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Development of an Alternative Bitumen Rejuvenator Consisting of Waste Oils and Reacted and Activated Rubber (RAR)

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Abstract

After a long-term in service on pavement, bituminous material exposure to environmental conditions and traffic loads leads to inevitable aging, causing an increase in binder viscosity and making it stiffer. On the other hand, the absence of proper elimination and illegal dumping of waste oil and tire rubber can lead to serious environmental risks. This research aims to investigate the possibility of using waste engine oil (WEO) and waste cooking oil (WCO) separately combined with reactive and activated rubber (RAR) as new alternative rejuvenating agents. In this study, 5% of waste oil (WCO, WEO) and 15% of RAR were added to the aged bitumen with a penetration grade of 20/30. The virgin, aged, and rejuvenated bitumen were investigated for their physical and chemical properties using penetration, softening point, and Fourier transform infrared spectroscopy (FTIR) tests, as well as the effect of thermal cycles (heating and cooling) on the cracking resistance of bituminous mixtures containing regeneration bitumen using the Fénix test. The study outcome indicated that these rejuvenators could effectively soften and restore the basic properties of the aged bitumen to a normal level of 40/50. Meanwhile, the bituminous mixture with the aged bitumen rejuvenated with both oils and tire rubber (AB-WRCO and AB-WREO) showed the best cracking behavior. Therefore, these alternative solutions involve not only reusing the aged bitumen but also reusing the waste oil and tire rubber in order to attain environmental protection and economic benefits.

Keywords

bitumen, rejuvenator, waste oil, waste tire rubber, Fénix test

1 Introduction

After a long period of service, the bituminous material is subjected to various factors, such as thermal cycles (heating and cooling) and traffic loads, which lead to inevitable aging [1, 2]. The oxidation of the bituminous binder and the loss of volatile compounds (some light oils) are the main causes of aging, resulting in an increase in viscosity and a stiffer bitumen [3]. Aging is considered one of the main costs of the degradation of pavement performance [4, 5]. Henceforth, this significantly reduces the service life of flexible pavements [6]. At the present time, the use of recycled asphalt pavement (RAP) materials worldwide is a preferable method due to environmental sustainability and economic benefits [7–10]. The key to reusing the RAP is to reuse the aged bitumen [9]. Regenerators can be used so as to recover the initial ratio (aromatics/satures) in aged bitumen and reconstituting the volatile substances [11]; they are rejuvenating and softening agents that are introduced as a technique for restoring the lost characteristics of aged bitumen [8, 12].

Owing to the high demand for the modifiers and regenerators used in asphalt pavement, there is a need for the use of alternative or waste material. Numerous studies have revealed that the use of waste engine oil (WEO) and waste cooking oil (WCO) has an effective rejuvenation impact [7–9, 13, 14]. Moreover, the rapid increase in the number of vehicles and living conditions worldwide generates significant amounts of waste oils such as WEO, which is produced every year in the processes of maintenance

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and repair of vehicles (routine oil change) [1, 15], in addition to WCO that can be collected correctly in restaurants and food industries with global production at a rate of 29 million tons annually [15].

A serious problem is that the majority of waste oil is dumped illegally into rivers and sewer systems, causing water and soil pollution with environmental problems [16]. It is commonly known that one ton of waste oils could contaminate one million tons of consuming water [14].

Thus, recycling waste oils in bitumen is one of the solutions to achieve environmental protection and economic benefits [15]. WCO and WEO are the most important types of rejuvenators which contain a similar molecular structure to bitumen [10, 13]. However, the rejuvenated bitumen revealed high sensitivity to humidity because of the poor adherence. For this reason, in order to improve it, other modifiers are need. On the other hand, bitumen modified by crumb rubber from tires (CR) has a good adhesion property [17]. Globally, 1.5 billion tires are produced every year. Approximately, two-thirds of them will become waste tires. This can lead to serious environmental risks in absence of proper elimination [18, 19]. Incorporating CR in asphalt pavement as a modifier of binders or an alternative for aggregate has increased dramatically in recent decades [20].

Several previous studies revealed that the combination of waste oil with crumb rubber leads to producing rejuvenation binder as they complement each other.

Yi et al. [17] studied the development of a new rejuvenator binder that consists of WCO and CR concluding that it has a similar rejuvenation effect as a commercial rejuvenator. Bilema et al. [21] stated that the combination of WCO and CR with aged bitumen revealed no chemical reaction and comparable rheological properties to the virgin binder. In the case of Yi et al. [15] research, the sulfoxide index indicated that three hours reaction time of aged bitumen with waste rubber oil (WRO) gave a better rejuvenation result. Bilema et al. [22] found that the incorporation of this waste in Reclaimed Asphalt Pavement (RAP) has improved rutting and moisture resistance, as well as the workability.

Based on the previous studies cited above, this research aims to investigate the possibility of using WEO or WCO combined with reactive and activated rubber (RAR) as a rejuvenating agent for the aged bitumen, which was aged innovatively. In addition, the effects of thermal cycles (heating and cooling) on the cracking resistance of bituminous mixtures using the Fénix test. Furthermore, no attempts have investigated the impact of rejuvenation agent composed by waste oil with RAR.

The findings of this inquiry might be used to guide future studies.

2 Materials and methods

2.1 Materials

The conventional Bitumen (40/50) was used for this study. As shown in Fig. 1. The virgin bitumen was aged naturally through an innovative and effective method. In the Saharans region, where it is located Bechar, in south-west Algeria, the climate is hot in summer (100 days) and the temperature reaches 50 °C, during this time. In order to obtain aged bitumen, the virgin bitumen was poured into plates and exposed to various natural climatic conditions (Temperature changes between day and night, wind, rain, sunlight, and moonlight…etc).

Fig. 2. shows the temperatures that were measured, so the maximum reach was 78.4 °C in the surface of the bitumen as well as the minimum was 12.6°C. With respect to WCO and WEO used in this study, they were collected from restaurants and auto repair shops located in Bechar, respectively. To assure regeneration efficacy, they were filtered to eliminate solid particles according to Li et al. [13]. As a rubber modifier, RAR was developed [23]. Particles generally contain 22% soft bitumen, 62% fine crumb rubber, and 16% fillers. The fine crumb rubber has a maximum size of 0.6mm [19]. In order to achieve an effective product, it is advisable to use at least 15% of RAR [23].

Fig. 1 Bitumen before and after aging

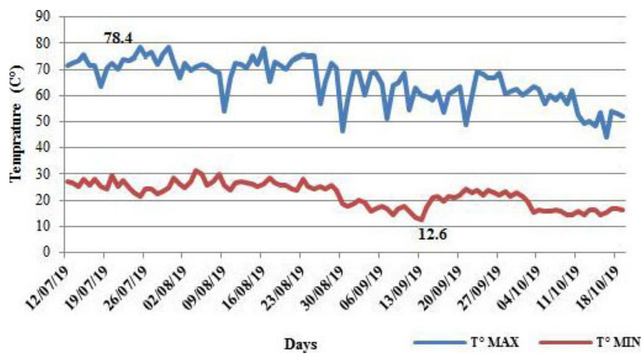


Fig. 2 The temperature of bitumen during the summer period

Regarding the aggregates, three fractions of limestone granular classes (0/3 mm, 3/8 mm, and 8/15 mm) as well as filler were used at 36%, 31%, 32%, and 1%, respectively. These aggregates were from the SARL BOUCHTA quarry in Bechar, Algeria. Table 1 shows the results of physical, chemical, and mechanical tests. These materials were acceptable and had good intrinsic characteristics for the composition of bituminous mixtures according to national specifications.

2.2 Experimental method

In this study, the experimental work was divided into three main phases:

- The initial phase of preparation and testing of the six types of bitumen as illustrated in Table 2, using penetration, softening point, and FTIR tests.
- The second phase of preparation and testing of the four different types of bituminous mixtures with four different types of bitumen (VB, AB, AB-WRCO, and AB-WREO).
- The final phase of preparation of all bituminous mixtures with regeneration bitumen, and testing before and after the simulation of the thermal cycles.

2.2.1 Preparation and testing bitumen

Preparation of rejuvenated bitumen

Based on previous studies shown in [16, 23, 24], 5% of waste oil (WEO and WCO), and 15% of RAR by the weight of aged bitumen were selected. The following steps were used to produce the regenerated bituminous: first, aged bitumen and waste oils were mixed for 10 min at a rotation speed of 2900 rpm at 180 ± 5 °C using local equipment.

Subsequently, the required amount of RAR was poured into the mixer for 5 min in order to achieve a homogenous mixture [23]. Finally, the six different samples were available to be tested later.

Basic properties tests

Fig. 3 illustrates the penetration and softening point tests that were conducted to evaluate the regeneration effect of WEO, WCO, WREO and WRCO as regenerates and their impact on aged bitumen [14]. The penetration test is used to determine the bitumen's consistency and the softening point test was carried out to determine the temperature sensitivity of bitumen according to EN 1426:2015 [25] and EN 1427:2015 [26] respectively.

FTIR test

In order to identify the functional groups in compounds, the Fourier transform infrared spectrometry test (FTIR) is widely used [20]. In this study, the Agilent Cary 630 FTIR shown in Fig. 4. Was used to characterize the chemical composition of virgin, aged, and rejuvenated bitumen [9]. For each scan, the wave numbers ranged between 600 and 4000 cm^{-1} [20].

2.2.2 Preparation and testing bituminous mixture

Marshall test

The Marshall test is the most often used in Algerian laboratories to characterize the bituminous mixture according

Table 1 Aggregate characteristics

Characteristics	0/3	3/8	8/15	Reference standard
Bulk density (g/cm^3)	1.61	1.44	1.44	–
Real density (g/cm^3)	2.7	2.69	2.69	–
Flatness (%)	/	13%	8%	NF P 18-561
Los Angeles (%)	/	22%	22%	NF P 18-573
Micro-Deval (%)	/	17%	17%	NF P 18-572
Sand equivalent (%)	67%	/	/	NF P 18-597
Methylene blue value (%)	1.25	/	/	NF EN 933-9

Table 2 Bitumen samples identification

Order	Sample ID	Bitumen composition
1	VB	Virgin bitumen
2	AB	Aged bitumen
3	AB-WEO	Aged bitumen +5% WEO
4	AB-WREO	Aged bitumen +5% WEO+15% RAR
5	AB-WCO	Aged bitumen +5% WCO
6	AB-WRCO	Aged bitumen +5% WCO+15% RAR

(a) (b)

Fig. 3 Basic properties tests (a) Softening point test, (b) Penetration test

Fig. 4 FTIR equipment

to EN 12697-34:2020 [27]. As shown in the Table 3 the best Marshall properties were obtained with a bitumen content of 6.15%. This content will be used as a reference in this investigation. Following that, different types of bituminous mixtures with different types of bitumen were prepared to be tested later using the Fénix test.

Simulation of thermal cycles on heating and cooling

In the hot regions, sudden changes in day and night temperatures lead to heating and cooling phenomena that cause the aging of bitumen and cracking of bituminous mixture, affecting the life of the asphalt pavement [2]. In this study, a simulation of the heating and cooling thermal cycles was carried out in the FIMAS laboratory, which is equipped with an air conditioner to maintain 25 °C, an oven to perform heating for 12 h and keep constant at 60 °C for 8 h as shown in Fig. 5 [28]. And an aerator to accelerate binder oxidation, and a timer socket to turn the devices on and off. Simulation of these temperatures represents the climate conditions of hot regions [29]. Fig. 6 shows devices for the simulation of thermal cycles for heating and cooling.

The purpose in this phase is to simulate the climatic conditions experienced by pavements in hot regions:

- Four different samples of bituminous mixture with four different types of bitumen (VB, AB, AB-WRCO, and AB-WREO) are intended as a reference (without thermal cycles).

Table 3 Marshall test results

Formulations	A	B	C	D
Richness Modulus	3.45	3.60	**3.75**	3.90
Bitumen Content (%)	5.66	5.90	**6.15**	6.39
Marshal Stability (KN)	12.62	12.04	**13.38**	10.05
Flow (mm)	3.67	3.58	**3.80**	3.65
Quotient (KN/mm)	3.44	3.36	**3.52**	2.75

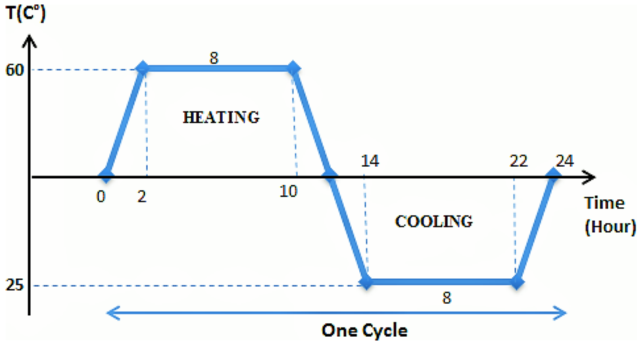


Fig. 5 One thermal cycle on heating and cooling of bituminous mixture [28]

Fig. 6 Devices for the simulation of thermal cycles for heating and cooling

- Fourteen samples of bituminous mixture with the regeneration bitumen by WRCO and WREO were exposed to different thermal cycles (15 days, 1 month, 2 months, 3 months, 4 months, 5 months, and 6 months), two samples exposed in the terrace for 6 months (AB-WRCO TRS, AB-WREO TRS), and two samples with anti-aging (anti-aging WREO and anti-aging WRCO, made from VB plus WRCO and VB plus WREO, respectively).

Fénix test

To evaluate the effect of waste oil plus RAR on the cracking resistance of the bituminous mixture, a new direct tensile test was used. The Fénix test was developed by the Road Research Laboratory at Universitat Politècnica de Catalunya in Barcelona, Spain [30]. The Fénix specimen is semi-cylindrical with a diameter of 101.6 mm, prepared by Marshall or gyratory compaction, and has a 6 mm-deep notch in the middle to induce cracking. The test is carried out at a constant displacement rate of 1 mm/min and at specific temperature (25 °C). The Fénix test steps are illustrated in Fig. 7.

Throughout the test, load and displacement data are recorded to determine a variety of parameters such as the maximum strength, the displacement at 50% of the post-peak load, and fracture energy [31]:

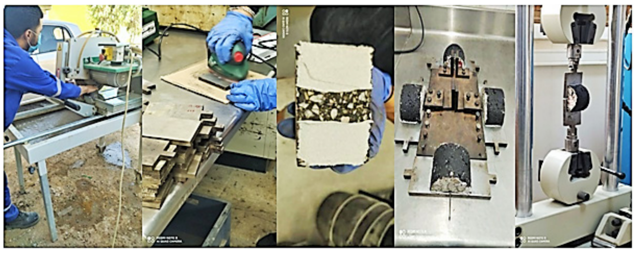


Fig. 7 The Fénix test steps

- The maximum strength, RT, is defined by Eq. (1):

$$\mathrm{RT} = F_{max} / S. \quad (1)$$

- Displacement at 50% of the post-peak load, (Dmdp, mm).
- Fracture energy GD is calculated by the Eq. (2):

$$\mathrm{GD} = \frac{\int_0^{df} F(x)\,dx}{S} \times 10^6, \quad (2)$$

where, RT is the maximum strength (MPa), GD is fracture energy during cracking (J/m^2), F is the tensile load (N), x is displacement (m), S is surface fracture (mm^2), and df is displacement at the end of the test (m).

3 Results and discussion

3.1 Bitumen test results

3.1.1 Penetration test

Fig. 8 displays the effect of adding different waste oils and RAR on the penetration grade of aged bitumen. The most observable result is that the aged bitumen, which has a penetration grade of approximately 20/30, has become stiffer because of the effect of aging. It is noticeable that the value of penetration grade has dramatically increased by adding both WEO and WCO to aged bitumen and has recovered by more than 115% and 136%, respectively, compared to the aged bitumen. However, it is worth noting that the addition of these two substances has an unequal effect on penetration. The effect of adding WCO was greater compared to that of adding WEO on penetration. The components of WCO have been examined, and the outcome of this scrutiny indicates that WCO includes numerous light components, and therefore it increases the fluidity of the aged bitumen [13]. Nevertheless, adding RAR to both WEO and WCO decreased the penetration grade value. As a result, the aged bitumen returns to the base bitumen level (virgin bitumen). This aspect indicates that the performance of the bitumen has improved.

3.1.2 Softening point test

Fig. 9 illustrates the impact of adding both WEO and WCO on the values of softening point of aged bitumen. It also demonstrates the decrease of the softening point of aged bitumen while adding both of WEO and WCO and its recovery by more than 104% and 96%, respectively. Consequently, bitumen becomes subject to obtaining higher temperature sensibility in comparison with the aged bitumen. However, the addition of RAR to both WEO and WCO led to increasing the softening point and hence returning the aged bitumen to 40/50 penetration grade with softening points of 55 °C and 52 °C, respectively. It is notable that these new softening points are higher than that of the original 40/50 bitumen (50 °C), which resulted in rejuvenated bitumen with lower temperature sensibility.

It is, therefore, relevant to conclude that the regeneration of aged bitumen by waste oil and RAR exhibits higher temperature resistance and could be beneficial in hot climate zones.

3.1.3 Fourier transforms infrared spectrometry (FTIR) analyses

Infrared spectrograms at wavenumbers ranging from 600 to 4000 cm^{-1} are utilized to evaluate the control bitumen as well as the change in each functional group of aged and rejuvenated bitumen [14]. Fig. 10 illustrates the FTIR analysis of control and aged bitumen, as well as the

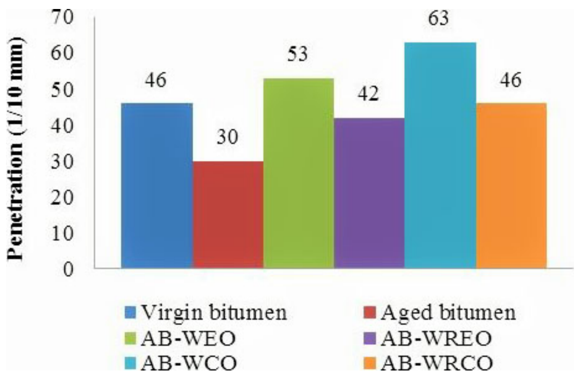


Fig. 8 Penetration results

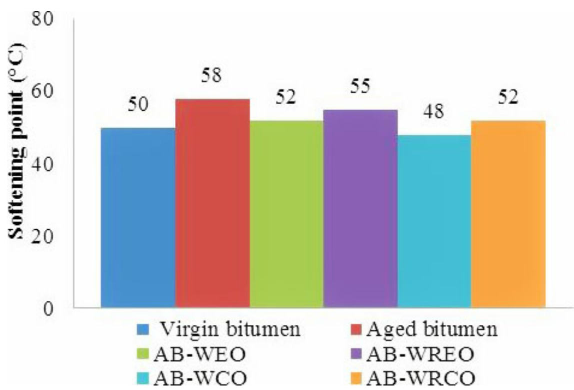
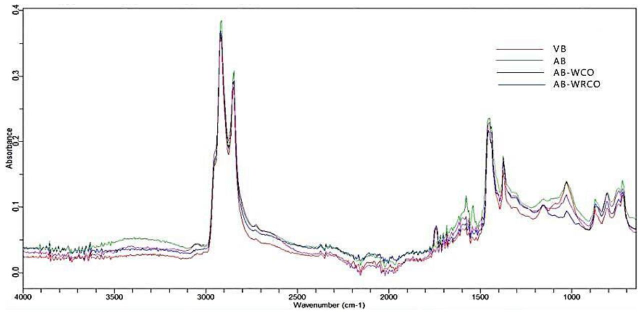
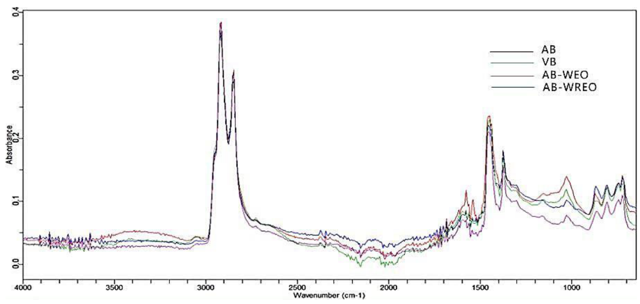


Fig. 9 Softening point result



(a)



(b)

Fig. 10 (a) FTIR results of aged bitumen with WRCO and (b) FTIR results of aged bitumen with WREO

rejuvenated aged bitumen by WCO, WEO, WRCO, and WREO. The FTIR spectrum of aged bitumen (AB) reveals the existence of an OH broad stretching absorption band at a wavenumber of 3442.2 cm^{-1}, as well as two CH alkane absorption bands at 2916.6 and 2849.5 cm^{-1}. It, additionally, exhibits a strong absorption band at 1030.6 cm^{-1}, that is attributed to the presence of a sulfoxide group (S=O), and two absorption peaks: one at stretching frequency at 1578 cm^{-1} and another one at bending frequency at 743.6 cm^{-1}, which indicate the presence of the C = C function group in AB, which agrees with the previous studies by [8, 13]. The peak areas that reflect the aging and rejuvenating degree of bitumen are sulfoxide (S=O) and carbonyl (C=O), which correspond to wavenumbers of 1030 and 1700 cm^{-1}, respectively [7, 9, 13]. The higher intensities of these peaks are related to the higher content of asphaltene in the bitumen [9]. As it is demonstrated in the Fig. 10 (a), the new absorption peaks at 1742.5 cm^{-1}, 1159.2 cm^{-1}, and 1157.3 cm^{-1} in the AB-WCO and AB-WRCO samples correspond to the ester carbonyl functional group and C–O stretching, respectively. These are the WCO's characteristic peaks and the ones responsible for softening the bitumen [32]. These results are, indeed, similar to those found in Yi et al. [17] that examined the development of a rejuvenator that combined WCO and crumb tire rubber.

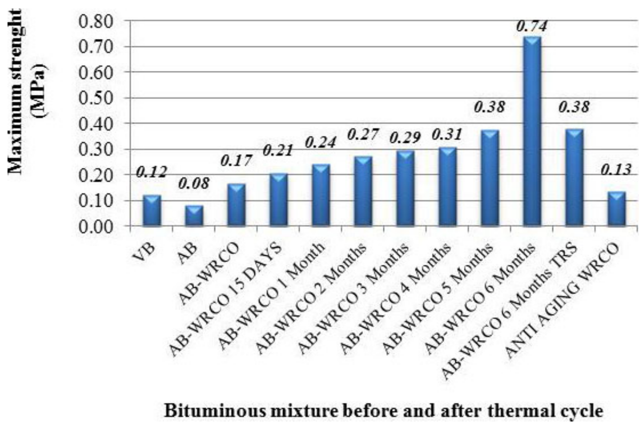
According to a previous study conducted by Chen et al. [20] to examine RAR modified bitumen, there were no new peaks noticed among the existing peaks of bitumen because the RAR particles contained bitumen components. This result reveals that there could be no possibility for these components to react chemically. Furthermore, it is clear from Figs. 10 (a) and (b) that after adding WRCO and WREO to the aged bitumen, the intensity of the C = C and S = O bands decreases due to a reduction in the asphaltene content. As a result, the ratio of asphaltene to maltene decreases, which had an effective impact on the performance of rejuvenated bitumen [8].

3.2 Bituminous mixtures test results

3.2.1 Fénix results

Maximum strength (RT)

Fig. 11 illustrates the changes in maximum strength for different bituminous mixtures with different types of bitumen, and the effect of thermal cycles in heating-cooling on AB-WRCO and AB-WREO mixtures. It can be deduced from Fig. 11 that the bituminous mixture with aged bitumen showed the lowest maximum strength, while the bituminous mixture with AB-WRCO and AB-WREO clearly increased as the thermal cycle in heating-cooling



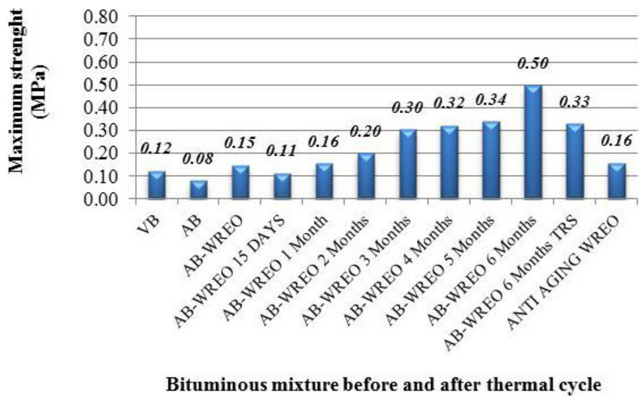


Fig. 11 Maximum strength results

increased. Comparing the maximum strength between mixtures with WRCO and WREO, mixtures with WRCO have greater resistance, although this difference is not constant; when the mixtures are tested after applying cycles during 3 and 4 months, maximum strength is even slightly greater for mixtures with WREO.

In particular, the bituminous mixture with AB-WRCO and AB-WREO after six months of thermal cycles in heating-cooling showed a higher maximum strength due to the combination of waste oil and RAR, which means that the bituminous mixture needs great strength and energy to crack. Moreover, It is interesting to notice that the anti-aging WRCO and WREO they produced the same value as AB-WRCO and AB-WREO mixtures, respectively, demonstrating that the new rejuvenators also had anti-aging properties. Furthermore, it can be concluded from the results that the mixture undergoes a stiffening phenomenon when subjected to thermal cycles in heating and cooling, causing the aging of bitumen [2].

Displacement at 50% of post-peak load

Fig. 12 shows the displacement at 50% of the post-peak load (Dmdp) of different types of bituminous mixtures.

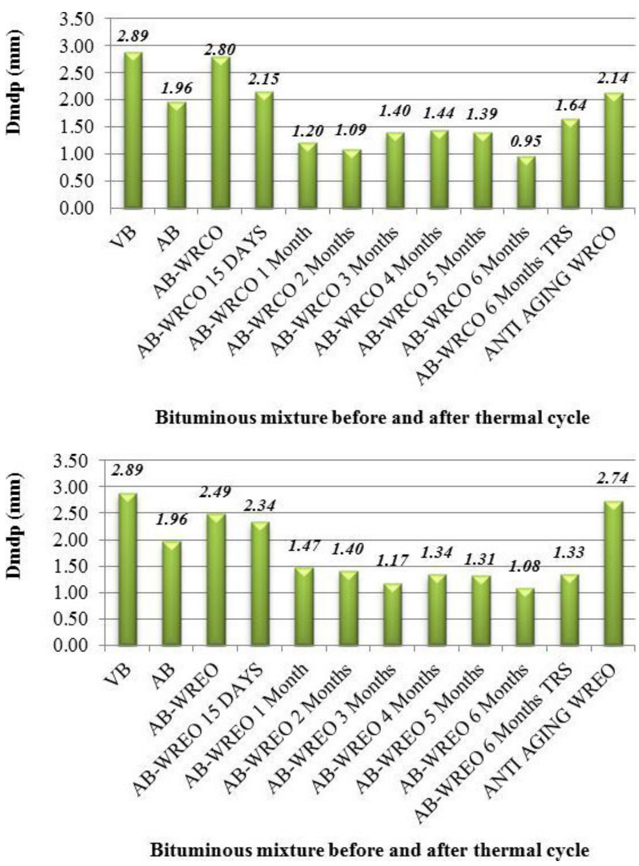


Fig. 12 Displacement at 50 % of post-peak load

The increase in Dmdp is proportional to the ductility value, which leads to an improvement in the crack resistance of the bituminous mixture. The bituminous mixture with AB presents the lowest displacement at 50% post-peak load due to the higher stiffness and fragility of the aged bitumen, whereas the Dmdp increases with the addition of the rejuvenators WRCO and WREO. It also becomes clear that the trend was unstable and the decline of Dmdp is not significant enough to have a negative impact.

Fracture energy (GD)

The variation of the fracture energy illustrated in Fig. 13, the bituminous mixtures with AB-WRCO and AB-WREO after one month of thermal cycles in heating-cooling showed a continuous increase in the response to cracking. While the bituminous mixture with AB showed the lowest fracture energy.

All the parameters obtained from the Fénix test, such as RT, GD, and Dmdp, provide an indication of the cracking resistance of the bituminous mixtures [30]. In service, aging is considered one of the main causes of deterioration in the performance of pavements [5]. The WRCO and WREO rejuvenators not only regenerate the aged bitumen but also ameliorate the performance of the bituminous mixture under the stress of thermal cycles in heating-cooling. In general, the bituminous mixture will benefit from

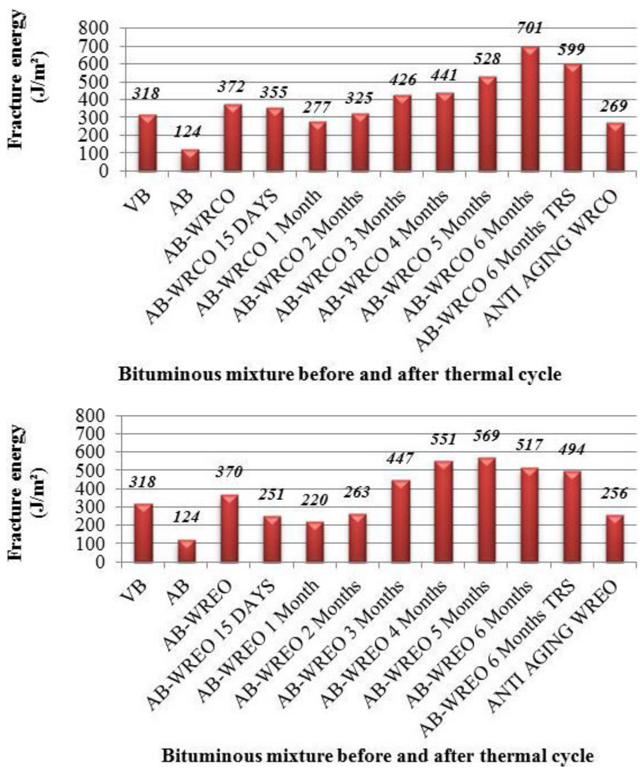


Fig. 13 Fracture energy results

having high maximum strength, high fracture energy, and a high displacement value at 50% post-peak load.

The comparison between bituminous mixtures with VB and AB shows that the mixture becomes stiffer due to aging conditioning, and thus the fracture energy and displacement decrease. Concerning the heating and cooling cycles, there is a clear trend toward increased maximum strength and fracture energy, as well as decreased displacement. Regarding TRS conditioning, it is observed that the effect is similar to heating and cooling cycles of 4 months in terms of strength and displacement. In terms of fracture energy, TRS conditioning shows greater dispersion between 3 and 6 months of heating and cooling cycles.

Comparing the bituminous mixtures with aged bitumen and anti-aging treatments bitumen (ANTI-AGING WRCO and WREO), the protective effect is shown through an increase in strength, displacement, and fracture energy. Comparing the bituminous mixtures with AB and AB treated with a rejuvenator consisting of waste oil and RAR (AB-WRCO and AB-WREO), the improvements are clear and even greater than the anti-aging treatment bitumen (higher strength, displacement, and fracture energy). Another interesting finding is the clear trend between maximum strength and fracture energy, as can be seen in Fig. 14.

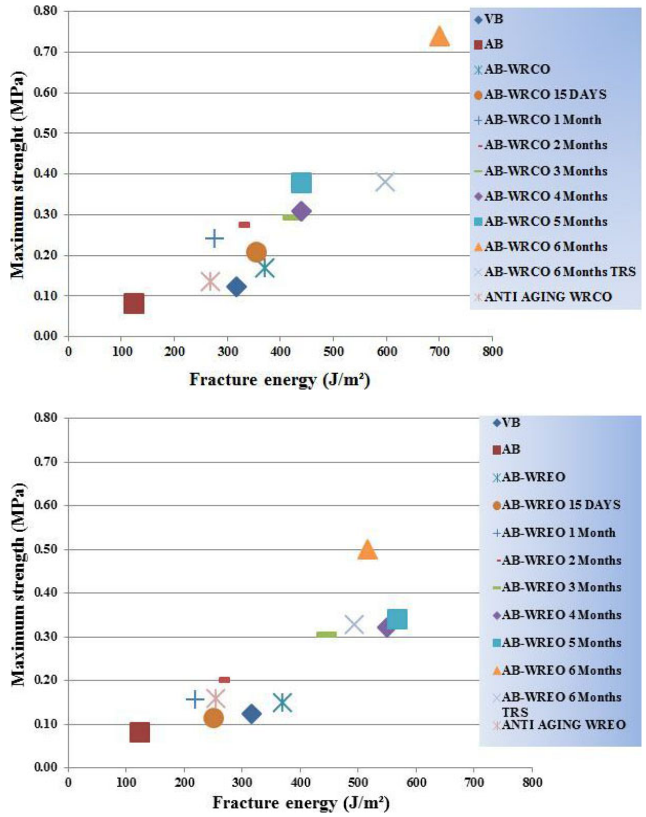


Fig. 14 Maximum strength versus fracture energy

4 Conclusions

The goal of this study was to look into the possibility of using waste oil plus RAR as a new alternative rejuvenator to restore and improve the properties of aged bitumen by evaluating its physical and chemical properties, as well as the effect of thermal cycles on heating and cooling in the cracking resistance of bituminous mixtures containing regeneration bitumen. According to the findings of this study, the following conclusions can be drawn:

1. The addition of 5% waste oil (WEO, WCO) and 15% of RAR could effectively improve the basic physical properties of aged bitumen to a normal level, before aging.
2. The regeneration of aged bitumen by WREO and WRCO exhibits higher temperature resistance than the original bitumen and could be beneficial in hot climate zones.
3. From the FTIR analysis, there were no new peaks or significant changes noticed among the existing peaks of the bitumen due to the non-chemical reaction of the additives.
4. The bituminous mixture with AB-WREO and AB-WRCO showed the best response to cracking.
5. The WREO and WRCO rejuvenator not only regenerates the aged asphalt but also ameliorates the performance of the bituminous mixture under the stress of thermal cycles in heating-cooling. Meanwhile, it could not only reuse the waste bituminous mixture but also reuse the waste oil and rubber, which is beneficial to environmental protection and economical alternative.
6. According to this experimental study we can recommend:
   - Conducting mechanical tests on bituminous mixtures modified by waste oils and Reacted and Activated Rubber by the dry method.
   - Make an in-depth study on the new anti-aging agent of bitumen composed of waste oils and Reacted and Activated Rubber.

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