. 4 illustrates the SFE value for asphalt binder. Overall, the VA binder was found to have the highest SFE value among all binders, followed by WCO3 and R30. The result shows that the SFE value of asphalt binders was reduced as the ageing duration was enhanced. This is because the increase in asphaltenes content in asphalt binder leads to a decrease in SFE value [24]. These results were similar to the findings of Hossain et al. [23], who found that the SFE value of asphalt binder reduces after ageing. However, as the SFE value of asphalt binder decreases, the cohesion work decreases, making the binder more susceptible to cracking [20]. This can be linked to the alterations in the surface roughness of the asphalt binder that occur during the ageing process. At unaged condition and 20 hours of PAV, WCO3-20 had the highest SFE value compared to R30-20. This result illustrates that WCO3 had higher cracking resistance compared to R30. A similar result was found by Ahmet et al. [12], who found that WCO effectively improved the asphalt binder's cracking resistance.

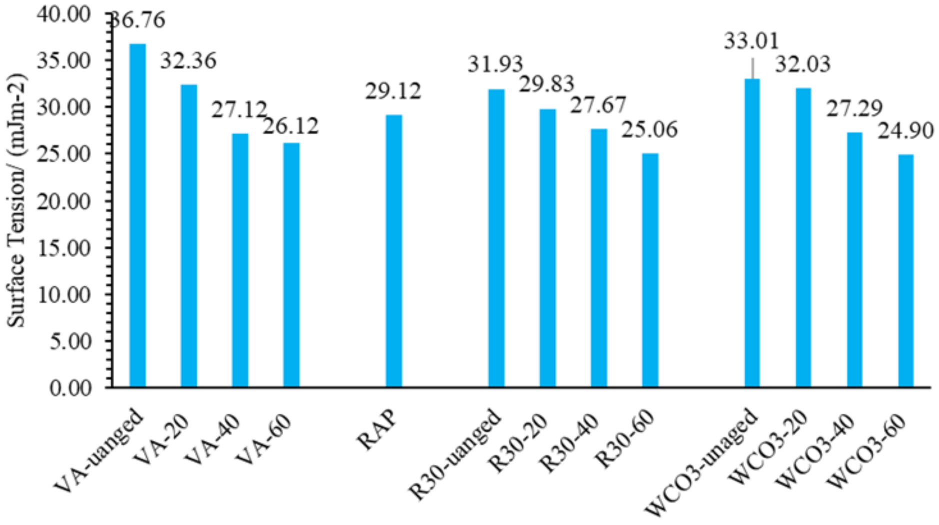
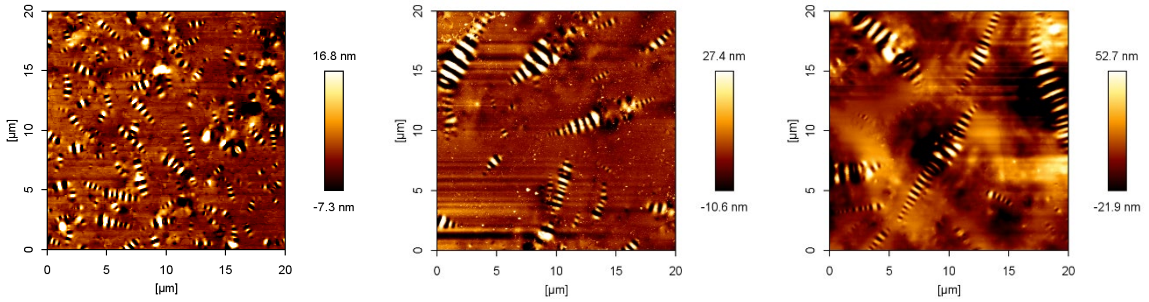


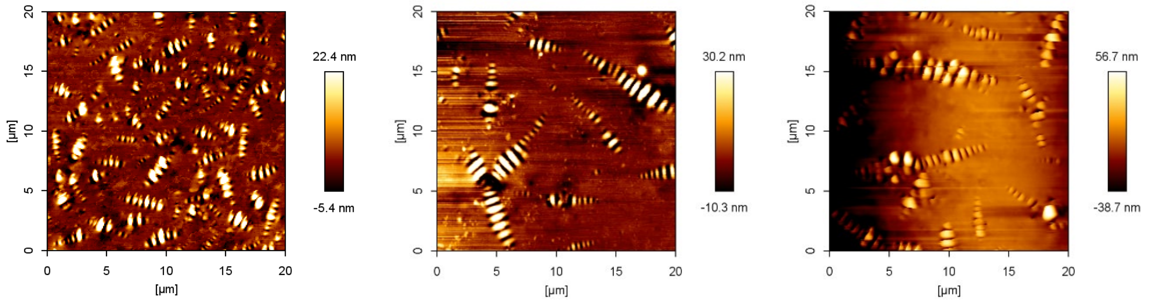
Fig. 4 *Total SFE value for asphalt binder*

3.2 Ageing Effect on Surface Morphology

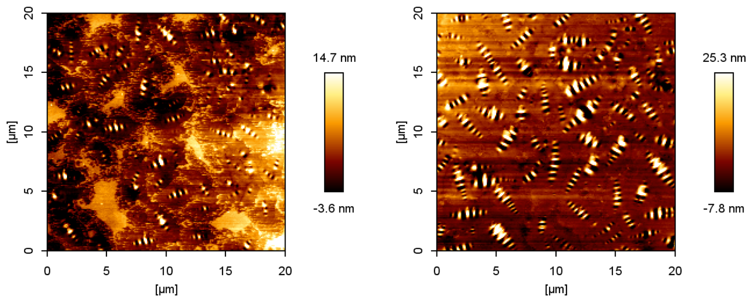
The AFM images were utilised to determine the surface morphology and roughness of the asphalt binder. Fig. 5 illustrates the AFM images of asphalt binders. Notably, three typical phases are found in all AFM images, which are catana, peri, and para phases. According to Samsudin et al. [7], the catana phase can be noted from the white and dark line, which indicates the topographic line (bee-like structure). Peri phase is the area surrounding the catana phase, while para phase is the adjacent area to peri phase. Notably, the catana phase becomes bigger or elongated as the asphalt binder ages. In the unaged condition, the VA-unaged binder was found to have the lowest height of the bee structure compared to WCO3-unaged and R30-unaged. As the binder aged, the height of the bee structure also increased. This is due to the increase in asphaltene content in the asphalt binder due to oxidative ageing. According to Wang et al. [25], the catana phase or bee structure is linked to the asphaltene contents in the asphalt binder. It also noted that after ageing simulation, WCO3 remained had a higher bee structure compared to VA binders. However, among all asphalt binders, the RAP binder exhibits the smallest bee structure and the lowest height of topography. This may be due to the formation of a non-crystalline product that creates a thin surface film on the asphalt surface, which is induced by UV light and oxygen during service time, as reported by Das et al. [26].



VA-unaged *VA-20* *VA-60*



WCO3-unaged *WCO3-20* *WCO3-60*



RAP *R30-unaged*

Fig. 5 *AFM images for asphalt binders*

Fig. 6 illustrates the surface roughness of asphalt binders. It can be noticed that the value ranges from 2.537 to 12.63 nm. Notably, the values are increased as the ageing duration increases. Similar results were indicated by Abdelaziz et al. [8], who also identified that as the asphalt binder aged, the surface roughness of the asphalt binder also increased. On the contrary, the introduction of WCO into the asphalt binder reduced the size of the catana phase, consequently decreasing the surface roughness of the asphalt binder. Asphalt binder's surface roughness characteristics are related to the adhesion properties and the asphalt binder's self-healing properties. In theory, the presence of high surface roughness is predicted to decrease the adhesive performance of the asphalt binder during the coating process with the aggregate [15]. Therefore, as the asphalt ages, the adhesive performance of the asphalt binder with aggregate will decrease. Hence, it leads to cracking in the asphalt mixture. Additionally, it should be noted that WCO possesses the capability to decrease the roughness of the asphalt binder's surface, hence improving its coating ability. It was noted that, at unaged condition, the highest surface roughness was R30-unaged, followed by VA-unaged and WCO-unaged. Indicate that introducing the WCO in aged binder will improve the asphalt binder's cracking resistance. However, as the asphalt binder aged, the rejuvenated asphalt binder was found to have higher surface roughness compared to the virgin binder.

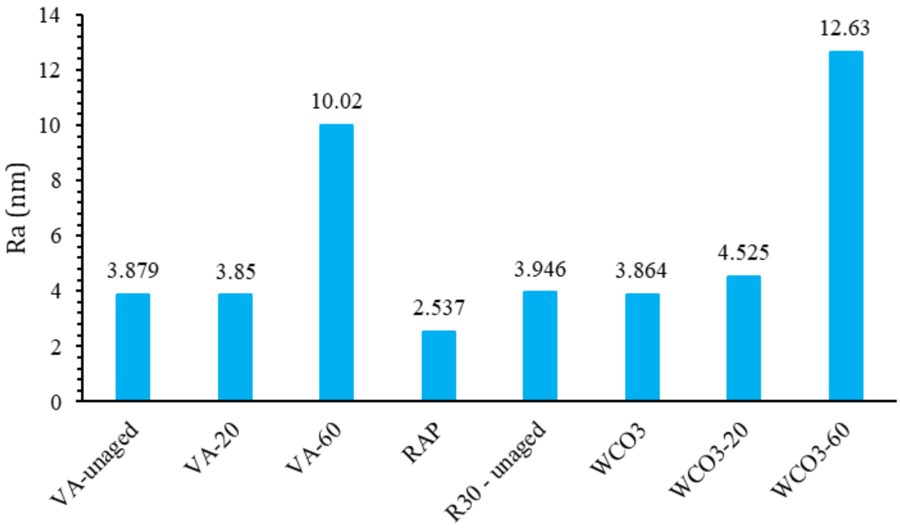


Fig. 6 *Asphalt binder's surface roughness value*

4. Conclusion

The effect of ageing and rejuvenation on SFE value and surface morphology of the rejuvenated asphalt binder incorporated with WCO was explored using the Sessile drop method and tapping mode AFM. A relationship between the asphalt binder's surface roughness and SFE value can be made. From the analysis obtained from this research, it can be concluded that ageing will increase the asphaltene content in the asphalt binder. Consequently, increase the surface roughness of the asphalt binder. This contributes to the increasing contact angle and reduces the SFE value of the asphalt binder. It is also observed that the introduction of WCO into aged asphalt binder can increase the SFE value and reduce the surface roughness of the asphalt binder. Consequently, increases the adhesion and cohesion properties of the asphalt binder, making it more resistant to cracking.

Acknowledgement

The authors gratefully acknowledge the support received by this study from the Ministry of Higher Education under the Fundamental Research Grant Scheme (FRGS/1/2022/TK01/UTM/02/1), Universiti Tekonologi Malaysia, Skudai, Johor Baharu, and Universiti Teknologi Mara, Shah Alam, Selangor.

Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***Study conception, formal analysis, validation, and initial draft:*** *Muhammad Ibrahim Khalili Abd Rahim, Norzita Ngadi, Ekarizan Shaffie;* ***Data collection:*** *Muhammad Ibrahim Khalili Abd Rahim;* ***Analysis and interpretation of results:*** *Mohd Rosli Hainin, Mohd Khairul Afzan Mohd Lazi, and Muhammad Naqiuddin Mohd Warid;* ***Writing, review and editing:*** *Haryati Yaacob and Mohamad Ikhwan Jamaludin. All authors reviewed the results and approved the final version of the manuscript.*

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