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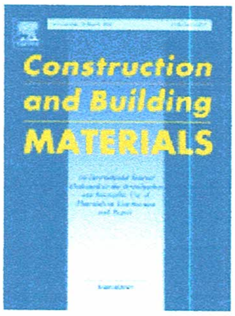

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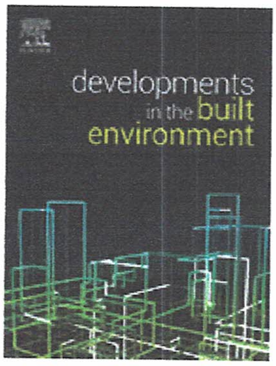

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Effects of Rediset on the adhesion, stripping, thermal and surface morphologies of PG76 binder

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HIGHLIGHTS

- Effects of adhesion, stripping, surface morphologies and thermal behaviour of PG76-Rediset were investigated.
- The adhesion and stripping properties were improved by the anti-stripping agent in Rediset.
- Changes can be seen clearly by the shapes and distribution of the bees' structures.
- Thermal decomposition takes place at temperature between 300 and 500°C.

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ABSTRACT

Apart from lowering the production and application temperature, Warm Mix Asphalt (WMA) additive has the potential to enhance the adhesion property of the base binders. Surface free energy (SFE) from contact angles was determined by sessile drop device (SDD) to find out the work of adhesion of WMA additive-modified binders. It is shown that addition of WMA additive at different weight percentage would initiate dissimilar implications on surface properties. Test data are compared with the nanoscale adhesion force of WMA additive-PG76 using atomic force microscopy (AFM). The connection between the contact angle analysis with the topography was also investigated; SFE and AFM results exhibit some agreement in findings. The WMA additive had improved the adhesion characteristics of warm mix additive-modified binders. In addition to that, adhesion and stripping behaviour of the blends were also observed via the Boiling Water test. The test demonstrated that the adhesive characteristics of the blending are at its best and the results inclined to the conclusion that the WMA additive had effect on the adhesion and stripping behaviour. The thermogravimetry analysis (TGA) was carried out to observe the thermal behaviour of the samples. It is found that the thermal decomposition of these blends takes place at temperature between 300 and 500 °C. Together, all the findings are in agreement that the addition of Rediset into PG76 could benefit for the WMA application.

1. Introduction

Polymer-modified asphalt (PMA) binder is known to improve the durability of virgin asphalt binder. Apart from that, the most important properties that has been improved is adhesion. Even though adhesion comes from the ability of asphalt binder to liquefy when heated; in order to perform as a component of pavement, it has to be in solid form when cooling [1–3]. By modifying the asphalt binder with polymer such as plastomeric or elastomeric thermoplastic like polyethylene or styrene–butadienestyrene (SBS), or engineered thermoplastics (ETP), the pavement durability seems to be increased [4–7]. These polymer modification can increase the heat resistance and cohesiveness of asphalt binder, enhance the elasticity and/or improve its low temperatures behaviour [8,9]. In addition, the adhesive characteristics of PMA binder also have improved largely [10]. Modifications on asphalt binder have been of interest today and researchers are constantly looking

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for improvement. For example, Md. Yusoff et al. [11] and Abdullah et al. [12] incorporating nano-clay and Warm Mix Asphalt (WMA) additive into PMA binder for greener construction approach. At the same time, Khairuddin et al. [13] looked into the potential of polyurethane as a modifier in improving the virgin binder.

WMA additive has been developed to enable the asphalt mixture to be produced at a lower temperature. The additive comes in various types namely; chemical based, water zeolite based and wax based; such as Rediset, Advera, and Sasobit [14,15]. A lot of previous studies [16–19] conducted identified WMA additives to not only reduce the mixing temperature but also improve other asphalt mixture properties [20]. Despite the fact that WMA additives are mainly used for the purpose of reduction of the asphalt binder's viscosity and other effects such as aging, alteration of rheological properties and mechanical characteristics in binders and mixes were also studied [21,22]. Ahmed et al. [23], Arowski et al. [24] and Ghabchi et al. [19] discovered that addition of WMA additive into asphalt binder improved the adhesion property.

Interfacial adhesion of the asphalt binder to aggregates is important in determining the resistance of the pavement to fatigue cracking and moisture-induced damage. Adhesion force can be defined as the work needed to create a new surface area of any material in a vacuum. Despite the molecular orientation theory, chemical reaction theory, and molecular dynamics, surface free energy (SFE) theory has been applied to study the adhesiveness of binder-aggregate interface [25]. The SFE value plays an important role in generating the adhesive force between two unlike materials. This is important in evaluating water damage and fatigue cracking of pavement. Atomic force microscopy (AFM) has been used to characterise morphologies of different plain and modified binders by many researchers [26–28]. Meanwhile, Wang et al. [27] are among others who used AFM to measure the adhesion between silicon or silicon–nitride AFM tips and asphalt binder. These studies have created an innovative niche to examine interactions between aggregates and binders at the nanoscale. However, nano-Newton forces measured using AFM tips with nanoscale radii cannot be directly used in micro-cracking models of asphalt mixtures as discovered by Li et al. [29].

On the other hand, amongst the studies to investigate the thermal stability of asphalt binder as well as its structural characteristics, thermal analysis offers thermodynamic parameters in most proficient way [30,31]. The use of efficient thermodynamic methods is very important because Zhang et al. [32] investigated the thermal stability of stone-mastic asphalt with the effect of sulphur by means of integral procedural decomposition temperature (IPDT) method or the onset temperature of mass loss and found that these techniques are not very reliable in characterising the structural characteristics of binder and some errors occurs sometimes. Alamawi et al. [33] had used this method to study the decomposition of virgin asphalt binder replaced with various percentage of palm kernel oil polyol (PKO-p). Since the fingerprint of asphalt binder is determined by the decomposition temperature of each component in the material [34,35], Thermogravimetric Analyser (TGA) tests were carried out to study the pattern of thermal decomposition on tested samples. From the tests, TG curves defining the relationship between the mass of test sample and temperature can be obtained.

This study emphasis on the analysis of adhesion behaviour of PG76 upon addition of WMA additive, Rediset LQ1106. Contact angles data are employed to calculate surface energy of the tested binders prior to determination of the adhesion work. Adhesive forces are obtained directly with AFM analyser. Their correlationship is used to uncover the dissimilarity between macro- and micro- tests on adhesion behaviour of asphalt binders.

2. Materials and methods

WMA additive namely Rediset LQ 1106 (herein after referred as Rediset) was incorporated into polymer-modified binder, namely PG76. This additive is known to have been used to increase stability [36], tensile strength [37], anti-rutting properties and life-span [38]. Rediset was added at percentage weight of PG76 varying in 1, 2, 3, 4, and 5%. The blending was done beforehand, by adding respective percentage weight of Rediset into PG76 at 160 °C and mixed at 1250 rpm for 20 min. The binder substrates were prepared as thin film for adhesion measurements by sessile drop method and AFM. The properties of PG76 and Rediset used are presented in Table 1 and Table 2 respectively.

2.1. Boiling water test

In boiling water test, boiling water was used to assess the effect of water on asphalt aggregate mixture. The test was performed following the ASTM D3625 specification [39]. In order to execute the test, about 250 g of asphaltic-coated aggregate mixture was placed into approximately 500 ml boiling water. The water then was bringing back to boiling and be let to boil for another 10 min ± 15 s. After the 10 min, the container was removed from the heat source. Free asphalt binder on the water surface was skim off to avoid recoating. The asphaltic-coated aggregate mixture was poured onto wet paper towel and leave cooling at room temperature. The mixture after that is observed visually to see how much asphalt binder coating retained on the aggregate (coarse and fine) surfaces. Clean surface area with no means of binder is looked-for. Any thin, translucent, or brownish areas are to be considered as fully coated. The visual inspections must be made as soon as the sample is placed on the white paper towel. Similar amount of fresh asphaltic-coated aggregate mixture be placed into a second container with similar amount of unheated distilled water for 10 min. This setup is for comparison to the one in boiling water which has been beforehand. Afterwards, the water decanted and the mixture drained onto a white paper towel. The degree of stripping was evaluated visually to the same prerequisite as the one in boiling water. The ratio of "clean" area to that of coated counted in percentage was use to estimate the moisture damage.

2.2. Thermogravimetric analysis

Thermogravimetric Analyser (TGA) is a tool used to test the weight change of the sample compared to temperature under controlled conditions. This TGA device can operate at maximum 600 °C. Its main use is to determine the thermal properties of a substance or sample. The analysis was carried out by utilising Shimadzu TGA-50 thermogravimetric analyser in a protective atmosphere of nitrogen, with a flux of 50 ml/min and a heating rate of 10 °C/min. A small amount of binder sample ranging from 2.0 mg to 5.5 mg was used for this purpose. The binders were kept at room temperature at room temperature and placed in an aluminium cell to initiate the analysis. After that, the samples were heated from temperature room temperature to 600 °C. Their weight loss was recorded by a microbalance. The analyses was carried out with reference to ASTM E 1311-08 [40].

2.3. Sessile drop device and SFE

The sessile drop (SD) device, which consist of simple apparatus is somewhat reliable, precise, and inexpensive for the measurement of surface energies via the determination of contact angles between binders and various probe liquids [17,18]. The method

Table 1
Properties of PG76.

Test	Unit	Result	Requirement	Test Standard
Quality specification				
Penetration	0.1 mm	46	Min. 45	ASTM D5
Softening point	°C	93	Min. 70 °C	ASTM D36
Flash point	°C	343	Min. 260 °C	AASHTO T48
Performance specifications				
Viscosity at 135 °C	Pa s	2.45	Max. 3 Pa s	ASTM D4402
Dynamic shear, G*/sinδ	kPa	2.10	Min. 1.00 kPa	AASHTO T315
Test temp @ 10 rad/s, 76 °C				
Rolling thin film oven test (RTFOT) residue (AASHTO T240)				
Mass loss	%	0.04	Max. 1.00%	AASHTO T240
Dynamic shear, G*/sinδ	kPa	3.40	Min. 2.20 kPa	AASHTO T315
Test temp @ 10 rad/s, 76 °C				
Pressure ageing vessel (PAV) residue (ASSHTO R28)				
PAV ageing test temperature				
Dynamic shear, G*/sinδ	kPa	1200	Max. 5000 kPa	AASHTO T315
Test temp @ 10 rad/s, 37 °C				

Table 2
Physical properties of Rediset.

Item	Index
Appearance @25 °C	liquid
Pour point, °C	3
Flash point, °C	170
Viscosity @40 °C, mPa s	216
Density @40 °C, g/cc	0.99

provides essential information about the material surface, such as its wettability and adhesiveness. The SD method is one of a few ways done for solid surface energies as well as liquid surface energies characterisation [41]. This method works by calculating the surface energy of the solid surface via its contact angle determined by the shape of the drop of a liquid droplet with known surface energy referred as probe liquid. Normally three different probe liquids are required to calculate the surface energy of the sample. After that, acid-base theory is used to calculate the SFE components as per Eq. (1). This study assessed the SFE of unaged binders in consideration of the initial stage of mixing production, before binder started to age, where the process of binder - aggregates coating/bonding occurs.

The specimens were prepared by dropping a small amount of previously blended asphalt binder on a glass slide. The glass slide with asphalt binder droplet was then heated in the oven at softening point temperature; enough for the droplet to freely flow and form a thin layer. The samples were then being kept cool in covered petri dish ready to be tested. One sample will be used for each liquid probe. Thus, three specimens of each sample were prepared for this test. For each test, 3 replicates for each sample were conducted.

In this research, the three liquid probe used was distilled water, glycol and formamide, which have high and different SFE from asphalt binder. They are insoluble in asphalt binder. SFE and components data of probe liquids are tabulated in Table 3.

By using Eq. (1), SFE of asphalt binder is determined.

$$1+\cos\theta = 2\sqrt{\gamma_s^d}\left(\frac{\sqrt{\gamma_l^d}}{\gamma_l}\right) + 2\sqrt{\gamma_s^p}\left(\frac{\sqrt{\gamma_l^p}}{\gamma_l}\right) \quad (1)$$

where θ is the contact angle between liquids and asphalt binder, γ_l (mJ/m2) is the liquid's SFE, γ_l^d (mJ/m2) represents the liquid's dispersion component, γ_l^p(mJ/m2) is the liquid's polar component, γ_s^d (mJ/m2) represents the solid's dispersion component, and lastly,γ_s^p (mJ/m2) is the solid's polar component.

Furthermore, the work of adhesion between asphalt binder and respective aggregates can be calculated by using Eq. (2) [42]:

$$W_{adhesion} = \gamma_{asphalt} + \gamma_{aggregate} - \gamma_{asphalt-aggregate} \quad (2)$$

Alternatively, the work of adhesion can also be characterised according to Fowkes [41] and Owen-Wendt's theory [43] as follow:

$$W_{adhesion} = 2\sqrt{\gamma_{asphalt}^d \gamma_{aggregate}^d} + 2\sqrt{\gamma_{asphalt}^p \gamma_{aggregate}^p} \quad (3)$$

where$W_{adhesion}$represents the work of adhesion between the binder and aggregate, each dedicated subscripts represent the binder, aggregate and binder-aggregate interface.

2.4. AFM analysis

In contrast to SFE derived from interactions of two different subjects, namely liquid and solid, AFM strength measurements directly examine the mechanism of solid–solid interaction by means of tip to contact the asphalt samples. While SDD is a straightforward physical test, AFM offers the potential as a powerful technique for pavement researchers to investigate the nano-mechanical properties of thin-film asphalt binders. It is well established that the microstructure of asphalt binder is responsible for its physical properties such as elasticity, plasticity, stiffness, adhesion, surface energy and healing. Undeniably, there is a need to understand the behaviour of asphalt binders at the nanometre scale. The interaction between the composite phases of asphalt

Table 3
SFE of probe liquids and their components.

Type of probe liquid	Non-polar component γ^{LW} (mJ/m^2)	Basic component γ^- (mJ/m^2)	Acidic component γ^+ (mJ/m^2)	Polar component γ^{AB} (mJ/m^2)	Total SFE γ (mJ/m^2)
Water	21.8	25.5	25.5	51.0	72.8
Glycol	29.0	47.0	1.9	19.0	48.0
Formamide	39.0	39.6	2.3	19.0	58.0

mixtures takes place at this scale. Such work will allow the nano-mechanical properties of the asphalt binders and the mix to be correlated with their micro- and macro-mechanical properties, and thereby better understand the behaviour of asphalt mixtures under applied load.

An AFM was utilised in this study to observe the changes in PG76 modified by different percentages of WMA. The employment of AFM will result the nano-mechanical and morphological characteristics of the materials. In this study, binder samples in form of thin film were observed by employing a XE-10 AFM from Park Systems Corporation, on the surface of each sample. The data acquisition is controlled with XEP software while XEI software used for image processing and analysis. In the test, a sharp AFM tip attached to a laser-tracked cantilever is used to investigate the surface of the sample and the attractive or repulsive force between the tip and the sample surface is measured. For this study, the area of AFM images obtained from the sample surface is 25 × 25 µm at 1.0 Hz scan rate. The non-contact mode method was employed using a silicon cantilever tip sized <10 nm tip radius at 0.2 N/m force constant for the quantifications of binder surface.

3. Results and discussion

3.1. Boiling water test

This study utilised the boiling water test to evaluate the effect of water on asphalt mix in terms of degree of stripping by using boiling water and the assessment is done visually. Even though the test is simple and straight forward to carry out, it is capable to appraise the effect of anti-stripping in the mixture incorporating with additives in order to diminish the adhesion loss between aggregate and asphalt binder [44]. Parker and Wilson [45] also

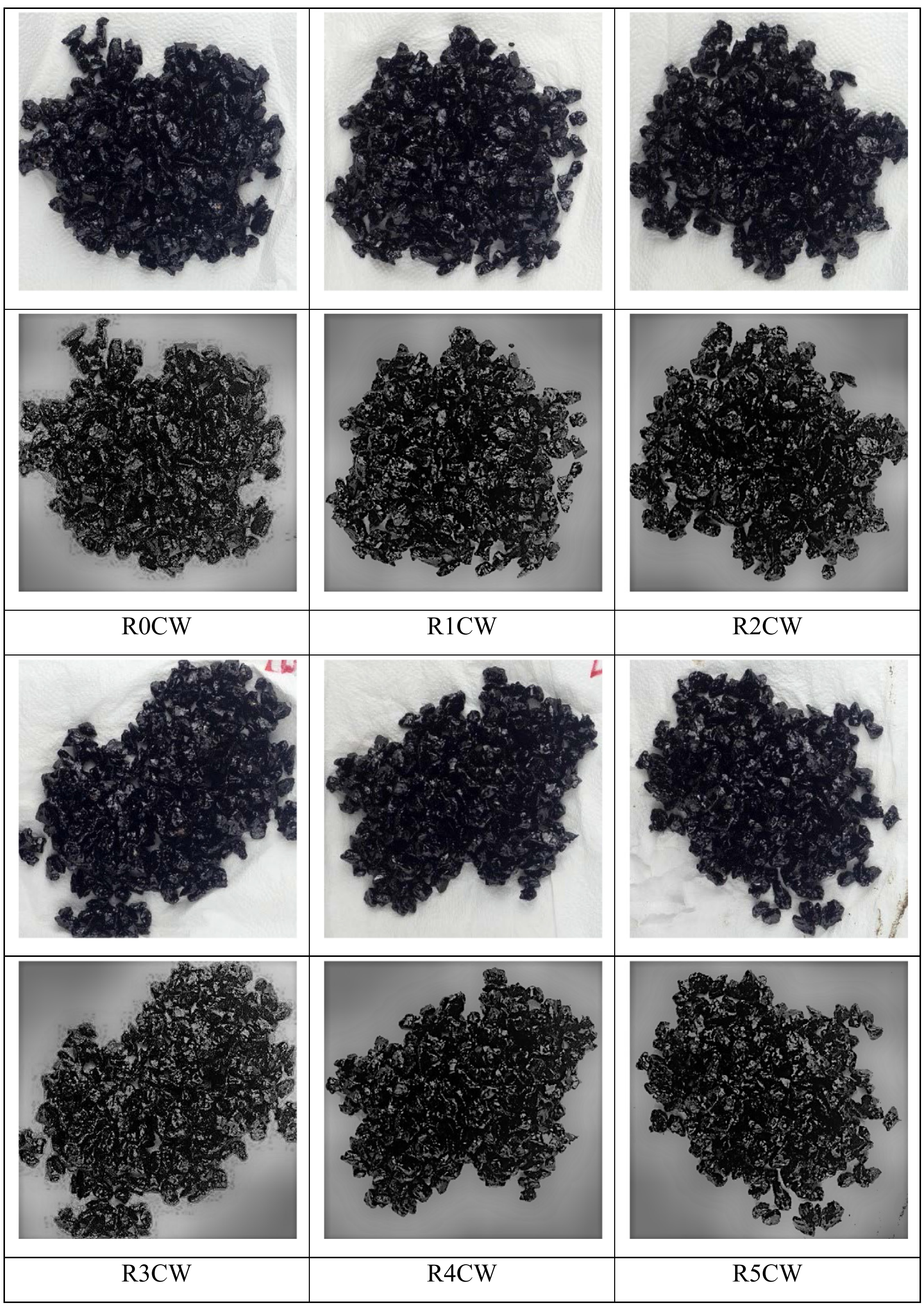


Fig. 1. Digital image of the boiling water test (cold water).

found that the boiling water test has provided a good interrelationship between experimental results and in-situ performance. In this test, the criterion of failure is visually identified on how much stripped (uncoated) aggregates after soaking in boiling water for 10 min. The test was initiate by visually estimate the asphalt binder coating on the aggregates before testing as reference condition. Another observation is done when the test finished and the two conditions are compared to find the degree of asphalt stripping from the aggregates. A digital camera was use to capture the images so that it can be analyse using digital image analysis software. Every picture has to go through the cropping process to get a consistent size. After that, by applying the same level of threshold, picture was transformed into a greyscale image. Each black pixel was counted using an image analysis software. The area of black pixel counted was compared to the whole specimen area to get the ratio of black pixels to the area.

As an example, Figs. 1 and 2 show the original images of the asphalt mixture together with their greyscale images for comparison which has been transformed using image analysis software for the all the binders. Firstly, the digital images went through image enhancement process by converting them into 8-bit type image. This process transformed the images into greyscale images. They were then filtered by enhancing the local contrast to get clearer disparity. By doing that, the portion of black pixels can clearly be seen from all the mixture. Finally, binarization was done to analyze greyscale distribution of image based on grey histograms. Fig. 3 is the plots of analysis results. The bar chart shows the pixel count per total area of the image subject. The higher the value means darker (black) pixels per area of the photo taken. However, the quantified results are only in estimation. In other words, the percentage of white portion cannot be affirmed as the real value of stripping. The image processing related factors, for

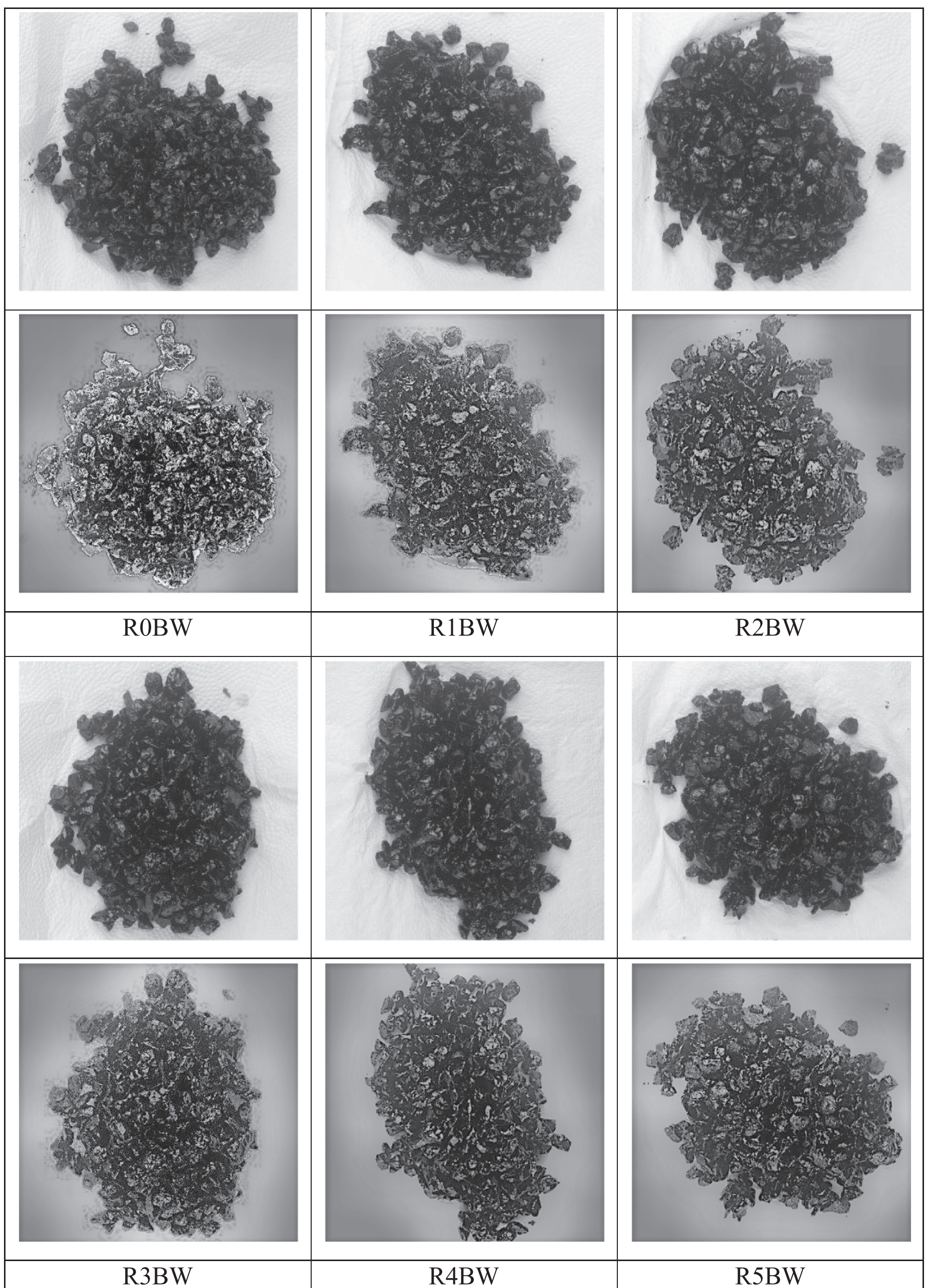


Fig. 2. Digital image of the boiling water test (boiling water).

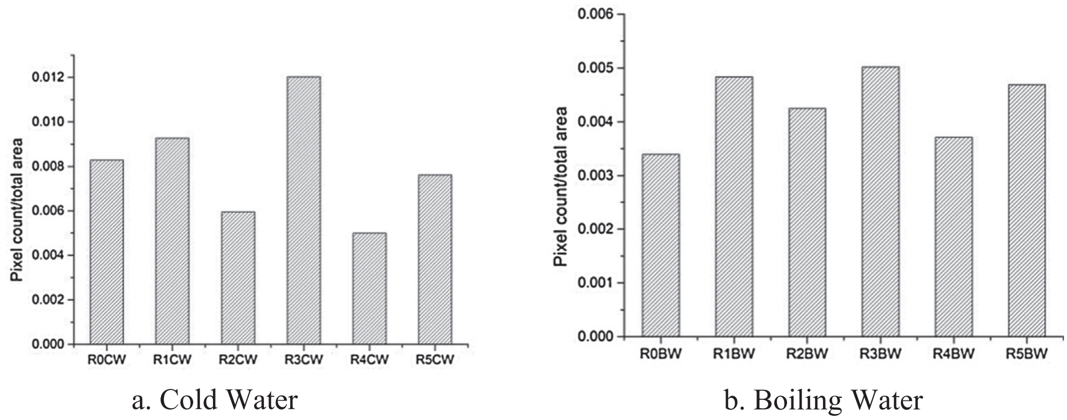


Fig. 3. Digital image analysis results for boiling water test.

example the level of threshold applied might influence the values presented. Nevertheless, Kim et al. [44] also agreed that a relative ranking among mixtures in an objective manner can still be made, because the comparison on similar factors made are employed to all mixtures.

3.2. Thermal analysis

The TGA results in this study shows agreement to Miguel et al. [46]. It is found that the decomposition of asphalt binder takes place in three steps the least, with the consideration of considering three temperature ranges, as shown in Fig. 4. Meanwhile, Table 4 describes the starting and ending temperature for the process as well as the total weight loss at the end of decomposition of samples. Overall, the composition took place from 66.9% up to 91.5%.

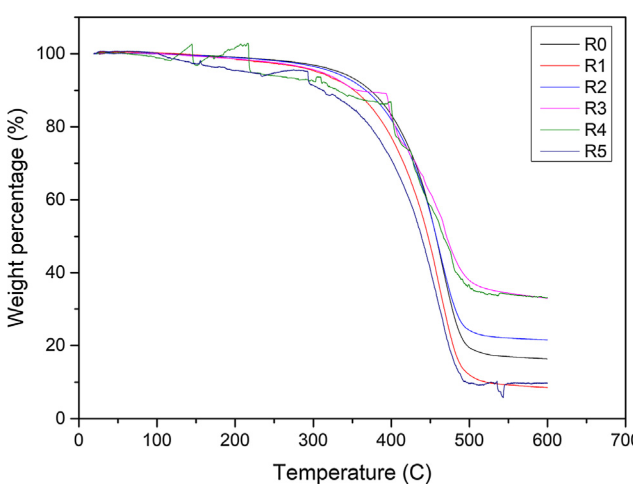


Fig. 4. TGA curves of all tested samples.

Table 4
Temperature range and total weight loss for tested samples.

Sample	Temperature		Weight loss	
	Start (°C)	End (°C)	mg	%
R0	25.7	599.8	−4.59	−83.73
R1	19.6	599.9	−2.29	−91.50
R2	20.6	599.8	−3.66	−78.51
R3	25.8	599.8	−2.97	−67.01
R4	19.6	599.9	−2.26	−66.92
R5	19.1	599.9	−1.46	−90.32

All samples were thermally stable up to about 300 °C except for R4 and R5 that carried 4% and 5% weight of Rediset. These two combinations started to become unstable at the temperature of 100 °C. At second phase of the decomposition process which took place at temperature > 300 °C > 500 °C, it can be seen that all samples decomposed drastically. As the process enters the third phase, all the subtracts remnants had meet a stable state. At this phase, the decomposition of binders had decelerated and the remaining weight is due to the evolution of hydrogen [47].

3.3. Sdd

The total surface energies were calculated from the contact angles of three probe liquids used in the study are illustrated in Figs. 5 and 6 respectively. On the other hand, Table 5 is the tabulation of contact angles of tested samples with reference liquid probes determined directly by SDD. Each liquid probe gives unique contact angle and contributes to different data pattern. Even though it has been a decrease in the value of contact angle upon addition of Rediset, there has not been a constant pattern with all probe liquid. The repeatability value of tested specimens is 1.41 for all samples. The pooled relative standard deviation (% RSD) for each specimen-liquid combination ranging from 0.15% to 7.86%. These values are acceptable for small numbers of sample (less than 30). Thus this test is repeatable. Generally, by addition of Rediset, the contact angle from distilled water for PG76 was decreased. Contrary to distilled water and formamide, the glycol contact angle had shown a slight rise in value during the inception but after that, it decreased when higher amount of Rediset was

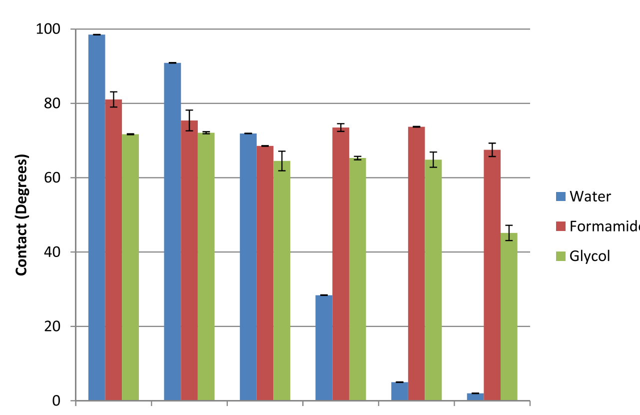


Fig. 5. The contact angles (degrees) of different probe liquids on all tested samples.

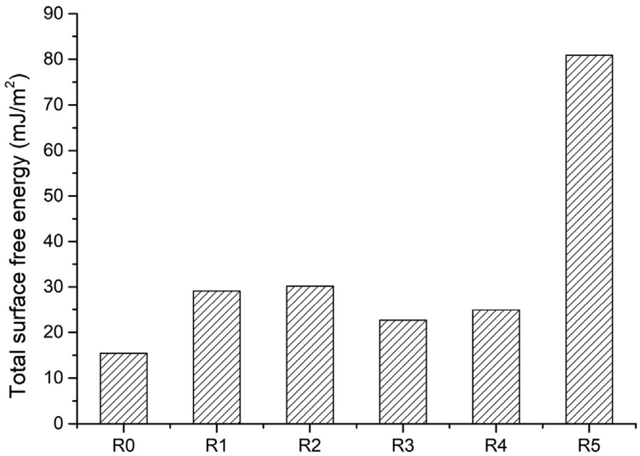


Fig. 6. The Total SFE of different probe liquids on all tested samples.

added. The contact angle then increased slightly with the addition of 3% of Rediset but the value had decreased with increasing amount of Rediset. The higher the amount of Rediset, the lower the contact angles of glycol for PG76 except at 3% Rediset. Formamide gives almost the same patterns of contact angle from water, except that, the values were slightly higher when 3 wt% of Rediset was added. Even though the angles decreased again for 4% and 5%, the angle for 4% has been slightly higher than it was of 2%. The data for formamide on the other hand, shows that Rediset initially decreased in contact angle at first and then increased with the addition of 3 wt% Rediset and decreased again with the addition of Rediset in PG76. Omar et al. [48] had applied the same method to observed the water damage resistance of nano-clay modified asphalt binder and discovered a promising results in relation to indirect tensile test.

3.4. Sfe

SFE for this study utilised the Owens, Wendt, Rabel and Kaelble (OWRK) method; of which a standard way for calculating the surface free energy of a solid from the contact angle with several liquid probes. The contact angles of binder-probe liquids had been determined by using sessile drop device. The OWRK method is used because this study investigating the contact between surfaces via wettability and adhesion of two different materials; asphalt binder and aggregates. This method was built on Fowkes method, where the surface tension of binder-aggregate $\gamma_{asphalt.aggregate}$ is calculated based on interfacial tensions $\gamma_{aggregate}$ and $\gamma_{asphalt}$ and the identical interactions between the phases. These interactions are clarify as the geometric mean of a disperse part γ^d and a polar part γ^p of the surface free energy. Fig. 6 shows the effect of adding Rediset on the surface energy of the PG76. The addition of Rediset can significantly affect the surface energy characteristics of the binders. The PG76 originally had a surface energy of 15.43 mJ/m^2. Upon addition of 1 wt% of Rediset, the surface energy of wetting increased to 29.12 mJ/m^2 an increment of almost 30%. With the addition 2 wt%, the surface energy increased to 30.16 mJ/m^2. The SFE then decrease to 22.7 mJ/m^2 when 3% of Rediset added to PG76 but the SFE slightly increased again to 24.92 mJ/m^2 when the Rediset content increased to 4 wt%. However, when the Rediset was added up to 5%, the SFE dramatically increased to 80.9 mJ/m^2 giving the highest value. The reduction in contact angle was constant only when Rediset was added up to 3 wt%. This means that PG76 has positive wettability but only with the addition of up to 3 wt% Rediset. With the increase in wettability, the adhesion properties will improve [49].

3.5. AFM observations

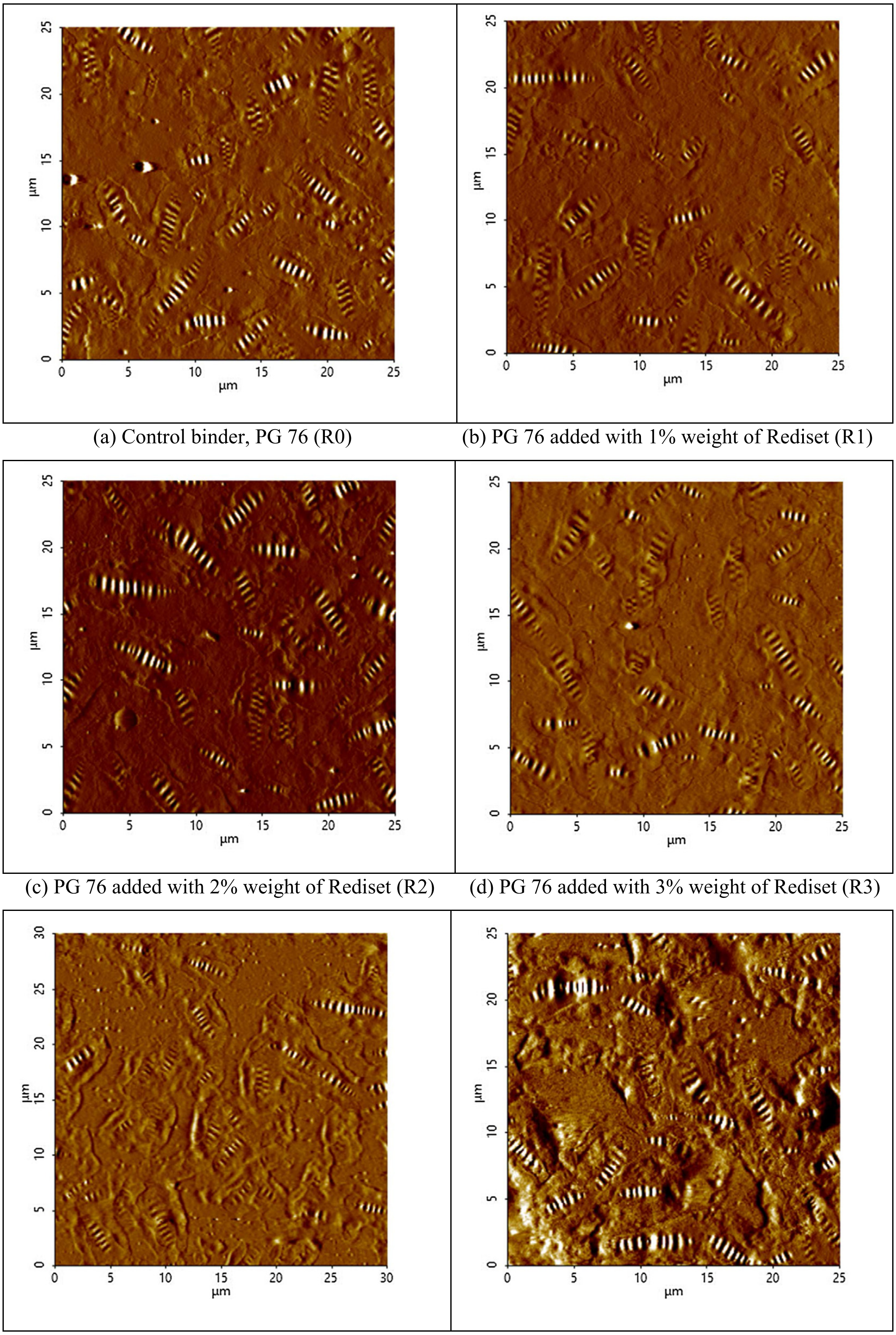
3.5.1. Morphological characterisation

AFM was utilised to obtain the images of various surface as shown in Fig. 7. AFM with soft silicone tip was employed to scan the surfaces of all the tested binder samples. Generally, all the binder's surfaces exhibit the typical bee-structures which mean that the asphalt binder, PG76 had been well blended with Rediset. As a whole, each image is make-up of the high-contrast bee-like structures in the centre and their lower (darker) vicinity. It also shows different features with each other, no two is the same which appears that the morphologies of the asphalt binders had been affected by Rediset. Theoretically, Pauli et al. [50] found that the bee structure formation is mainly effected by several factors such as the crystallizing wax content molecule diffusion ability and molecule polarity. The pronounced surface corrugations (up to hundreds of nm) of the bee-like structures are the consequence of local stresses developed during cooling of the asphalt binder adsorbing agent. These structures and their immediate surroundings are mostly composed of wax, which includes assortment of nonpolar alkane chains [51].

From the image captured, it can be seen that different weight of Rediset addition gives different morphology. Fig. 8 shows the grain counts analysed by the software. Figs. 7 and 8 are interrelated. The system will automatically identify grains in the image, and calculates important surface profile parameters of each grain. Afterward, it displays the distribution of surface parameters among detected grains. Plain PG76 that is referred as controlled sample shows obvious numbers of "bees". The grain count (Fig. 8) is 49. When Rediset is added (Fig. 7b), visually, the bees become lesser compared to PG76 without any addition can be seen in Fig. 7a. However, the grain number has increased to 67. With increasing of Rediset content, it could be the topography is more undulating than previous weight percent. It can be seen that the dispersed area also started to make more appearance. When the additive weight added increased to 3%, the bees visually increased too,

Table 5
Contact Angles of tested samples with probe liquids.

Sample ID	Water			Formamide			Glycol			Repeatability (multiple liquids) (Degrees)
	Mean contact angle (Degrees)	Standard deviation	%Pooled relative standard deviation (%)	Mean contact angle (Degrees)	Standard deviation	%Pooled Relative Standard Deviation (%)	Mean contact angle (Degrees)	Standard deviation	%Pooled relative standard deviation (%)	
R0	98.50	0.10	2.53	81.05	3.55	3.10	71.70	0.20	0.28	1.41
R1	90.90	0.10	3.70	75.40	4.80	4.53	72.10	0.50	0.69	1.41
R2	71.90	0.10	4.06	68.55	0.15	4.98	64.50	4.56	7.03	1.41
R3	28.40	0.10	1.59	73.50	1.80	1.94	65.30	0.80	1.23	1.41
R4	5.00	0.10	3.37	73.70	0.15	3.87	64.85	3.55	5.47	1.41
R5	2.00	0.10	6.00	67.50	3.10	6.44	45.15	3.55	7.85	1.41



(a) Control binder, PG 76 (R0)

(b) PG 76 added with 1% weight of Rediset (R1)

(c) PG 76 added with 2% weight of Rediset (R2)

(d) PG 76 added with 3% weight of Rediset (R3)

(e) PG 76 added with 4% weight of Rediset (R4)

(f) PG 76 added with 5% weight of Rediset (R5)

Fig. 7. AFM images of PG76 binder added with various percentage of Rediset.

more than the one with 2% weight content as per Fig. 7c. The grain number also keeps on increasing to 87. There are more dispersed area started to show compared to previous samples. The structure looks like they start to "swell" to.

As for 4% weight (Fig. 7d), the bees look like as if they had "swollen" and they are scattered around the area of interest. The bees' structure became wider and the dispersed area became obvious too. The grain count for this sample is 60, lesser than the

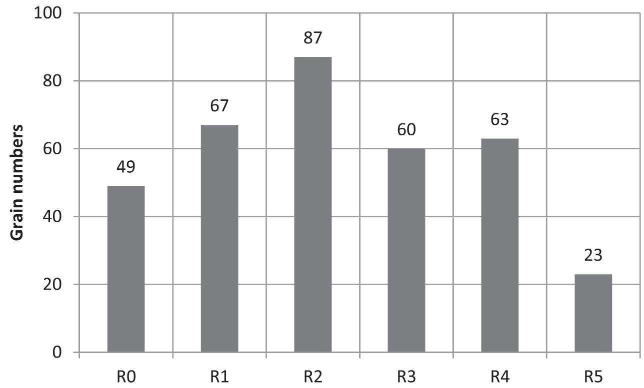


Fig. 8. Grain numbers for all tested samples.

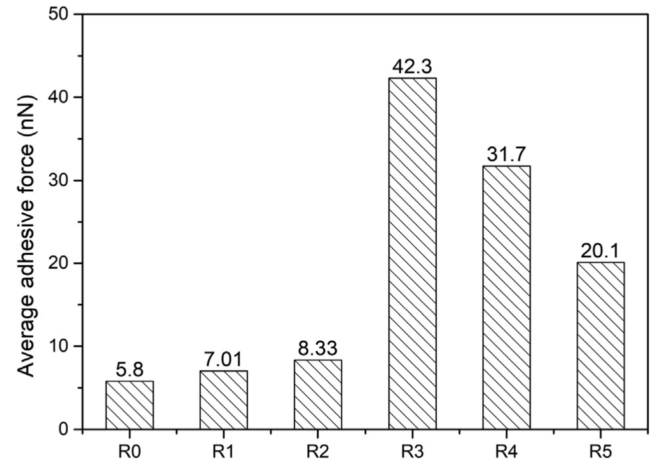


Fig. 9. Average adhesive force of tested samples extracted from AFM.

previous blending. The number of grain had increase by 3 for the sample with 4% Rediset (Fig. 7e) while in Fig. 7f, as for 5% weight of Rediset addition, the bees had blended with the dispersed area and the count had become the least; that is 23. Thus, as the weight percentages of Rediset increased, the amount of bee structures also increased but larger bee structures with relatively sparse distribution were seen when the amount of Rediset added >2%. These results prove that the asphalt binder morphologies were affected by the weight percentage of Rediset added. Morphologies of binder with different weight percent of Rediset clearly demonstrated microstructural variations in these asphalt binders. The finding is in agreement with that of Li et al. [29] who measure the adhesiveness between aggregate minerals and different type binders via particle-probe scanning force microscopes resulting various morphologies concluding that alumina-binder pairs yields the utmost adhesion value compared to silica-binder or calcium carbonate-binder pairs. Dourado et al. [52] also discovered a straightforward relationship between the presence of the bees and the value of the adhesion fore when the topography and slope images from 50/70 penetration grade asphalt binder are compared.

The results are in agreement with Loeber et al. [53] whom during early application of AFM for asphalt binder imaging uncovered the existence of a particular surface structures that they called 'bumble bees'. Through this study, Masson et al. [54] utilised the phase modes and perceived the material heterogeneity in the phase images, which were recorded in amplitude modulation mode. Although some researchers refer the 'bumble bees' to asphaltene [55], others relate them as being mainly composed of semi-crystalline wax including a variety of alkanes [56], while others hypothesized as surface wrinkling due to buckling of the bee laminate [57].

3.5.2. Adhesive force measurement

Average adhesive force of PG76 added with Rediset extracted by using soft tips is shown in Fig. 9. It can be seen that by incorporating Rediset into the PG76, it induces remarkable changes to values of asphalt binders' adhesive force although the surfaces of these samples practically look nearly similar. In accordance with current documentations, these bee structures might happen in the very top layer of the binder which known to be a surface phenomenon [58]. Given that this study emphasis only on the adhesion property of modified asphalt samples influenced by weight percentage of Rediset, details about the phenomenon is not specifically discussed here. Obviously, results obtained by each sample which contains different amount of Rediset are totally dissimilar. Adhesive forces obtained from the analysis indicate that Rediset only introduce substantial alterations to the adhesion property of PG76 for 3% and more. While addition of 3% Rediset may escalate the adhesive forces of PG76, further addition of Rediset reduce the adhesive force compared to the one with 3%. However, the value is still way higher than the plain and one with 1% and 2% of Rediset. PG76's adhesive forces started at 5.80 nN without any addition of Rediset and increasing to 7.01 nN and 8.33 nN when added with 1% and 2% of Rediset respectively. The adhesive force shoots up to 42.30 nN when the concentrate of Rediset added up to 3%. The force then slowly decreases with the increase of concentration of Rediset. The adhesive force value is in coherent to the deformation of quantification region on surface of the binder.

3.6. Adhesion analysis

3.6.1. Theoretical work of adhesion between asphalt binder and basalt by SFE calculation

Fig. 10 presents the theoretical work of adhesion between asphalt binder and basalt determined by means of SFE calculation. While there are a handful of aggregates choices to study adhesion, this study chose basalt as the subject aggregates because they are one of a few commonly used types of rocks for road construction. The relationship is quite relevant even though it's not consistent. Theoretical adhesion work of the base binder PG76 for this study is 138.59 mJ/m^2. The value increased with the increment of Rediset quantity added. It seems by adding 1% of Rediset, the adhesion work increased by almost 40% to 188.29 mJ/m^2. The value keeps increasing when more Rediset is added. The adhesion work had increased almost 70% when 2% Rediset is added compared to the increment by 1% Rediset. 3% weight of Rediset into the base binder

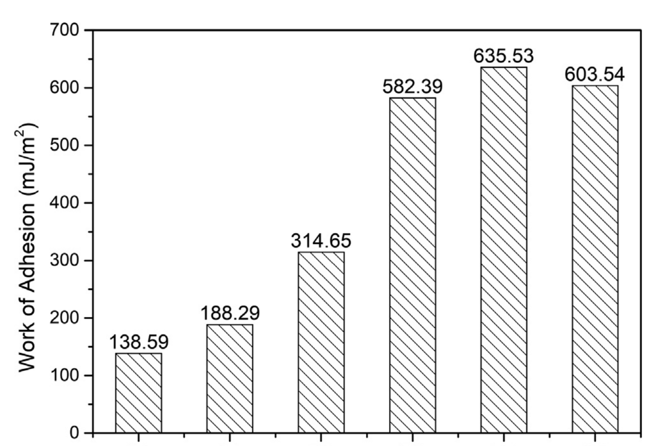


Fig. 10. Work of adhesion determined by SFE calculation.

of PG76 give the most increment compared to previous addition of 2%. With 3% of Rediset, compared to that of 2%, the increment had been 85%. The adhesion work then increased a little when 4% Rediset added. The value then dropped slightly by 5% when maximum weight of Rediset for this study is added. While Wei et al. [59] analysing the relationship between SFE and chemical properties of binders, Gong et al. [26] had done an extensive study comparing the work of adhesion of bio-binder modified binder. The data demonstrated in a way that bio-binder magnifies the interaction between bio-binder modified binders (with 70 penetration grade as the base binder) and basalt but it weakens the interaction between bio-binder modified asphalt binders (with 30 penetration grade as the base binder) and basalt. Meanwhile, Koc and Bulut [60] found that the laboratory tests point towards concurrency of results tested on aggregate specimens from the SD device and that from the past studies on clay and talc minerals. However, they are not in agreement with the results acquired on the same aggregate types using the universal sorption device (USD). Thus, this study employed the SFE from literature [61] for basalt. Of all the referred previous works, none showed a linear relationship or consistency between the modification rate and work of adhesion values. It is found that in this study, 4% weight of Rediset addition gives the highest value of adhesion, and the increasing consistency of theoretical value was then started to decrease when 5% weight of Rediset was added in.

3.6.2. Comparisons between SFE results and adhesive forces

In order to better characterise asphalt binder's adhesion behaviour, adhesive force from AFM analysis and work of adhesion from SFE are tabulated in Table 6. Generally, Table 6 shows values of that with addition of Rediset, the total SFE of PG76 increased. However, this relationship only true up to 3% weight. Any more of that amount, the SFE drops compared to those of 1% and 2% but the drop does not go any lower than the SFE of control sample, PG76. The fact that the trend of adhesive force from AFM are contradict with the total SFE trend makes this observation fascinating. It is found that there is no direct links can be found between the SFE of the tested samples and adhesive forces. This phenomenon might be related to the fact that the van der Waals force is no longer dominated in the interactions between the AFM tip and asphalt binder.

Table 6 discloses the adhesion work for all samples with basalt obtained from SFE calculation and adhesive force acquired directly from AFM tip. However, there is no straight connection between the findings of these two groups is found because there is a difference between adhesion at the molecular level and adhesion in engineering [62]. The disparity of it can be ascribed as follows:(a) while SD method probe substance are liquids, the measurements of AFM utilises solid tip as probe material. Basically, liquid–solid interactions are different with solid–solid interactions. (b) regarding to the vicinity of data compilation, the method of contact angle covers contact area of several square millimetres while that of AFM, the area is very small; around 2 nm^2. These variations are due from different test scales as well. Finally, (c) SFE calculation resulting of dispersive and polar component [63–65] while AFM adhesive force result comprises mainly van der Waals force and capillary force without any polar force [66]. Thus, hypotheoretically, the adhesive property from these methods i.e. the dispersive components in SFE and AFM adhesive force have more commons.

Table 6
Work of adhesion (SDD) and adhesion force (AFM) of tested samples.

Sample	Work of adhesion (SDD) (mJ/m^2)	Average adhesive force (AFM) (nN)
R0	138.59	5.8
R1	188.29	7.01
R2	314.65	8.33
R3	582.39	42.3
R4	635.53	31.7
R5	603.54	20.1

4. Conclusion

From the series of tests done, it is found that with the addition of Rediset, the adhesion properties of the PG76 binder had been improved. The combination was introduced to explore its adhesiveness in the view of physical and microcosmic mechanism. In this study, the water boiling test gave good results of the adhesive characteristics between the studied asphalt binder and aggregates because the polymer-modified binder has already had good adhesion. Furthermore, the adhesion properties were improved by the anti-stripping agent in Rediset. On the other hand, the thermogravimetry analysis done for all the samples showed that the decomposition process which took place at temperature > 300 °C > 500 °C.

The analysis of adhesion using surface energy can help to select compatible asphalt binder–aggregate combinations. The surface energy of the asphalt binder can be effectively computed from the contact angles measured with the Sessile Drop method. The addition of Rediset would cause different impact on the contact angles of different probe liquids on polymer modified binder samples. This fact is the consequent from definite compatibilities between PG76 and different weight percentages of Rediset used in this study. It is also found that different asphalt binders have quite different surface energy characteristics, which reflect the variation of their surface physical chemistry. On the contrary, AFM offers qualitative and quantitative measurement for the phenomenon. However, SFE results seem to agree well with AFM results in the term of adhesion property. The differences between SFE and AFM measurements can be contributed by differences in probe substance, the scale of test and the components measured. Via microscopic analysis, the changes in each sample can be seen clearly by the shapes and distribution of the "bees" structures. This indicates that Rediset had been well blended and affects PG76 physically. The existence of this structure and the grain count might have relationship with the adhesion force.

CRediT authorship contribution statement

Suzielah Rahmad: Writing - original draft. **Nur Izzi Md. Yusoff:** Writing - review & editing, Supervision, Funding acquisition. **Sri Atmaja P. Rosyidi:** Writing - review & editing. **Khairiah Haji Badri:** Writing - review & editing, Supervision. **Iswandaru Widyatmoko:** Writing - review & editing, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.conbuildmat.2019.117923.

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