Effect of different rejuvenating agents on the mechanical performance of recycled bituminous mixture

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Abstract. Reclaimed asphalt pavement (RAP) is a waste material generated from pavement milling activity. Rejuvenating agent is introduced to restore the properties of aging bitumen in RAP. Many studies have been investigated the performance of recycled mixture incorporating RAP with oil-based (OB) rejuvenating agent, but there is still limited research on water-based (WB) rejuvenating agent. Therefore, this study evaluated the performance of mixture incorporating RAP-OB and RAP-WB. OB and WB rejuvenating agents were added at recommended content of 0.70% and 52.13% by weight of bitumen, respectively. Indirect tensile strength, resilient modulus, dynamic creep and asphalt pavement analyzer (APA) tests were conducted to assess the resistance of the mixtures toward permanent deformation and fatigue cracking. It was found that the addition of OB rejuvenating agent enhanced the tensile strength, resilient and stiffness modulus by approximately 41%, 94% and 158%, respectively, relative to the control mixture. APA recorded that RAP-OB produced the lowest rut depth, followed by RAP-WB and control mixture. It indicates that RAP-OB produced the highest resistance to permanent deformation and fatigue cracking. Thus, the OB rejuvenating agent significantly improved the performance of the recycled mixture compared to the WB rejuvenating agent.

1. Introduction

Reclaimed asphalt pavement (RAP) is one of the alternative materials for pavement in order to overcome the problems of increases in the price of asphalt materials and reduction of natural resources [1–4]. The incorporation of RAP as asphalt pavements could offer great impacts on the environment and in economic terms. In addition, RAP is a waste material that is transformed into very efficient use of resources thereby promoting sustainability in pavement field. However, RAP consists of aged bitumen and aggregates that lead to pavement cracking, thereby affecting its serviceability. Thus, many studies have been conducted to ensure the amount of RAP used in asphalt mixture is appropriate. Also, the use of rejuvenator to recover the aged properties of RAP mixture can be seen to offer better solution to overcome this problem. Rejuvenator is an additive used to soften the RAP binder and restore the physical and rheological properties to conventional binder condition. It also can enhance the cracking resistance of the asphalt mixture as has high resistance to rutting. Many studies have been conducted on the performance of RAP binder and mixture that were added with rejuvenator. For instance, a soybean-derived rejuvenator in PG 58-28 bitumen and extracted RAP binder [5]. The results showed that the rejuvenator improved the low and high temperature properties of the RAP binder as well as increased the resistance to aging. Oil-based rejuvenators, i.e., petroleum-tech based, green-tech based and agriculture-tech based, enhanced the resistance to cracking of the high-RAP (65% RAP) mixtures [6]. A research on effects of rejuvenator from vegetable origin to asphalt mixtures containing various amounts of RAP [7]. It was found that the fatigue performance of asphalt mixtures that contained 40% or more RAP contents were improved with the addition of the rejuvenator. Meanwhile, an improvement in rutting and fatigue cracking resistance in 100% RAP mixtures with the addition of different
rejuvenators namely, waste vegetable oil, waste vegetable grease, organic oil, distilled tall oil, and aromatic extract and waste engine oil [8]. Similar results were also found, wherein the RAP mixtures rejuvenated by waste engine oil (WEO) and waste vegetable oil (WVO) implied less brittleness and high durability compared to RAP mixtures without rejuvenator [3]. Rejuvenated RAP mixtures exhibited high resistance to cracking and rutting compared to the control mixture [9]. The inclusion of the rejuvenators in RAP mixtures has also lowered the susceptibility of the mixtures to moisture. They also mentioned that the performance of rejuvenated RAP mixtures could be affected by the amounts of rejuvenator added in the RAP mixtures. Hence, optimum amounts of rejuvenator must be obtained to properly improve the performance of the RAP mixtures. Based on the previous studies, it can be seen that most of the rejuvenators that were evaluated in RAP mixtures are oil-based. Limited research has been conducted incorporating water-based rejuvenator in the performance of asphalt mixture containing RAP. Therefore, this study investigated the mechanical performance of recycled mixture using oil-based and water-based as the rejuvenating agent. The recycled mixture was comprised of 60% fresh material and 40% RAP. The selected percentage was based on the previous study by [10]. The optimum content of rejuvenating agents was obtained by carrying out penetration, softening point and viscosity tests on the RAP binder prior to use in the preparation of RAP mixtures. Subsequently, the mechanical performance tests, i.e., Marshall stability and flow, indirect tensile strength, resilient modulus and dynamic creep tests, were then performed on the rejuvenated RAP mixtures.

2. Materials and methods

2.1. Materials

2.1.1. Reclaimed asphalt pavement (RAP). Reclaimed asphalt pavement (RAP) used in this study was collected from hot mix asphalt (HMA) surface layer, i.e., Secondlink Highway, Johor, which have been exposed to environment and traffic for twenty years.

2.1.2. Bitumen. Bitumen PEN 60/70 was the conventional bitumen that was used as the control sample. Table 1 shows its physical properties.

| Description                  | Result | Requirement | Specification |
|------------------------------|--------|-------------|---------------|
| Penetration at 25 °C (dmm)  | 66.4   | 60-70       | ASTM D5       |
| Softening point (°C)        | 47.5   | 49-56       | ASTM D36      |
| Viscosity at 135 °C (Pa·s)  | 0.12   | <3 Pa·s     | ASTM D4402    |

2.1.3. Rejuvenating agents. The rejuvenating agents used in this study were oil-based (OB) and water-based (WB). The OB and WB rejuvenators were prepared using CECABASE® and bitumen emulsion, respectively, at different contents in order to obtain optimum amounts suitable to be used in the asphalt mixtures. For the OB rejuvenator, 0.3, 0.5 and 1.0% of CECABASE® was added into the mixture of bitumen and then mixed manually since the mixture incorporated very little amount of CECABASE®. As for the WB rejuvenator, soap solution was prepared by combining the acid, emulsifier and water. After that, the soap solution were mixed with fresh bitumen in colloid mill to produce bitumen emulsion. Next, the bitumen emulsion at content of 35, 40 and 50% was blended with RAP binder by using high shear mix for one hour at the speed of 500 rpm. The amounts of OB and WB rejuvenators added by the total weight of 60% bitumen PEN 60/70 and 40% RAP binder.
2.1.4. Aggregate. Fresh granite aggregate used in this study was obtained from MRP Quarry in Johor, Malaysia. The AC 14 gradation was designed with 60% fresh granite and 40% RAP. The gradation is based on Public Works Department (PWD) specification [11].

2.2. Methods
Two phases of work were carried out in this study. First phase involved the bitumen properties tests to determine the optimum content of rejuvenator suitable to be added in the asphalt mixture. Subsequently, the performance of the asphalt mixture containing RAP and rejuvenating agents was conducted in the second phase. Figure 1 shows the experimental design of this study.

| P | H | A | S | E | I |
|---|---|---|---|---|---|
| Mixing: | 1. 40% RAP binder + 60% PEN 60/70+ oil-based (OB) rejuvenating agent (0.3, 0.5, 1.0 %) | Bitumen properties test: | 1. Penetration | Determine optimum content of rejuvenating agents |
| | 2. 40% RAP binder + 60% PEN 60/70+ water-based (WB) rejuvenating agent (35, 40, 50 %) | 2. Softening point | 3. Viscosity |
| Performances tests: | 1. Marshall stability and flow | Type of mixtures: | 1. Control: 100% fresh aggregates | Preparation of AC14 asphalt mixture at optimum bitumen content (OBC) |
| | 2. Indirect tensile strength | 2. RAP-OB: 40% RAP + 60% fresh aggregates + OB rejuvenator |
| | 3. Resilient modulus | 3. RAP-WB: 40% RAP + 60% fresh aggregates + WB rejuvenator |
| | 4. Dynamic creep | |
| | 5. Asphalt pavement analyser (APA) | |
| Figure 1. Experimental design.

2.2.1. Bitumen properties test. Bitumen properties tests, i.e., penetration, softening point and viscosity were then performed to determine the optimum content of OB and WB suitable to be used in the preparation RAP mixture.

Penetration test was carried out in accordance with ASTM D5 [12]. The blend bitumen was poured into a penetration cup. The sample was cooled, then placed in the water bath at 25 °C for a minimum of 1 hour prior to testing. The sample was then subjected to 100 g loads for 5 seconds during the test. Average of three readings were taken for each sample.

Softening point test was conducted according to ASTM D36 [13]. Bitumen was softened and poured into the rings and cooled approximately for 30 minutes. After that, the rings ball centering guides were placed on the ring holder in a liquid bath. Steel balls were then positioned on the sample. The sample was heated and the temperature at which the bitumen touches the base plate was recorded.

Viscosity test was performed according to ASTM D4402 [14]. The sample of bitumen was heated and poured in the sample chambers. Cylindrical spindle number 27 was then submerged in the sample in order to measure the torque required to maintain a constant rotational speed of 20 RPM at a constant temperature. In this study, the test was carried out at 135 °C and 165 °C in regards to the construction temperature.

2.2.2. Preparation of asphalt mixture containing RAP and rejuvenating agents. The control and RAP mixtures containing OB and WB were prepared. The control mixture was prepared using 100% fresh aggregates. Meanwhile, RAP mixture was produced by mixing 60% fresh aggregates, 40% RAP and bitumen PEN 60/70 [10]. The optimum content of the OB and WB that was obtained from the bitumen properties tests was then added into the RAP mixtures. Since RAP consists of aged bitumen, the combination of bitumen PEN 60/70 and RAP binder need to be equivalent to the optimum bitumen
content (OBC). The OBC of AC 14 asphalt mixture was 5.3% and the bitumen content for 40% RAP was 2.14%. Therefore, the amount of bitumen PEN 60/70 needed was 3.16%, which produced an OBC of 5.3%.

2.2.3. Marshall test. Marshall Test was conducted to analyse the stability, flow, density, void in total mix (VTM), void filled with bitumen (VFB) and stiffness of the control and RAP mixtures. The Marshall stability and flow test was carried out according to ASTM D6927 [15]. The prepared mixtures were submerged in the water bath at 60 °C for 40 minutes prior to testing. The mixture was then subjected to compressive loads at 50.8 mm/min, which was recorded as stability, while the deformation that occurs simultaneously due to the loads was taken as flow.

2.2.4. Indirect tensile strength test (ITS). Indirect Tensile Strength (ITS) was used to measure the cracking potential of the mixture. The test was conducted according to ASTM D6931 [16]. The mixture was placed in a plastic bag and conditioned in 25 °C of water for 45 minutes prior to testing. The mixture was then subjected to loading along its diameter plane at a constant rate of 50 ± 5 mm/min. This loading configuration develops a relatively uniform tensile stress perpendicular to the direction of the applied load along the vertical diameter plane. ITS was then calculated using the equation (1).

\[ S = \frac{2000P}{\pi t D} \]  

where S is the indirect tensile strength (kPa), P is the maximum load (N), t is the specimen height immediately before test (mm) and D is the specimen diameter (mm).

2.2.5. Resilient modulus test. Resilient modulus test was used to measure the recoverable strain of a mixture under repetitive stress. This test was performed in accordance with ASTM D4123 [17]. The mixture was conditioned in the Universal Testing Machine (UTM) for a minimum of 4 hours before being tested. The test was conducted at 25 and 40 ºC to simulate the resistance of the mixture to fatigue cracking and rutting, respectively [18]. The resilient modulus value was then obtained by the software using equation (2).

\[ M_R = \frac{P}{Ht} (0.27 + \mu) \]  

where \( M_R \) is the resilient modulus (MPa), \( P \) is the applied load (N), \( H \) is the horizontal deformation (m), \( t \) is the sample thickness (m) and \( \mu \) is the Poisson ratio.

2.2.6. Dynamic creep test. Dynamic Creep was conducted to evaluate the rutting resistance of the mixture. This test was carried out according to BS EN 12697-25 [19]. The mixture was conditioned at 40 ºC in Universal Testing Machine (UTM) for 4 hours prior to testing. The test was performed for 3600 cycles. From this test, the creep stiffness modulus and creep strain slope (CSS) were calculated using equations (3) and (4).

\[ E = \frac{\sigma}{\varepsilon} \]  

\[ CSS = \frac{\log \varepsilon_{3600} - \log \varepsilon_{2000}}{\log 3600 - \log 2000} \]
where $E$ is the creep stiffness modulus (MPa), $\sigma$ is the applied stress (kPa), $\varepsilon$ is the cumulative axial strain at 3600 cycles (mm), CSS is the creep strain slope, $\varepsilon_{3600}$ is the strain at 3600 cycles and $\varepsilon_{2000}$ is the strain at 2000 cycles.

2.2.7. Asphalt Pavement Analyser (APA). Asphalt pavement analyser (APA) was used to simulate the permanent deformation or rutting of asphalt mixture under repetitive wheel loads. This test was conducted according to NCHRP Report No. 508 [20]. The mixture was prepared with a diameter of 150 mm and a height of 75 mm which compacted to 4.0± 0.5% air voids. Then, the mixture was conditioned at 60 °C prior testing for a minimum of 6 hours. The test was carried out under dry condition with the applied wheel load of 445 N for a maximum of 8000 cycles.

3. Results and discussion

3.1. Determination of optimum rejuvenator content

Figure 2 shows the results of penetration test for OB samples at different percentages, which were 0.3%, 0.5% and 1%. It can be seen that the penetration increased with the increased content of OB. The results indicated that the bitumen softens with the increasing of the OB contents. OB-0.3% and 0.5% obtained very low penetration values of approximately 31 dmm and 44 dmm, respectively. Meanwhile, OB-1% obtained the high penetration value of 77 dmm, which exceeded the range of 60-70 dmm. Hence, the middle value of conventional bitumen, 65 dmm, was used to obtain the optimum OB content, which was 0.82%. Meanwhile, Figure 3 presents the results of penetration test for WB samples at 35%, 40% and 50%. As can be seen, the penetration increased up to 40% WB and then decreased for 50% WB. The 40% WB attained higher penetration value compared to the 30% and 50% WB. Therefore, the recorded optimum WB content at the peak was 47.6 dmm, which indicated softer bitumen compared to other percentages.

![Figure 2. Penetration value of OB.](image)

Control Bitumen

Penetration value at 25 °C (dmm)

Cecabase content (%)
The softening point results of different content of OB was shown in Figure 4. It can be seen the trend of softening point was in contrast with the trend of penetration. The trend showed decrease with the increase of the OB content. The increase of OB content resulted in lower softening point value which indicated softer bitumen. Thus, the optimum content of WB, which was 0.70%, was chosen based on the softening point of the conventional bitumen. Subsequently, Figure 5 shows the softening point results of WB. The softening point decreased as the WB content increased until 40% WB. At 50% WB, the softening point increased slightly higher than the 40% WB. Softer bitumen was achieved by the 40% WB compared to the 35% and 50% WB. The resulted optimum WB content was 42% with the softening point value of 49°C.

Figure 3. Penetration value of WB.

Figure 4. Softening Point of OB.
Figure 5. Softening Point of WB.

Figure 6 shows the viscosity results of OB at temperature of 135 and 165 °C. Similar trend was observed in the viscosity results for both temperatures. At 135 °C, the viscosity decreased with increase of OB contents, while at 165 °C, the viscosity can be seen reduced to lower values than at 135 °C. This is because the bitumen tends to soften at higher temperature. By using the viscosity of the control bitumen as a reference, the optimum OB contents obtained at 135 and 165 °C were 0.84% and 0.44%, respectively. Figure 7 presents the viscosity results of WB at 135 and 165 °C. At 135 °C, the viscosity of the bitumen decreased consistently with the increased content of the WB. It can be seen that the addition of 35% WB into the bitumen has produced highest viscosity of about 1.20 Pa.s, which is even higher than the viscosity of the OB. On the other hand, at 165 °C, the viscosity decreased until 40% WB and then remained constant at 50% WB. This showed that higher addition of the WB content had no effect on the viscosity of the bitumen, especially at higher temperature. By using the viscosity of control bitumen as a reference, the optimum WB contents for 135 and 165 °C were found to be 76% and 56%, respectively.

Figure 6. Viscosity of OB.
Based on the optimum content of the rejuvenators obtained from the penetration, softening point and viscosity, the average optimum content for the OB and WB was 0.7% and 52.13%, respectively. These optimum values were then used in the performance of the asphalt mixture in subsequent section.

3.2. Marshall analysis

Marshall analysis was performed on the control, RAP-OB and RAP-WB mixtures. Figures below illustrate the results of each parameter of Marshall analysis. Figure 8 shows an increasing trend of VTM of the mixtures. VTM indicates amount of air voids in the compacted mixture. As can be seen, RAP mixtures contained higher air void content compared to the control mixture and the highest VTM was exhibited by RAP-WB. However, the VTM values remained in the specification range of 3 -5%. VTM that is too high can lead to moisture damage and aging of the mixture, while VTM that is too low resulted in bleeding and plastic deformation [21]. Hence, RAP-OB mixture can be seen to attain a moderate value of VTM. The results of VFB was shown in Figure 9. The values were in the specification range between 70 and 80%. It can be seen VFB decreased from control to RAP-OB mixtures and then increased for RAP-WB mixture. As a result, RAP-OB mixture exhibited optimum value of VFB. The result of VFB was in contrast with the result of VTM, whereby high VFB has led to low VTM and vice versa. This implied that amount of VFB affects the VTM of the mixture.
Figure 10 presents the result of stability of the mixtures. As can be seen, RAP-OB mixture exhibited highest stability compared to the control and RAP-WB mixtures. Meanwhile, the stability of RAP-WB was lower than the control sample. High stability of RAP-OB could be due to its moderate air void content. It displayed adequate densification which has high strength to withstand the applied loads. As for the RAP-WB mixture, the stability seems to be affected since it showed lower stability compared to the control mixture. This could be due to higher VTM of the RAP-WB mixture compared to control and RAP-OB mixtures. In addition, the WB rejuvenator might be a factor in the reduction of the stability, wherein the WB rejuvenator increases lubrication and movement of the aggregates, in which the ability of the bitumen to hold the aggregates together was reduced. The stability, however, satisfied the requirement of PWD since the values exceed 8000 N.
Figure 11 shows the results of flow. Flow was obtained simultaneously with the stability of the mixture in which flow measures the vertical deformation of the mixture which subjected to the applied load. It can be seen that the flow for both of the RAP-OB and RAP-WB was higher than the flow of the control mixture. However, the flow of the RAP-OB did not satisfy the PWD requirement wherein the value exceeded the range between 2 and 4 mm. Consequently, the stiffness of the mixtures were obtained from the ratio of stability and flow. Figure 12 shows the results of stiffness. A decreasing trend was observed in the stiffness of mixtures whereby control mixture depicted the highest stiffness followed by RAP-OB and RAP-WB mixtures. The control mixture obtained the highest stiffness even though it exhibited the lower stability than the RAP-OB mixture. This was attributed to low flow value of the control mixture compared to the RAP-OB and RAP-WB mixtures. Nevertheless, the stiffness of the RAP mixtures was still acceptable since the value was greater than 2000 N/mm.

![Figure 10. Stability vs mixture.](image1)

![Figure 11. Flow vs mixture.](image2)
3.3. Indirect tensile strength (ITS)

Figure 13 presents the result of ITS at 25 °C. It can be seen that the RAP mixtures exhibited higher tensile strength compared to the control mixture. The addition of OB and WB in the mixture improved the tensile strength by about 41% and 32%, respectively, compared to the control mixture. Higher tensile strength of the RAP mixtures depicted higher resistance of the mixtures to cracking. Apart from that, the highest tensile strength of the RAP-OB mixture could be attributed to the high stability of the mixture which led to high internal strength of the mixture towards the impact loads. It also exhibited moderate stiffness of the mixture compared to the control and RAP-WB mixtures.

Figure 12. Stiffness vs mixture.

Figure 13. Indirect tensile strength.
3.4. Resilient modulus

Figure 14 illustrates the resilient modulus results of the control, RAP-OB and RAP-WB mixtures. Similar trend of resilient modulus was observed at 25 and 40 °C. It can be seen that the RAP mixtures have higher resilient modulus compared to the control mixture for both temperatures. At 25 °C, RAP-OB and RAP-WB mixtures improved about 94% and 43%, respectively, compared to control mixture. Moreover, RAP-OB recorded higher resilient modulus than RAP-WB by about 35%. Higher resilient modulus at 25 °C indicated that the RAP mixtures have higher recoverability to return to the original condition after being loaded. This showed that the RAP mixtures have higher resistance to fatigue cracking at intermediate temperature. In addition, the high resilient modulus of RAP mixtures implied that the mixtures were stiffer and have higher resistance to rutting compared to the control mixture. This can be seen for resilient modulus at 40 °C, wherein RAP-OB and RAP-WB mixtures increased about 95% and 45%, respectively, higher than the control mixture. Apart from that, RAP-OB mixture still showed greater performance than RAP-WB mixture about 35%. Since RAP contained hardened bitumen compared to fresh bitumen, it might lead to high susceptible to fatigue cracking at lower temperature. Nevertheless, the high resilient modulus at 25 °C could help the mixture to withstand the rutting at higher temperature of 40 °C better than the control mixture. Thus, RAP-WB significantly enhanced the resistance of the mixture to fatigue cracking and rutting.

![Figure 14. Resilient modulus.](image)

3.5. Dynamic creep

Figure 15 shows the results of stiffness modulus and its relationship with creep stiffness slope (CSS). As can be seen, higher stiffness of the mixtures exhibited lower CSS and vice versa. The RAP mixtures implied higher stiffness modulus and lower CSS value compared to the control mixture. It was found that RAP-OB mixture has highest stiffness modulus of 783 MPa followed by RAP-WB and control mixtures, which were 469 and 303 MPa, respectively. The highest stiffness modulus of the RAP-OB mixture of about 158% relative to control mixture has led to lower CSS value, which was 0.16. RAP-WB also exhibited similar CCS of 0.16, while the CSS of the control mixture was 0.44. These results coincided with other performances of asphalt mixture wherein the RAP attained higher performance compared to the control mixture. Apart from that, the addition of WB can be seen reduce the stiffness of the mixture, thereby increasing the CSS during the applied repetitive stress. Consequently, the mixture

![Diagram](image)
exhibited lower resistance to rutting compared to mixture rejuvenated with OB. Nevertheless, RAP-WB still performed higher than the control mixture.

![Figure 15. Relationship of stiffness modulus and CSS.](image)

**Figure 15.** Relationship of stiffness modulus and CSS.

### 3.6. Asphalt pavement analyzer (APA)

Figure 16 shows the results of APA for control, RAP-OB and RAP-WB mixtures. The results showed that the rut depth values for all the mixtures were less than 8 mm which met the value recommended by the NCHRP Report No. 508 [20]. As can be seen, RAP-OB mixture attained the lowest trend of rut depth followed by RAP-WB and control mixtures. This indicated that RAP-OB mixture has highest stiffness and produced lesser rut depth about 66% lower than the control mixture. Meanwhile, the rut depth of RAP-WB mixture increased about 28% compared to RAP-OB. This revealed that the inclusion of WB rejuvenator in RAP mixture reduced the stiffness of the RAP binder and increased the rut depth.

![Figure 16. Average APA results for control and RAP mixtures.](image)

**Figure 16.** Average APA results for control and RAP mixtures.
4. Conclusions
The following conclusions can be drawn from this study:

1. Based on the penetration, softening point and viscosity tests, the optimum content of OB and WB rejuvenators to be added into the recycled mixture was 0.70% and 52.13%, respectively.
2. The Marshall stability and flow, indirect tensile strength, resilient modulus, dynamic creep and asphalt pavement analyser tests showed that the RAP-OB and RAP-WB mixtures exhibited higher performances compared to control mixture.
3. The stiffness of the RAP binder was significantly reduced by the incorporation of the WB rejuvenator relative to OB rejuvenator. Consequently, the mechanical performances of the RAP-WB were lower than the RAP-OB.

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