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RESEARCH PAPER

Rheological and Microstructural Investigation of the Effects of Rejuvenators on Reclaimed Asphalt Pavement Bitumen by DSR and AFM

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Abstract
Utilization of RAP in asphalt pavements may lead to severe quality problems, mostly due to asphalt aging phenomenon. Within the scope of this study, the use of rejuvenators to improve the properties of RAP is investigated. Two different types of waste oils together with a commercial rejuvenator are used. The rejuvenated bitumen samples are evaluated from both rheological and microstructural aspects. In addition, mixture testing also employed to investigate the effectiveness of used rejuvenators. Experimental studies on bitumen samples included conventional and Dynamic Shear Rheometer (DSR) testing and Atomic Force Microscopy (AFM) imagings. The results unveiled that, the utilization of rejuvenators can significantly improve the quality of recycled asphalt and threatened the aged bitumen quality. Missing bee-structures of aged bitumen samples evolve in a different type of surface topography while the mixtures performance evolves in terms of workability. The rejuvenation process provides viscous behavior to aged bitumen. The Rejuvenation Index (RI) which is a dimensionless value, simply evolves this fact. While the RI of the non-rejuvenated aged bitumen is taken as 1 (one) as per RI definition, the neat bitumen RI is calculated as 0.79, the RI of waste oils are calculated as 0.74 and 0.79 while the commercial sample RI is calculated as 0.60. Among the rejuvenators, waste oils showed similar results with almost same volume of peri- and para-phase since the commercial rejuvenator behaved more similar to neat bitumen. The rejuvenated samples workability performance improves using rejuvenating additives. Binder and mixture testing demonstrated high consistency and conformity with each other in terms of performance and aging characteristics. The results proved that, waste oils can be used as rejuvenating agents within RAP. Rejuvenators can significantly restore the properties of aged RAP bitumen and increase the amount of RAP in bituminous mixtures.

Keywords Reclaimed asphalt pavement · Rejuvenator · Bitumen aging · Atomic force microscopy · Dynamic shear rheometer

1 Introduction

Reclaimed Asphalt Pavement (RAP) is old flexible material that can be gained through different methods such as; cold milling or ripping [1–3]. RAP materials can be used in a wide range of applications in road construction. RAP can be utilized as backfill material in embankments or can be reutilized in various courses of flexible pavements [4–6]. Depending on the area of application, re-utilizing of RAP helps significant savings [7].

Recent technologies introduce innovative methods in recycling of RAP [8]. It is important to designate the best valuable field of use for recycling of RAP in pavement engineering. Different studies have addressed plenty of

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areas of application for RAP involving the optimum amount within the mentioned area [9]. In addition to scientific studies, private sector has developed innovative methods to use high amounts of RAP [10]. Although private sector technologies are controlled and confirmed by authorities in accordance with specifications, these technologies are not usually published for public use due to know-how concerns.

As RAP contains both aggregates and bitumen, recycling of RAP helps saving significant amounts of natural resources. Recycling of RAP is known as an eco-friendly technology. Recycling of RAP provides also many economical benefits for pavement industry [11]. Recycling methods can generally be categorized as Cold and Hot recycling [9]. Cold recycling of bituminous mixtures involves RAP, water and a recycling agent involving mixing of all without applying heat. This is possible generally by use of emulsions. Hot recycling implements these materials generally through mixing with recycling agents in presence of heat. Both methods can be executed in plants or in-place. The common point in most recycling methods is presence of recycling agents which help easier application and mixing. Use of rejuvenators, also called recycling agents, is one of the applicable methods introduced to facilitate the use of RAP especially within the upper courses of flexible pavements such as binder and surface courses [12]. Various kinds of rejuvenators have been introduced to asphalt recycling industry so far, some commercialized and some still remained within the literature. These rejuvenators mostly are used in the hot recycling applications. Many of rejuvenators are oily basis since it is believed that, aged asphalt pavement mainly lacks the oily components of bitumen disappeared during the service life [13].

Efficient utilization of raw materials with economic issues is the main objective to increase the amount of RAP in bituminous mixtures. The objective of this study is to seek for innovative recycling agents mainly originated from waste oils. In this regard, two kinds of waste oils available in Turkish market have been evaluated and compared to a commercial recycling agent. Waste Engine Oil (WEO) and Waste Vegetable Oil (WVO) and a Commercial Recycling Agent (CRA) are the studied agents.

2 Experimental

2.1 Materials

The bitumen used in this study was chosen based on penetration grading in line with Turkish highways specifications. In selection of the penetration grade, the region in which the study took place was taken into consideration. The region from which the RAP material reclaimed is Izmir, Turkey. The bitumen was provided from TÜPRAŞ Aliağa Oil Terminal of Turkish Petroleum Refinery Corporation, used as the fresh bitumen. This grade of penetration is imperatively used in Aegean region due to the climatic conditions. In Aegean region, the summers are hot, arid, and clear and the winters are long, cold, wet, windy, and partly cloudy. Over the course of the year, the temperature typically varies from 4 to 35 °C and is rarely drops below 0 °C or raise above 38 °C. Thus, 50/70 penetration grade bitumen is used regionally. Conventional test results for virgin bitumen conducted at Dokuz Eylul University laboratory of bituminous materials are given together with the specifications limits in Table 1 [14–19].

The aged bitumen samples were obtained through extracting bitumen from a grinded asphalt at the end of its service life. It is important to ensure that the aged bitumen and RAP characteristics counter to the real field conditions in terms of durability and traffic loads. Conventional test results for extracted RAP bitumen are presented in Table 2 [14–16].

RAP can be used as raw material in the construction of newer roads or can be rejuvenated and used over and over again. The point is that during the service life of a pavement, asphalt binder and aggregates are subject to many physical and also chemical changes, which can highly affect the quality of the whole mixture. Therefore, the use of RAP requires paying particular attention and other extensive tests to ensure that, the target mixture is sufficient in terms of specifications. Many specifications would

Table 1 Laboratory test results for virgin bitumen

Test	Specification	Results	Specification limits
Penetration (25 °C; 0.1 mm)	ASTM D5/D5M-13	63	50–70
Softening point (°C)	ASTM D36/D36M-12	49.7	46–54
Viscosity at (135 °C)-Pas	ASTM D4402	0.425	–
Viscosity at (165 °C)-Pas	ASTM D4402	0.1	–
Rolling thin film oven (163 °C)	ASTM D1754/D1754M-09		
Change of mass (%)		0.05	0.5 (max)
Retained penetration after RTFO (%)	ASTM D5/D5M-13	74	50 (min)
Softening point rise after RTFO (°C)	ASTM D36/D36M-12	4.5	7 (max)
Specific gravity	ASTM D70-09e1	1.038	–
Flash point (°C)	ASTM D92-12b	+ 260	230 (min)

Table 2 RAP bitumen conventional test results

Test	Specification	Results
Penetration (25 °C; 0.1 mm)	ASTM D5/D5M-13	38
Softening point (°C)	ASTM D36/D36M-12	61
Viscosity at (135 °C)-Pas	ASTM D4402	0.538
Viscosity at (165 °C)-Pas	ASTM D4402	0.188

not be adequate enough for use of RAP, since many of them have not been designed to include the usage of old pavement materials. For this reason, highway authorities in so many countries have skeptical view on high recycling material contents. It is important to characterize RAP materials before putting it to use. Reclaimed type I wearing surface course, subjected to traffic loads for a period of 12 years has been used as RAP material. The original RAP contains 50/70 penetration grade bitumen since it is mandatory to use this penetration grade in the region that RAP was reclaimed. 16 × 1000 gr. of batch samples were selected randomly using a random separator. The bitumen content and the gradation of RAP materials were determined. Aged bitumen was extracted and distilled using a laboratory type extractor and distillatory. According to the test results, the bitumen content of the RAP was found as 4.30%.

Three kinds of rejuvenators were used in this study. All rejuvenators were selected based on the literature review carried out on the rejuvenating agents. The approach in the selection of oily additives has mainly been the employment of waste oils as asphalt rejuvenators. Two of the agents used were selected from waste oils available in the market and the third rejuvenator was chosen from several commercial products being sold as asphalt rejuvenators. The physical and chemical properties of the rejuvenators are given in Tables 3 and 4, respectively.

Table 3 The physical properties of the rejuvenators

Characteristics	WEO	WVO	CRA
Color	Dark brown	Straw yellow	Amber
Density (g/mL)	0.91	0.92	0.9
Viscosity @ 40 °C, mm^2/s (centistokes)	90	50	50
Viscosity @ 100 °C, mm^2/s (centistokes)	15	10	15
Flash point (°C)	≥ 200	≥ 280	≥ 140
Boiling point (°C)	≥ 280	≥ 250	≥ 350
Pour point (°C)	≤ -25	≤ 10	≤ 0
Freeze point (°C)	N/A	≤ 5	≤ -10

Table 4 The chemical properties of the rejuvenators

Compound	Content
WEO	
Arsenic	< 1.0 ppm
Ash	0.66%
Cadmium	0.28 ppm
Chromium	< 4.0 ppm
Lead	14.0 ppm
Sulfur	0.19%
Total Halogen	396 ppm
WVO	
Oleic acid	43.6%
Palmitic acid	38.35%
Linoleic acid	11.39%
Stearic acid	4.33%
Myristic acid	1.03%
CRM	
Methyl oleate	30–40%
Free fatty acid	10–20%
Triglyceride	30–45%

2.2 Methodology

Basically, there are several steps which can be followed to evaluate the rejuvenation process of the aged binder. The first step is to determine the optimum content of rejuvenating agent, which is supposed to enhance the properties of aged RAP bitumen. Besides, it is important to develop a blending process which has to be similar to the process applied in the plant or field. To determine the optimum rejuvenator content, some evaluation tests should be conducted both on aged and rejuvenated RAP bitumen together with the virgin bitumen.

Comparative study of binder tests results can help to understand the effect of the rejuvenation process on aged RAP bitumen. The first step should be the designation of an experimental plan consisted of appropriate binder testing, suitable to exhibit the differences between rejuvenated and non-rejuvenated bitumen samples. Within the study, both conventional and rheological evaluations of bitumen samples have been employed to fully understand the effect of rejuvenating agents on aged RAP bitumen. Additionally, AFM microscopy was employed to investigate the microstructural impacts of rejuvenators on aged RAP bitumen.

2.3 Determination of Optimum Rejuvenator Content

Many studies suggest the use of penetration test in determination of optimum rejuvenating agent [20–22]. The optimum content of rejuvenators in the modified RAP bitumen was determined as the target content to obtain a rejuvenated bitumen sample having the same penetration value of virgin bitumen sample. In other words, when the RAP bitumen is modified with this rejuvenating additive content, the acquired sample should have the same penetration value of virgin bitumen sample. To perform this task, RAP bitumen was modified with various contents of each rejuvenator. The range was chosen based on literature review and preliminary studies [23]. The modification was processed for 5 min at 140 °C using a laboratory blender at normal shear rates (700 rpm) to obtain a homogenous rejuvenated bitumen sample. This modification method has been figured out as adequate in terms of time, temperature and shear rate, since longer times and/or higher temperatures and shear rates may cause the volatilization of rejuvenating agents. This is also true for production of recycled RAP in plants due to short-term aging issues.

Following the determination of optimum rejuvenator contents, samples were prepared with the determined contents with ± 1 and ± 2% variation by weight of extracted RAP bitumen. In this regard, samples containing 3, 4, 5, 6, and 7% of WEO and WVO by weight of aged RAP bitumen together with samples containing 5.5, 6.5, 7.5, 8.5, and 9.5% of CRA by weight of aged RAP bitumen were prepared and classified as rejuvenated bitumen samples. All rejuvenated samples and aged RAP bitumen together with the virgin bitumen were tested for the conventional bitumen tests. Conventional bitumen tests included penetration test, softening point tests before and after RTFO together with rotational viscosity test.

2.4 Rheology Investigation

Rheological tests to investigate the behavior of rejuvenated, non-rejuvenated samples, and virgin bitumen were performed in a controlled-stress Malvern Dynamic Shear Rheometer (DSR) Asphalt Rheometer (developed by Malvern Instruments®). Complex modulus, which is defined as the ratio of maximum shear stress to maximum strain and provides a measure of the total resistance to deformation when the bitumen is subjected to shear loading and phase angle, which is the phase difference between shear stress and strain in an oscillatory test at different temperatures, were recorded automatically during the test. Using these two values, rutting parameter ($G^*/\sin(\delta)$) and fatigue parameters ($G^*.\sin(\delta)$) can be calculated. With the scope of this study, Performance Grade (PG) upper limit value of the PMB samples were defined by determining the maximum temperature at which $G^*/\sin(\delta)$ value is more than 1 kPa for rejuvenated samples and virgin bitumen. PG tests were conducted on samples containing optimum contents of rejuvenators.

2.5 Microstructural Investigation

The AFM microscopy was employed to investigate the differences on rejuvenated, non-rejuvenated samples, and virgin bitumen. All tests were conducted with Nanosurf ECS 204 electrochemistry stage imaging device and an extra isostage controller for active vibration isolation. The system uses a software equipped to imaging technology. Multi 75E-G silicon probes with 75 Hz. resonant frequency and 3 N/m force constant coated with Cr/Pt conductive coating were used through the tapping method. This device allows performing simultaneous AFM imaging and electrochemical measurements on electrodes and various samples.

3 Results and Discussion

3.1 Penetration Test Results

The penetration value at 25 °C was recorded as 63 for the virgin bitumen. The test results meet the Turkish specification limits in terms of mass change, retained penetration and softening point after RTFO. The same tests were also applied on rejuvenated samples with optimum contents of rejuvenating agents to make sure that, these samples also meet the long-term criteria as per national specifications.

The results showed that, the penetration value of extracted RAP bitumen is lower than the penetration value of neat bitumen. After rejuvenating the RAP bitumen samples, the penetration values were obtained. The results are presented in Fig. 1.

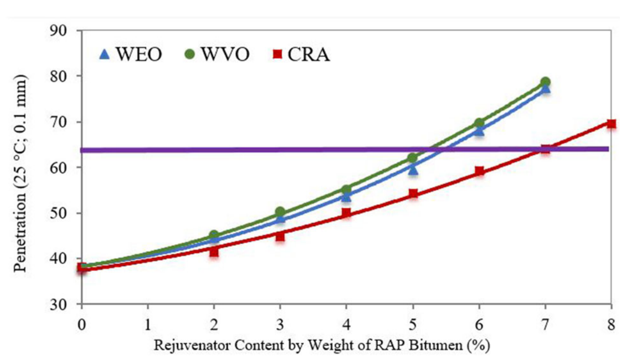

Fig. 1 Penetration values corresponding rejuvenator contents

As shown in Fig. 1, the penetration value increases as a result of rejuvenation process. WEO and WVO additives yielded similar results with increasing penetration value. The slope of increasing for CRA was detected to be slightly lower than WEO and WVO. It is possible to derive the desire penetration value by extracting the formula or through interpolation method. It can be concluded that, the desired penetration value, which is the penetration of virgin bitumen (63), can be achieved by making necessary alteration in additive content. On this regard, the optimum rejuvenating contents were calculated as 5.4% for WEO, 5.1% for WVO, and 6.8% for CRA. Following the calculations, the samples containing the optimum rejuvenating values were produced and tested for all conventional tests. The most concerned issue in bitumen rejuvenation is that, if the rejuvenated samples can meet the long-term criteria or not. The results showed that the rejuvenated samples can meet all Turkish criteria indicated within the specifications as can be seen in Table 5.

Many studies have developed aging indices based on the ratio of aged bitumen sample viscosity to the viscosity of neat bitumen [24, 25]. Moreover, the bigger the aging index, the more aged the sample is. According to this theory, it should be possible to define the rejuvenating indices based on the viscosity test results for rejuvenated samples. The rejuvenating indices are calculated as the ratio of the viscosity value of a rejuvenated sample over the viscosity of aged RAP bitumen at a defined temperature (135 °C). Therefore, it is possible to say that, the increase in the rejuvenating indices brings in the increased workability to the sample. Table 3 represents the rejuvenating indices and conventional test results of non-rejuvenated, optimally rejuvenated RAP samples, and the virgin bitumen. Figure 2 shows the rejuvenation indices for all rejuvenated samples produced using the optimum amounts of rejuvenating agents. As the denominator of the index is viscosity value of RAP bitumen, ratio 1 indicates that bitumen sample rejuvenation has not done yet. The ratio of neat bitumen viscosity value over the viscosity value of aged bitumen sample indicates the normal deviation. The other indices can be interpreted having these values on hand.

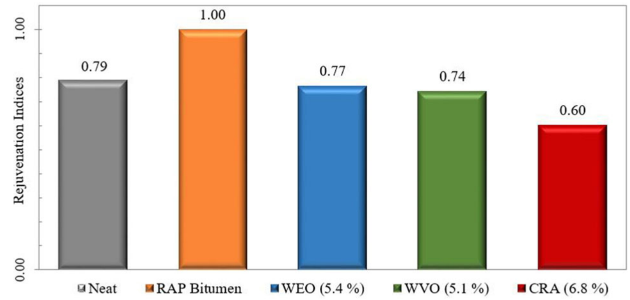

Fig. 2 Rejuvenation indices for optimally rejuvenated samples

As can be seen in Fig. 2, using optimum amount of CRA, it is possible to achieve a bitumen sample, which is more convenient to process in terms of mixing and compacting compared to other samples including neat bitumen. Optimum WEO and WVO samples have similar rejuvenating indices which can be explained as the similar behavior in terms of viscosity. All rejuvenated samples have lower rejuvenation indices compared to neat bitumen.

Table 5 Rejuvenating indices and conventional test results for optimum rejuvenating contents

Test	Specification	Bitumen Samples					Specification limits
		Neat	RAP Bitumen	WEO (5.4%)	WVO (5.1%)	CRA (6.8%)	
Penetration (25 °C; 0.1 mm)	ASTM D5/D5M-13	63	38	63	63	63	50–70
Softening point (°C)	ASTM D36/D36M-12	49.7	61	50	51.5	50.5	46–54
Penetration Index	–	− 0.72	0.57	− 0.65	− 0.27	− 0.52	
Viscosity @ 135 °C (Pas)	ASTM D4402	0.43	0.54	0.41	0.4	0.33	–
Rolling thin film oven (163 °C)	ASTM D36/D36M-12	RTFO Aged Samples Results					
Change of mass (%)	–	0.05	N/A	0.12	0.1	0.08	0.5 (max)
Retained penetration after RTFO (%)	ASTM D5/D5M-13	74	N/A	53	59	61	50 (min)
Softening point rise after RTFO (°C)	ASTM D5/D5M-13	4.5	N/A	6	5	5	7 (max)

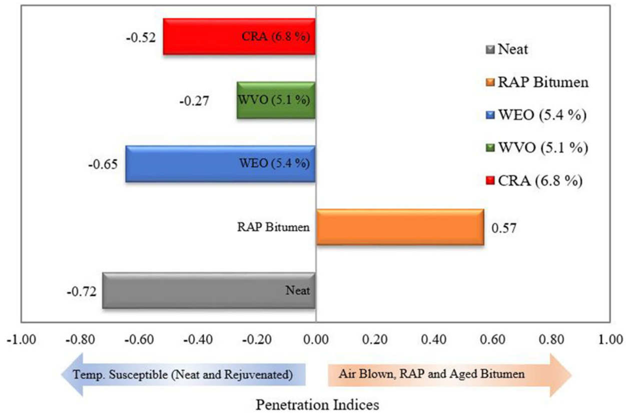

Fig. 3 Penetration indices for optimally rejuvenated samples

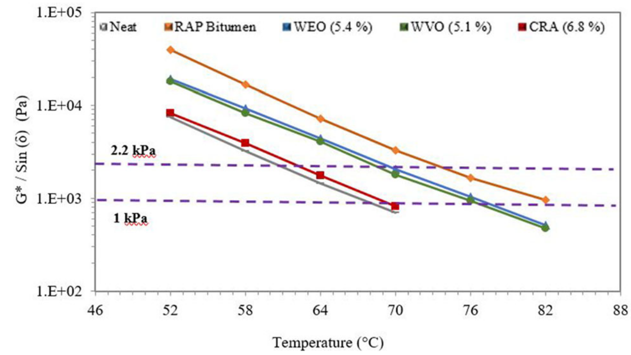

Fig. 4 PG test rutting parameters corresponding to temperature variations

Table 6 The temperatures corresponding to the 1 kPa of rutting parameters for rejuvenated samples

Rejuvenator type	TG*/sin δ (10 rad/s) corresponding 1 kPa
WEO	77.2
WVO	76.9
CRA	61.9

Based on the results, it can be said that, rejuvenated samples workability performance compared to the neat bitumen improves after rejuvenation. Penetration indices are used to evaluate the effect of aging and air blowing. Figure 3 shows the penetration indices for the all sample.

As shown in Fig. 3, the optimally rejuvenated samples behave similar to neat bitumen after rejuvenation process. Although neat bitumen penetration index is considered as the desired value among all samples, the rejuvenated samples have all been soften compared to aged RAP bitumen. The temperature susceptibility of bituminous samples depends on the penetration indices [26]. Therefore, it can be indicated, the WEO rejuvenated sample is more susceptible to temperature changes compared to other rejuvenated samples.

3.2 Rheological Test Results

The rejuvenated bitumen samples should be treated as fresh bitumen samples, since these samples shall fulfill all specifications limits. All optimally rejuvenated bitumen samples were subjected to Performance Grading (PG) test to determine PG upper limits. Although penetration grading was the reference grading system within the scope the study, performance grading was used to check the rejuvenated bitumen samples according to AASHTO T315 [27]. Complex modulus, which is defined as the ratio of maximum shear stress to maximum strain and provides a measure of total resistance to deformation while subjected to shear loading and phase angle, which is the phase difference between shear stress and strain in an oscillatory test at different temperatures, were recorded automatically during the test. Using these two values, rutting parameter (G*/sin (δ)) can be calculated. Performance Grade (PG) upper limit value of the bituminous samples was defined by determining the maximum temperature at which G*/sin (δ) value is more than 1 kPa for unaged samples (virgin bitumen and rejuvenated RAP samples) and 2.2 kPa for non-rejuvenated RAP sample. The specification upper limit of neat bitumen was confirmed as 64 °C, while the rejuvenated bitumen samples were recorded as 76, 76, and 64 °C, respectively, for WEO, WVO, and CRA rejuvenated bitumen samples. On the other hand, the upper limit of non-rejuvenated RAP bitumen was recorded as 70 °C. According to the specification, the PG temperature is determined with the 6 °C of temperature intervals. However, to compare the PG upper limits changes depending on the rejuvenator type, the temperature corresponding to the 1 kPa of rutting parameters is determined with assuming the linear relationship between temperature and rutting parameter. The results are obtained by Fig. 4 and given in Table 6.

As can be seen in Fig. 4, the rutting performance of all the samples decrease almost linearly in the logarithmic scale by increasing the temperature. Additionally, rejuvenating with CRA, WEO, and WVE gained virgin bitumen's performance to the RAP bitumen in terms of rheological characterization. The CRA rejuvenated sample behaved more similar to neat bitumen rather than other rejuvenated samples. Moreover, waste oils rejuvenated samples demonstrated a performance between neat bitumen and aged RAP bitumen in terms of rheological characteristics at higher temperatures. WVO showed better performance in terms of rheology.

The most concerned issue in bitumen rejuvenation is to determine that the newer samples can meet the long-term

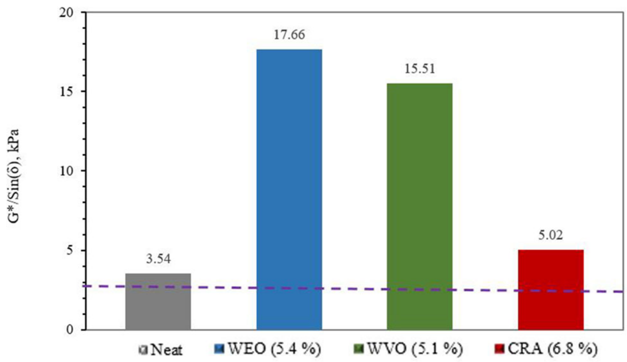

Fig. 5 PG check of RTFO aged rejuvenated samples and virgin bitumen

criteria or not. The rejuvenators used in this study are oily basis materials. For this reason, it is important to evaluate the aging effect on the final product considering the volatilization possibility of oily fractions. All rejuvenated samples were subjected to RTFO aging and rutting parameters values were checked as per AASHTO T315 specification limits. In this regard, G*/Sin (δ) shall be equal or greater than 2.2 kPa. The rutting parameter values at 64 °C of aged rejuvenated RAP samples and neat bitumen are given in Fig. 5.

As can be observed in Fig. 5, all rejuvenated and neat bitumen samples exhibited greater values than the specification limit set for after RTFO aging process. CRA rejuvenated sample performed better than the waste oils in this context. Results showed high conformity with other DSR testing results. WVO's rutting parameter was recorded slightly lower than WEO's value. This means that, WVO rejuvenated sample remains still softer than WEO rejuvenated sample following the RTFO aging process.

3.3 Microstructural Test Results

The phase images of neat bitumen are consisted of catana phase, peri-phase, and para-phase under AFM [28]. Peri-phase, which surrounds the bee-structures, is stiff, while hard para-phase is softer and more viscous among the other phases [29, 30]. Therefore, it is possible to expect the increment of para-phase by the aging, since the materials becomes to be stiffer. In the same matter, rejuvenating the aged samples increases the volume of para-phase by the addition of oily fraction into the bitumen [31]. The differences of the intensity of these two phases might be interpreted as the distinction of the rigidity and the composition of the sample. There are some previous studies revealing the increment of the contrast between these two phases occurred depending on the aging properties of the sample [32].

There are many studies related to AFM and the aging of asphalt binder which prove that the microstructure of bitumen changes through aging process [31, 33–35]. Bee structures, which is the result of wrinkling of very thin surface films on the order of 10 nm thick, characterizes areas of higher and lower stiffness than the surrounding area [36]. It is possible to observe the bee-structures in the topographic images of the bitumen depending on the amount of waxy components of the material [37]. Therefore, evaluation of the appearance of bee structure might be a proof of a good rejuvenation of RAP bitumen. For this reason, non-rejuvenated and rejuvenated RAP samples and the neat bitumen are investigated by obtaining topography images with AFM. Figure 6 show the topography and phase images of neat bitumen, non-rejuvenated and optimally rejuvenated RAP bitumen samples using tapping mode of AFM. The phase images of the samples located on the right-hand side, while the topography images on the left-hand side.

As can be seen in Fig. 6, neat bitumen has the highest volume of para-phase, which is light phase and brings the viscous behavior into the bitumen. On the other hand, non-rejuvenated RAP sample has the highest amount of peri-phase, which is dark and results in the rigidity of the sample. After the rejuvenation of the RAP bitumen, it was observed that, the amount of para-phase starts to increase for all type of rejuvenators. Therefore, it is possible to say that, rejuvenation process is succeeded and provides the viscous behavior to the bitumen lost after aging. Among the rejuvenators, WVO and WEO showed similar results with almost same volume of peri- and para-phase. However, CRA behaved more similar to neat bitumen. It can be understood that, the bitumen viscosity changes intensely through rejuvenation with CRA. Also, the consistency of rejuvenation dispersion inside of the rejuvenated bitumen samples seem to be better than WEO and WVO rejuvenated bitumen samples. These results are coinciding with the rheological results.

In the topography images taken from the samples, almost 40–50 numbers of bee-structures can be counted in 225 μm^2 of the surface of the neat bitumen sample. On the other hand, except dark and light areas, no bee-structures can be found on the surface of the aged RAP bitumen sample. However, rejuvenating the aged RAP bitumen led to appearance of micro bee-structures. Even though bee-structures are not as visible as the ones of virgin bitumen, it still can be a clue of the absorbance of waxy structure of rejuvenators by aged bitumen. A very tiny (approximately 0.5 to 1 μm) bee-structures appeared in WEO, WVO, and CRA rejuvenated bitumen sample. The new-born tiny bee-structures inside of rejuvenated bitumen are approximately half in numbers and one-third in size compared to neat bitumen sample. The disperse parts of all rejuvenated

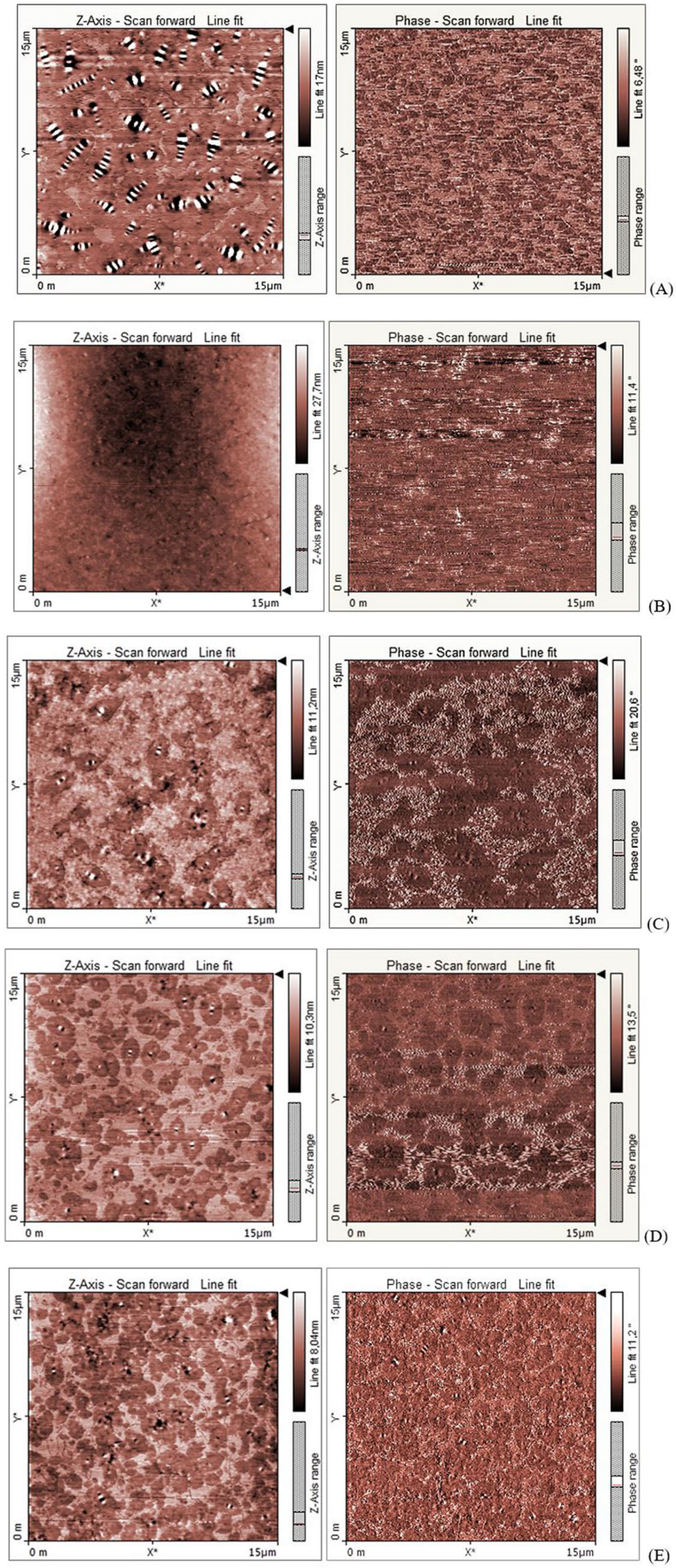

Fig. 6 Topography and phase images of all samples. **a** Neat bitumen. **b** Non-rejuvenated RAP sample. **c** WEO rejuvenated RAP sample. **d** WVO rejuvenated RAP sample. **e** CRA rejuvenated RAP sample

bitumen also differ in topography compared to aged RAP bitumen. Phase scan images show that, the material stiffness changes dramatically. Both waste oils showed similar results to the aged RAP bitumen in terms of topography and phase images. However, CRA had slightly different results especially on phase distribution.

Coming to conclusion, all rejuvenating agents change the aged bitumen micro topography and stiffness significantly. Although the neat bitumen AFM images are significantly different from rejuvenated bitumen samples but similarities in terms of stiffness and bee-structures can be observed compared to aged RAP bitumen.

4 Conclusion and Recommendation

Within the scope of this study, waste oils and a commercial rejuvenator produced locally were used to rejuvenate aged RAP bitumen. The new-born rejuvenated bitumen samples were investigated from rheological and microstructural aspects. The results showed that rejuvenating agents including commercial product and waste oils increase the penetration grade of aged bitumen to desired level. Viscosity of aged RAP bitumen also decrease in a desired manner by implementing rejuvenators. The rejuvenators are effective to restore the properties of aged RAP bitumen. WEO and WVO rejuvenating agents perform similar to each other in terms of conventional test results since CRA is more effective than waste oils. The rheological properties of aged RAP bitumen improve significantly by use of rejuvenators. WEO and WVO similarly improve the rutting performance of aged RAP bitumen, while CRA performs better. Using waste oils, although it is not actually possible to obtain a rejuvenated bitumen sample having the same rheological properties of neat bitumen, recycling additives have impressive influence on aged bitumen. CRA is more essential to restore the rheological characteristics of aged bitumen to its original properties. Although, the after RFTO aging performance of CRA rejuvenated bitumen is not at the same level with neat virgin bitumen. All rejuvenating agents restore the PG level of aged bitumen. The rejuvenated bitumen samples meet the after RTFO criteria limit, although the after RTFO properties are not as better as the virgin neat bitumen properties.

When AFM images of rejuvenated and non-rejuvenated bitumen samples are investigated, bee-structures observable on the surface of neat virgin bitumen cannot be viewed on the micro surface of aged bitumen. After rejuvenation, tiny bee-structures appear on rejuvenated bitumen samples. It can be concluded that all rejuvenating agents significantly change the micro topography and phase dispersion of aged bitumen. Although AFM images of virgin neat bitumen are significantly different from rejuvenated bitumen samples, similarities in terms of viscosity, rigidity, and bee-structures can be observed compared to aged RAP bitumen.

The recommendations which can be considered for the further research are mostly connected to fatigue and low-temperature properties of rejuvenated bitumen samples and recycled mixtures. Fatigue and low-temperature properties of rejuvenated samples should be investigated carefully. The most concerned deterioration reference for recycled asphalt materials is fatigue and low-temperature cracking. It is highly recommended to evaluate these properties in industrial applications. The low-temperature characteristics of rejuvenated bitumen samples should be studied using related tests such as BBR and DTT.

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