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Evaluation of the rejuvenation of asphalt by means of oil-saturated porous aggregates

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ABSTRACT

Road degradation is an increasing problem for assets managers. Roads deteriorate mainly due to the combination of different factors, such as temperature, climate and traffic loads. This causes the asphalt to age and, consequently, the bituminous mixtures become more fragile and microcracks begin to appear. To eliminate these degradation effects, the most widely used performed actions are based on the renovation of the pavement to achieve an asphalt surface under acceptable conditions of use. In order to reduce the application of corrective measures when the asphalt pavement is already facing significant defects, it is important to perform regular preventive maintenance that minimise the renovation works of the pavement and improves the asphalt durability. This paper shows a method for the continuous rejuvenation of asphalt, by analysing the evolution of asphalt mixture stiffness and the resistance to cracking on asphalt mixtures with rejuvenator added after having been submitted to a laboratory ageing protocol (short and long-term ageing). The rejuvenator was added to the mixtures following either of these two procedures: first, directly added to the mixture and the second, saturated in porous aggregates, what the authors call encapsulated. Results from this study demonstrate the effectiveness of the second method (encapsulated rejuvenator) as an original solution to achieve long-term performance mixtures with reduced cracking.

1. Introduction

Generally, asphalt roads last up to 20 years [1], provided they are constructed to specification and laid by experienced site operators. Despite their great longevity, the performance of in-service pavements is complex, involving many different factors such as temperature, climate, loading, construction quality, etc [2]. The combination of these factors makes asphalt susceptible to deterioration, being the two principal mechanisms by which road pavements fail structurally rutting and cracking [3]. This cracking in the bituminous materials eventually causes irreversible damage in the asphalt pavement that results in the need for larger maintenance and repair costs [4].

Improving asphalt pavement durability is one of the challenges the asphalt industry is facing today. Preventive maintenance is one of the most effective ways to extend pavement life in a cost effective manner. It can avoid many long-term problems that cause permanent damage to the asphalt pavement, and is far less expensive than a complete replacement of the asphalt prematurely.

A number of preventive maintenance types exist in literature, such as rejuvenators, slurry seals, surface treatments, and crack sealing [5]. However, to be effective as a preventive maintenance practice, these treatments must be placed much earlier in the pavement service life than is currently the practice [6]. Additionally, they should be repeatedly applied on sound pavements before excessive deterioration occurs.

Embedded encapsulated rejuvenating agents is a promising approach to increase the pavement service life that has received extensive attention in recent years, especially for healing of bituminous materials [7–10]. Although different rejuvenator categories exist [11], rejuvenating agents generally consist of lubricating and extender oils with a high proportion of maltenes constituents, which can restore the asphaltenes/maltenes ratio in the aged bitumen, turning back the loss of certain properties in the asphalt binders [12]. These characteristics of rejuvenators make possible their use in combination with reclaimed asphalt, to facilitate the incorporation of high proportion of reclaimed

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asphalt in the design of new hot mix asphalt [13–15]. In the study carried out by Passseto et al. [16] the effect of ageing was studied analyzing binders' responses in unaged, short-term and long-term aged conditions. Overall, experimental findings demonstrated the efficacy of the rejuvenation of recycled binders containing very high amount of aged bitumen.

However, the application of rejuvenator over the road's surface has shown some limitations. The problem is that for a rejuvenator to be successful it must penetrate the asphalt pavement surface [17]. In addition, if rejuvenator is applied and the road opened for traffic before allowing sufficient time for the diffusion of the rejuvenator, the pavement stability may be compromised because the layers are still too soft becoming more susceptible to rutting [18]. Encapsulated rejuvenators is an alternative that can solve these drawbacks.

The design of the capsule can vary significantly depending on the encapsulation method and the materials used. The most investigated one is the encapsulation of rejuvenator into a polymeric shell, by using methods such as in-situ polymerization and ionic gelation. The produced capsules have size ranges of 153 μm to 7 mm approximately [19]. A different approach for capsule preparation is the use of porous aggregate, in which the rejuvenator penetrates into the pores of the aggregate. The rejuvenator should be released over time from the pores of the aggregates by diffusion, reducing ageing and, therefore, preventing minor defects from becoming major ones. With this method, the produced capsules have a larger particle size (up to 6 mm) [20].

This technology offers a clear benefit in comparison with standard preventive maintenance. Preventive maintenance should be planned and cyclical in nature; while using encapsulated rejuvenators is a natural process that occurs autonomously.

Some promising methods for assessing asphalt concrete durability are cracking resistance as measured by semi-circular bend (SCB) testing at intermediate temperatures [21], as well as age hardening as measured by laboratory oven ageing tests [22].

The evaluation of the ageing recovery potential in asphalt mixtures is a quite complex process. In a recent study, a first attempt to validate the technical feasibility of these capsules using ageing tests and a further assessment on the properties of the recovered bitumen/rejuvenator blends was made [23]. However, during the recovery process, the binder/rejuvenator in the bituminous matrix can be forced out and blended, resulting in a homogenous blending. Other authors investigated the healing potential of asphalt binders due to ageing, rejuvenation and polymer modification by describing a healing model in which the positive effect of the use of rejuvenating agents and the healing ability of SBS binders at high damage levels was demonstrated [24]. There is a need, therefore, to validate whether the conclusions obtained on binder tests can be extrapolated to the mix.

In addition, although the ageing of bituminous materials is mainly linked to the ageing of the binder [25], some studies concluded that ageing of asphalt mixtures cannot be predicted by tests on asphalt binder alone since results show that aggregates have considerable influence on ageing [26].

Ageing causes the binder to harden, softening point and viscosity increase while ductility and penetration are reduced. Regarding mechanical properties, the stiffness modulus increases due to ageing and this increase can be up to four times depending on the type of asphalt [27]. This affects the asphalt pavement performance reducing its service life.

Asphalt ageing occurs in two stages. The first stage of ageing, referred to as short-term ageing, occurs during the production at high temperatures. The second stage of ageing, referred to as long-term ageing, occurs after the construction process and over the life of the pavement.

Ageing can be simulated in the laboratory by conditioning the bituminous mixture (loose or compacted) in an oven for different periods of time and at different temperatures [28]. The basic procedure is to artificially age the mixture and then assess the effect of ageing on key material parameters (e.g. stiffness, viscosity, strength, etc).

The evaluation of the cracking resistance of the asphalt mixtures is also a parameter that affects pavement durability. Typically, cracking appears after a number of years of damage accumulation on materials that have become embrittled. The semi-circular bend test is one of the most used methods to evaluate low temperature cracking. The cracking parameter and outcome of the SCB test for low temperature cracking is fracture energy [29]. Some recent studies using SCB to evaluate the fracture properties of asphalt mixtures with rejuvenator can be found in literature, most of them related to reclaimed asphalt mixtures. In these studies, rejuvenator was proved effective in enhancing the fracture resistance at both low and intermediate temperatures [30–34]. Also, others authors investigated the cracking resistance of self-healing asphalt mixtures using sodium alginate fibers [32–35] and even the SCB test was adopted to study crack propagation and its closure (healing) in an asphalt mix by others [10].

In this paper, a well-established test method alongside a more novel one, were selected to evaluate the effect of rejuvenator on the mixtures' short and long-term performance. These methods focus on the asphalt mixture, to simulate in a more realistic way what occurs in the field. First method evaluates the effect of rejuvenator on the indirect tensile stiffness modulus (EN 12697-26) and the second, on the cracking resistance of the asphalt mixtures through the Fénix test (NLT-383/20) [36,37,38].

The rejuvenator was added to the mixture in two different ways; first, directly added to the bitumen (prior to the mixing) and second, encapsulated. The capsules investigated in this study were those made of porous aggregate with rejuvenator, using sepiolite and vermiculite as a substrate.

The proposed test methods are an interesting approach to understand the rejuvenation of the asphalt mixtures and, therefore, to predict the lifetime extension of the asphalt pavements.

2. Materials and testing methods

2.1. Materials and methodology

An asphalt concrete for very thin layers (EN 13108-2), BBTM 11B type, was the asphalt mixture selected for the study. This mixture is characterised by a discontinuous grading and a high void content, between 12 and 18%. It was considered to be appropriate for the study since asphalt surfaces with high voids content are more susceptible to oxidative ageing due to greater exposure of the binder to air and higher temperatures. Literature reports that at voids content lower than 5%, very little ageing occurred in service; however, at void content higher than 9% the ageing is more noticeable [39].

Porphyry aggregate, limestone filler and PMB 45/80–65 (polymer modified bitumen), with a softening point of 67 °C, penetration of 57 dm at 25 °C and elastic recovery of 73%, were the materials chosen for the production of the conventional mixtures. This type of bitumen is often used in the manufacture of these mixtures. The asphalt mixtures composition and their particle size distribution are presented in Table 1 and Fig. 1, respectively.

The experimental asphalt mixtures included a known amount of asphalt rejuvenator (commercial petroleum-based product), either directly added to the mixture prior to mixing or encapsulated in porous aggregates, providing an approximated rejuvenator content of 0.70% by the total asphalt mixture. Therefore, different asphalt mixtures were prepared: some with 1.5% of sepiolite capsule, others with 1%

Table 1
Mixture composition (by aggregate mass).

Porphyry 6/12	71%
Porphyry 0/6	23.5%
Limestone	5.5%
Polymer Modified Bitumen 45/80–65	5.25%

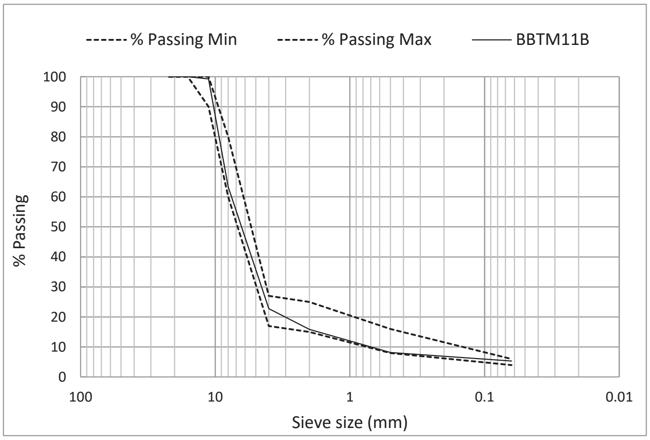

Fig. 1. Particle size distribution.

vermiculite capsule (Fig. 2) and finally without capsule, but containing an equivalent amount of rejuvenator. Higher percentage of sepiolite capsule was used to compensate the low proportion of rejuvenator in the capsule (40% in sepiolites versus 78% in vermiculite [23]). The rejuvenator selected for the study was a refined crude oil product with a high proportion of maltenes.

Finally, cylindrical asphalt specimens with diameter 100 mm and height of approximately 65 mm were compacted by gyratory compactor with the number of gyrations required to meet the target height. Four and six replicates of each asphalt mixture, depending on the test, were included in the study. According to the bitumen provider recommendations, the temperatures of 165 °C and 150 °C were adopted for the asphalt mixture production and compaction, respectively.

An overview of the research approach followed in this study is shown in Fig. 3, which is divided in two main parts: A) rejuvenator directly added to the mixture and B) rejuvenator encapsulated in porous aggregates. All the mixtures were subjected to an ageing protocol. Part of the specimens were then tested at the Technical University of Catalonia by the Fénix test and the remaining samples at ACCIONA by the stiffness test. To compare the results, all asphalt mixtures samples were prepared and aged in the same laboratory.

2.2. *Ageing protocol*

Ageing of asphalt mixtures can be simulated in the laboratory maintaining the mixture (compacted or loose) in an oven at a defined temperature and time. The ageing process can be separated into two stages as occurs in the field, one during the manufacture of the hot mix asphalt, known as short-term oven ageing (STOA), and the second during the service life of the pavement, referred to as long-term oven ageing (LTOA).

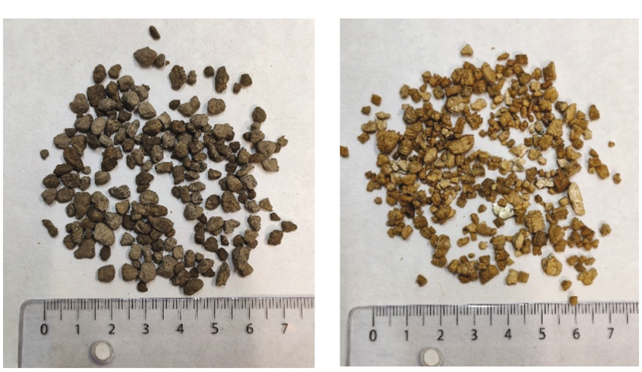

Fig. 2. Sepiolite (left) and vermiculite (right) capsule.

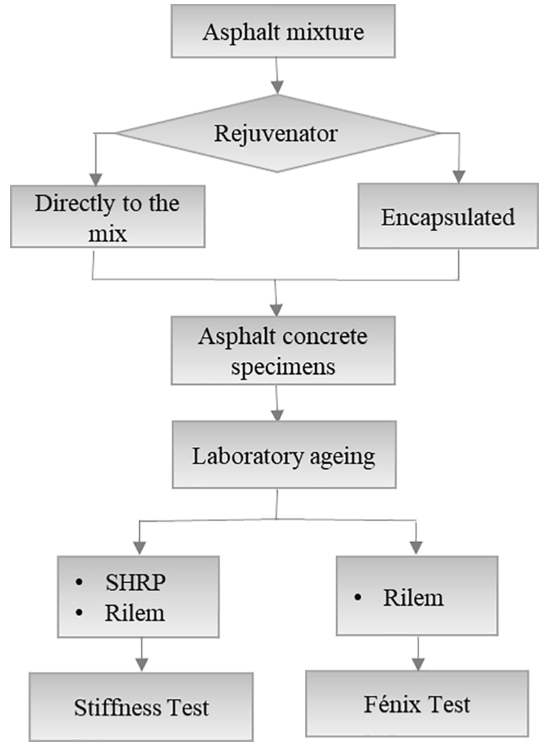

Fig. 3. Experimental flow chart.

In this study, two ageing methods were studied as shown in Table 2. The first one is the ageing procedure AASHTO R-30 developed within the Strategic Highways Research Programme (SHRP), which is one of the most commonly used methods. This standard protocol includes a short-term ageing, in which the loose mixture is aged in an oven for 4 h at 135 °C, and a long-term ageing, in which the mix, previously aged by STOA, is compacted and kept in the oven for five days at 85 °C [40]. Second, a new ageing procedure developed by the RILEM Technical Committee ATB-TG5 in 2009. In this method, the loose mixture is aged for 4 h at 135 °C for short-term ageing and for 9 days at 85 °C for long-term ageing [41]. However, in this study, the long-term ageing included also a conditioning of the loose mixture for shorter periods of 2, 5 and 7 days in order to have a deeper insight of the influence of the ageing with time. Additionally, 19 days conditioning of the loose mixture at 85 °C was included to evaluate the rejuvenator effect for longer term, especially when it is added in the form of capsule. Considering that the long-term ageing is reported to be representative for field ageing of about 15 years, depending on the weather conditions [42], 19 day ageing appears to be sufficient for field ageing over the lifetime of the road.

The effect of ageing on the bituminous mixtures was evaluated through the indirect tensile stiffness modulus and Fénix test at different steps of the protocol.

Table 2
Ageing programme.

Ageing Protocol	Ageing stage	Mixture condition	Conditioning time and temperature
SHRP	Short-term ageing	Loose mixture	4 h, 135 °C
	Long-term ageing	Compacted mixture	5 days, 85 °C
RILEM	Short-term ageing	Loose mixture	4 h, 135 °C
	Long-term ageing	Loose mixture	2,5,7,9,19 days, 85 °C

2.3. Indirect tensile stiffness modulus

The dynamic stiffness was determined in the laboratory using the indirect tensile test, according to the EN 12697–26 (Annex C) standard (Fig. 4). In this test, five pulse loads with a total duration of 3 s were applied, recording the variation of the vertical load and the horizontal displacement. The modulus was calculated by the following equation:

$$E = \frac{F(\nu + 0.27)}{z \bullet h} \tag{1}$$

where E is the stiffness modulus (MPa); F is the loading force (N); ν is the Poisson's ratio; z is the amplitude of the horizontal deformation obtained during the load cycle (mm) and h is the mean specimen thickness (mm).

The test was performed at 20 °C and a minimum of four cylindrical specimens were tested.

2.4. Fénix test

A new direct tensile test, the Fénix test, was developed by the Road Laboratory of the Technical University of Catalonia to determine the cracking resistance of bituminous mixtures. This test consists on applying a tensile stress on a half cylindrical specimen at a constant rate of 1 mm/min. The specimen has a small notch at the centre of the specimen, which induces the cracking of the sample (Fig. 5).

Stress and displacement data are recorded throughout the test to calculate the parameters involved in the cracking process: maximum strength, tensile stiffness index, fracture energy during cracking, toughness index and toughness displacement (Fig. 6).

The maximum strength (R_T) is related to the cohesion given by the asphalt mastic to the mixture. This parameter is calculated from Eq. (2):

$$R_T = \frac{F_{max}}{S} \tag{2}$$

where R_T is the maximum strength (MPa); F_{max} is the peak load (kN) and S is the cross-sectional area (mm^2). The cross-sectional area is calculated as the height of the specimen, h, multiplied by the reduced radius, r_r. The reduced radius is defined as the average specimen radius measured in the area where the crack was propagated subtracting its depth.

The tensile stiffness index (IRT) is defined as the slope of the stress-displacement curve between 25% and 50% of the peak load (upward curve part), divided by the cross-sectional area. It is an indicator of the asphalt mixture stiffness, being related to the mix modulus. It is calculated using Eq. (3):

$$IRT = 1000 \times \frac{F_{50} - F_{25}}{S \times (d_{50} - d_{25})} \tag{3}$$

where IRT is the tensile stiffness index (MPa/mm), F_{50} and F_{25} are the values corresponding to the 50% and 25% of the peak load (kN), S is the cross sectional area (mm^2) and d_{50} and d_{25} are the displacement values at 50% and 25% of the peak load (mm).

Fig. 4. Stiffness test equipment.

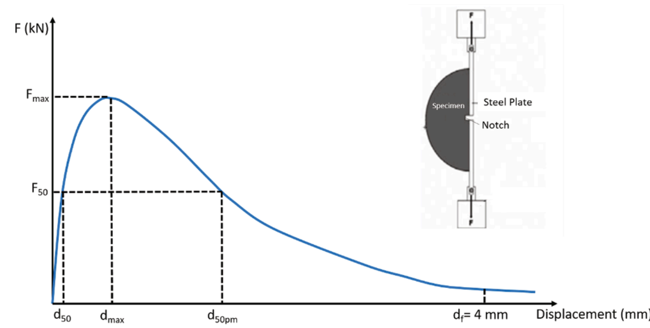

Fig. 5. Stress-displacement curve and Fénix test set-up.

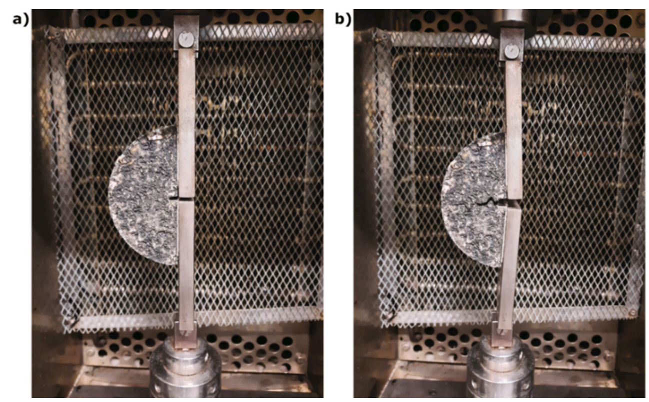

Fig. 6. Fénix test configuration and specimen a) before and b) after the test.

The fracture energy during cracking (G_D) uses the integral function to calculate the area below the load–displacement curve divided by the cross-sectional area. It means the effort required for the mixture cracking and it is calculated from Eq. (4):

$$G_D = 10^6 \times \frac{\int_0^{d_f} F(x)dx}{S} \tag{4}$$

where G_D is the fracture energy during cracking (J/m^2), $F(x)$ is the load (kN), x is the displacement (mm), S is the cross sectional area (mm^2) and d_f is the displacement at the end of the test (m). The test is considered to be completed when it reaches a displacement of 4 mm.

The toughness index (IT) quantifies the ductility of the material by multiplying the dissipated energy in the cracking process, after the maximum load is reached, by the displacement needed to reduce the maximum load to 50%, as shown in Eq. (5):

$$IT = 10^6 x \frac{\int_{d_{max}}^{d_f} F(x)dx}{S} \times DT \tag{5}$$

where IT is the toughness index (J/m^2)•mm, d_{max} is the displacement at the peak load (mm), d_f is the displacement at the end of the test, S is the cross sectional area (mm^2) and DT is the toughness displacement (mm).

The toughness displacement (DT) is calculated as the difference between the displacement at 50% of the peak load (mm), d_{50pm}, and the displacement at the maximum peak load (mm), d_{max}, Eq. (6). It is an indicator of the asphalt mixture ductility.

$$DT = d_{50pm} - d_{max} \tag{6}$$

3. Results and discussion

This section summarises the test results of the samples with rejuvenator directly added to the bituminous mixture, the ones with encapsulated rejuvenator in porous aggregates and the reference mix (without rejuvenator), all combined in subsections 3.1 and 3.2, to facilitate their analysis.

3.1. Recovery potential of bituminous mixtures stiffness under different ageing levels

Dynamic modulus tests were conducted at 20 °C using unaged, short-term and long-term aged specimens and using SHRP and RILEM ageing procedures, respectively. Table 3 summarises the stiffness results expressed as mean values from six individual specimens, together with the density and air voids content.

Table 3
Densities, air void content and stiffness modulus.

Sample	Ageing level	Density(g/cm^3)	Air voids (%)	Stiffness Modulus (MPa)
Reference	Unaged	2.092	17.31	3107
	SHRP short-term	2.094	17.25	3961
	SHRP long-term	2.092	17.33	5921
Rejuvenator	Unaged	2.090	17.48	1924
	SHRP short-term	2.094	17.31	3010
	SHRP long-term	2.091	17.40	3857
Sepiolite capsule	Unaged	2.089	17.77	2740
	SHRP short-term	2.088	17.84	3531
	SHRP long-term	2.089	17.79	4791
Vermiculite capsule	Unaged	2.093	17.89	2650
	SHRP short-term	2.095	17.77	3375
	SHRP long-term	2.093	17.85	4389
Reference	Unaged	2.094	17.25	3855
	Rilem short-term	2.090	17.40	4485
	Rilem 2 days	2.090	17.40	4887
	Rilem 5 days	2.090	17.40	5450
	Rilem 7 days	2.095	17.19	6068
	Rilem 9 days	2.091	17.35	6784
	Rilem 19 days	2.092	17.32	7792
Rejuvenator	Unaged	2.086	17.61	2070
	Rilem short-term	2.085	17.65	2443
	Rilem 2 days	2.085	17.66	2752
	Rilem 5 days	2.084	17.71	3056
	Rilem 7 days	2.084	17.71	3439
	Rilem 9 days	2.089	17.71	3801
	Rilem 19 days	2.085	17.50	4520
Sepiolite capsule	Unaged	2.089	17.87	2930
	Rilem short-term	2.092	17.66	3406
	Rilem 2 days	2.090	17.75	3778
	Rilem 5 days	2.092	17.60	4287
	Rilem 7 days	2.087	17.86	4733
	Rilem 9 days	2.095	17.57	5084
	Rilem 19 days	2.090	17.50	5731
Vermiculite capsule	Unaged	2.094	17.82	2717
	Rilem short-term	2.094	17.81	3132
	Rilem 2 days	2.093	17.85	3332
	Rilem 5 days	2.092	17.91	3641
	Rilem 7 days	2.090	17.96	4075
	Rilem 9 days	2.092	17.90	4497
	Rilem 19 days	2.095	17.79	5273

Stiffness results are also represented in graphic bars in Fig. 7 and Fig. 8, in which the error bars represent the standard deviation.

Ageing causes the stiffness modulus to increase, as shown in Fig. 7 & Fig. 8. From these figures, it can be seen that the sample containing rejuvenator shows the lowest modulus in all the scenarios analysed (unaged, short-term ageing and long-term ageing). This confirms the role of the rejuvenator in the asphalt mixtures. In particular, the stiffness modulus for the sample with rejuvenator after SHRP long-term ageing is almost on the same level than that of the short-term aged reference mix. For the RILEM procedure, the effect of the rejuvenator is even more pronounced, showing a comparable stiffness modulus after long-term ageing (9 days) to that of the unaged reference mix. These results indicate that the addition of rejuvenator to an asphalt mixture should be controlled to avoid deformable mixtures, which can cause premature rutting on the pavement.

It can be also observed that an ageing from 9 to 19 days conditioning is not negligible, although is less significant than from 0 to 9 days, providing an increase in the stiffness of 14% for the reference mix and 29% for the samples with rejuvenator compared to the aged samples at 9 days.

It can also be noted that the stiffness modulus values after long-term ageing for all the samples investigated are quite similar, regardless the ageing method used. This suggests that the effect of these two methods, SHRP (5 days conditioning at 85 °C on compacted specimens) and RILEM (9 days conditioning at 85 °C on loose mixture), on the bituminous mixtures stiffness is comparable.

These data was compared to the mean values of the samples with encapsulated rejuvenator. These samples show lower stiffness modulus than the ones without rejuvenator (reference mix), but higher than the ones with rejuvenator directly added to the mix, after the ageing protocol (long-term ageing). It indicates that rejuvenator was released from the capsules at some extent, but there is still some amount that remains in the pores of the aggregates, since at equivalent dosages of rejuvenator, the stiffness modulus for the samples with encapsulated rejuvenator is not at the same level than that of the samples with rejuvenator directly added to the mix.

A more in-depth analysis of the results by means of the quantification of the ratio aged/unaged shows interesting findings. According to the data in Table 3, the stiffness increases as the mixtures age; however, the samples with encapsulated rejuvenator do it at a lower rate than the reference mix. This can be confirmed by the stiffness ratio SHRP long term aged/unaged, which is 1.75 and 1.66 for the sepiolites and vermiculite, respectively, and 1.91 for the reference mix. Therefore, the samples with encapsulated rejuvenator after SHRP long-term ageing are less stiff than the samples without out (reference mix). Similar conclusions can be provided from the RILEM ageing protocol.

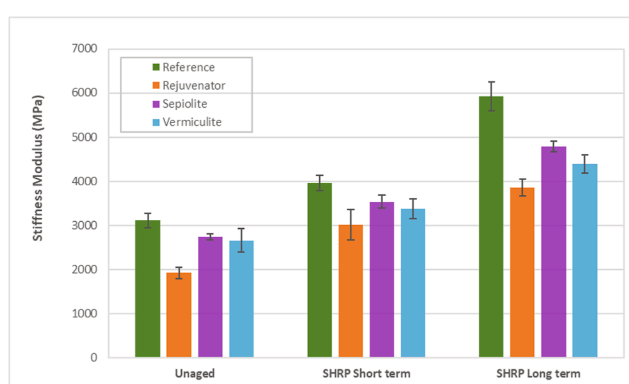

Fig. 7. Average values of the stiffness modulus of the BBTM 11B mixture. SHRP ageing method.

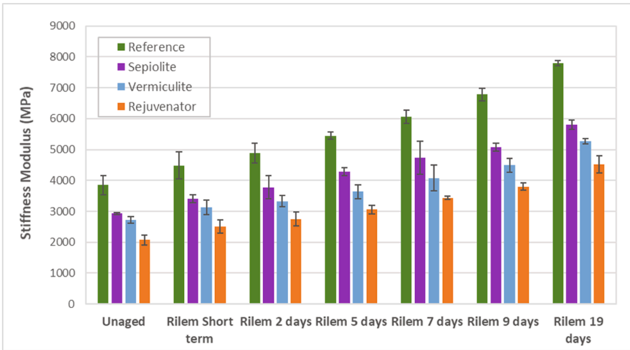

Fig. 8. Average values of the stiffness modulus of the BBTM 11B mixture. RILEM ageing method.

Additionally, when comparing the unaged/aged ratio for all the samples at the different stages of the protocol (short-term ageing, Rilem 9 days and Rilem 19 days), it was found out that in all the scenarios, this ratio was higher for the mix with rejuvenator directly added to the mix than that of the encapsulated rejuvenator. The ratio aged/unaged for RILEM short-term ageing was 1.16 for the reference mix, the same for the mixtures with encapsulated rejuvenator in sepiolites, 1.13 for the samples with vermiculite and 1.18 for the mixture with rejuvenator directly added to the mixture. A long-term ageing (19 days) results in an aged/unaged ratio of 2.02 for the reference mix, 1.96 for the sepiolites capsule, 1.94 for the vermiculite capsule and 2.18 for the rejuvenator. The differences observed between the two types of capsule in relation to the ratio aged/unaged of the mixtures are almost negligible. These results indicated that although the incorporation of rejuvenator directly added to the mix was effective in reducing the stiffness of the asphalt mixtures, its effectiveness was reduced with ageing. The incorporation of encapsulated rejuvenator, conversely, seems to solve this problem, showing a more moderate ageing. This demonstrates the benefit of using encapsulated rejuvenator over the use of rejuvenator directly added to the mix.

In general, results from this test reveal the potential effectiveness of the capsules studied. A change in the stiffness over time is observed in all the samples analysed, but to a lesser extent for the capsules than for the reference and rejuvenator samples.

3.2. Evaluation of the cracking resistance of bituminous mixtures under different ageing levels

Stress and displacement curves corresponding to the individual variables of the Fénix test for the reference sample (without rejuvenator) and the asphalt mixtures with rejuvenator, both directly added to the mixture and encapsulated in sepiolite and vermiculite, tested at 20 °C are shown in Fig. 9. Analysing the shape of the curve, two characteristics of the cracking process can be differentiated: the initial slope of the curve gives an idea of the stiffness of the mixture and the post-peak curve indicates the brittleness of the sample.

Thus, graphs in Fig. 9 show stress-displacement curves with steeper initial slope, reaching a higher peak load, which decreases faster in the post-peak part of the curve, as the mixtures age. It indicates that in all the scenarios tested, the samples become stiffer and more brittle with ageing.

Furthermore, the reference mixture shows higher initial slope and higher peak load than the asphalt mixtures with rejuvenator at each ageing level, being the samples with rejuvenator directly added to the mix those which shows the lowest initial slope and the lowest peak load and the curves for the samples with encapsulated rejuvenator fit between the reference and the rejuvenator mixture. This simple observation of the curves provides a first qualitative assessment of both, the effect of ageing and the way in which the rejuvenator is incorporated into the mix.

Based on the obtained curves, the influence of the ageing on the cracking resistance was quantified through the parameters obtained from the Fénix Test, as shown in Table 4, together with the density and air void content. Some of these parameters (RT, IRT and DT) were plotted for unaged, RILEM short-term aged, Rilem 9 days and Rilem 19 days aged samples as shown from Figs. 10–12. These parameters are considered the most representative ones from the data available from the Fénix test and for the purpose of this study. In addition, given the limited information provided by the results of ageing at intermediate

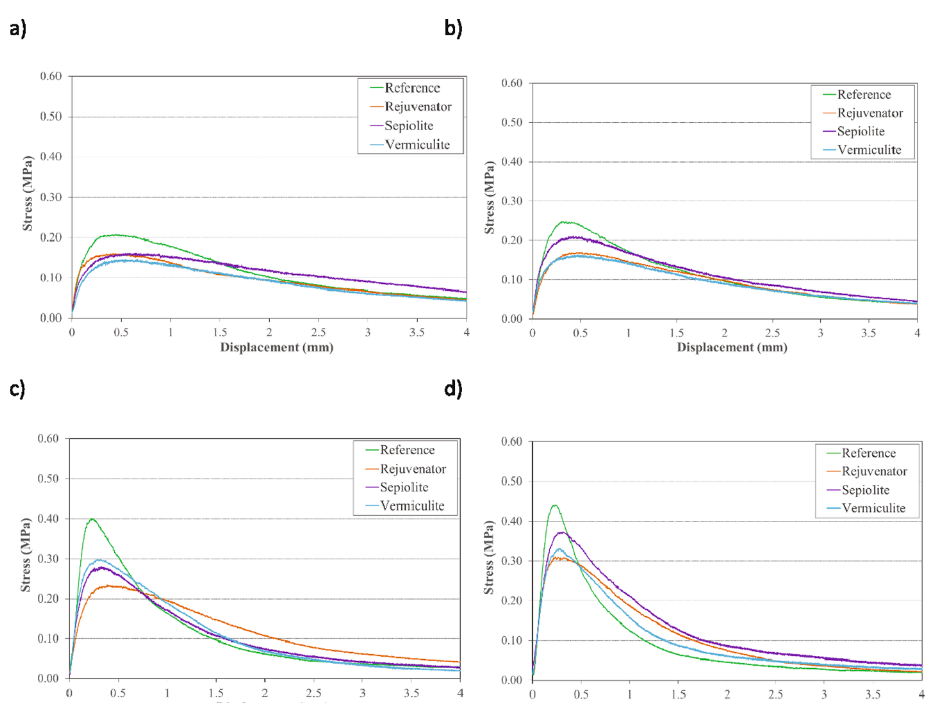

Fig. 9. Stress-displacement curves from the Fénix test. Asphalt mixture BBTM11B a) unaged, b) RILEM short term, c) RILEM 9 days and d) RILEM 19 days.

Table 4
Densities, air void content and parameters from the Fénix test.

Sample	Ageing level	Density (g/cm^3)	Air voids (%)	RT (MPa)	IRT (MPa/mm)	G_D (J/m^2)	IT (mN)	D_{50pm} (mm)	DT (mm)
Reference	Unaged	2.090	17.5	0.22	1.5	463	771	2.25	1.82
	Rilem short-term	2.075	17.9	0.24	1.7	475	626	1.80	1.42
	Rilem 2 days	2.093	17.5	0.35	2.3	456	300	1.00	0.72
	Rilem 5 days	2.090	17.4	0.39	2.5	504	358	1.06	0.77
	Rilem 7 days	2.096	17.2	0.37	2.2	486	335	1.07	0.75
	Rilem 9 days	2.095	17.5	0.40	2.5	390	180	0.76	0.51
	Rilem 19 days	2.096	17.9	0.44	3.1	346	125	0.63	0.40
Rejuvenator	Unaged	2.078	17.8	0.13	0.8	351	858	3.53	2.70
	Rilem short-term	2.085	17.6	0.15	0.9	351	637	2.53	2.02
	Rilem 2 days	2.084	17.6	0.19	1.0	453	733	2.50	1.92
	Rilem 5 days	2.082	17.7	0.20	1.3	381	540	1.86	1.39
	Rilem 7 days	2.083	17.7	0.21	1.3	480	677	2.16	1.67
	Rilem 9 days	2.081	17.7	0.23	1.3	417	500	1.71	1.31
	Rilem 19 days	2.084	17.7	0.30	2.4	430	373	1.20	0.93
Sepiolite capsule	Unaged	2.078	17.8	0.22	1.4	467	778	2.37	1.87
	Rilem short-term	2.077	17.9	0.20	1.3	459	753	2.29	1.81
	Rilem 9 days	2.103	17.3	0.30	1.9	389	257	1.05	0.72
	Rilem 19 days	2.079	17.8	0.35	2.3	458	341	1.08	0.81
Vermiculite capsule	Unaged	2.090	17.9	0.18	1.2	366	544	2.19	1.70
	Rilem short-term	2.085	17.8	0.17	0.9	363	506	2.08	1.58
	Rilem 9 days	2.122	16.8	0.31	2.1	431	332	1.15	0.84
	Rilem 19 days	2.079	17.9	0.32	1.9	385	231	0.96	0.65

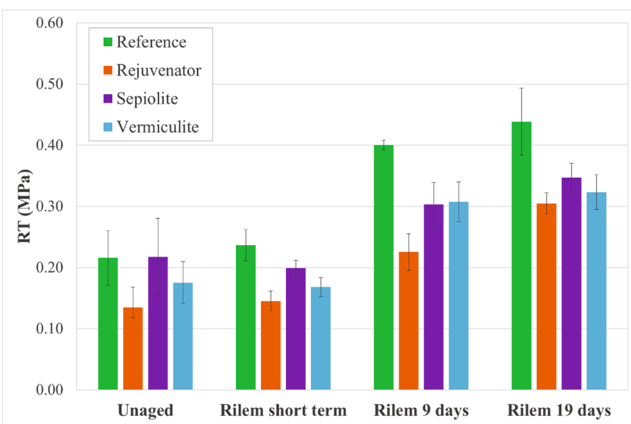

Fig. 10. Average values of the maximum strength of the BBTM 11B mixture.

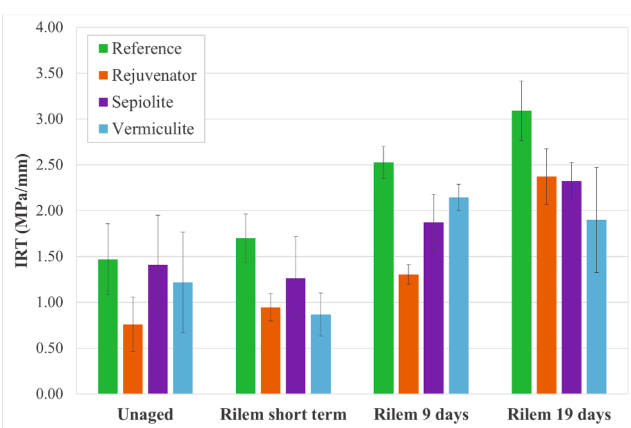

Fig. 11. Average values of the tensile stiffness index of the BBTM 11B mixture.

periods between the short and long term ageing (2, 5 and 7 days) on the reference mixture and the mixture with rejuvenator directly added to the mix, with no visible trends, the mixtures with encapsulated rejuvenator were only tested at short and long term ageing, maintaining the very long-term ageing (19 days). Therefore, the results for ageing at

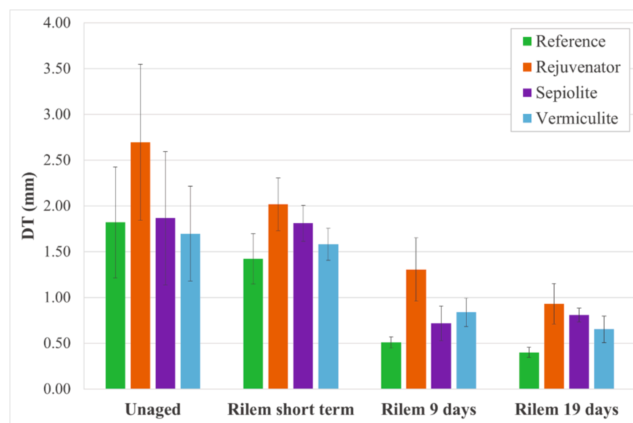

Fig. 12. Average values of the toughness displacement of the BBTM 11B mixture.

intermediate periods of 2, 5 and 7 days are not included in the figures to facilitate the data comparison. The values presented are the average of four specimens, and the error bars represent the standard deviation.

Fénix Diagram allows to graphically analyse the change in the strength and ductility of the asphalt mixture with temperature, by plotting the toughness displacement *(DT*) on the x-axis and the maximum strength (*RT*) on the y-axis. The diagram also shows the limits that, according to the authors' experience with this test method [43], distinguish between the fragile behaviour of the asphalt mixture, ductile and very ductile (vertical lines), and the asphalt mixtures with low strength (horizontal lines). Similarly, the *iso*-toughness curves can be plotted, in which the result of *RT*DT* remains constant. Fig. 13 shows the Fénix Diagram of the asphalt mixtures studied.

The results shows a clear effect of the ageing and the rejuvenator. In general, a decrease of the ductility (DT) and an increase of the resistance (RT) of the bituminous mixture is observed due to the ageing. However, when rejuvenator is added the ductility considerably increases and the resistance decreases, so that the mixture with rejuvenator after long-term ageing (9 days) shows a similar behavior to that of the reference mix (without rejuvenator) for a much shorter conditioning time (short-term ageing).

The reduction of the resistance and the significant increase of the ductility of the asphalt mixture with rejuvenator incorporated in

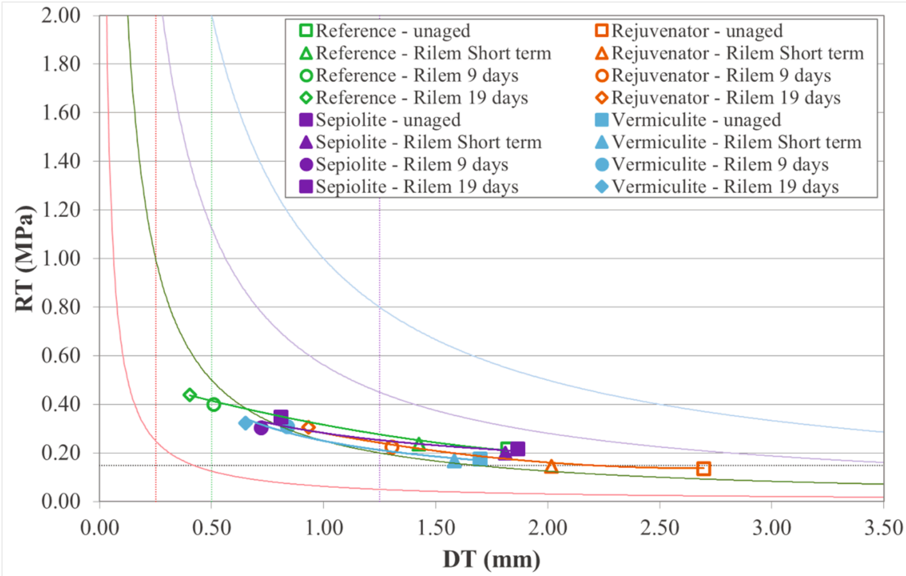

Fig. 13. Fénix Diagram of the BBTM 11B mixture. Reference sample (without rejuvenator) and sample with rejuvenator directly added to the mixture.

comparison with the reference sample without rejuvenator should be noted. This suggests that the addition of rejuvenator directly to the asphalt mixture would probably lead to an excessive deformable mix, especially during the first years in service (unaged), so the addition of rejuvenator in this way would not be recommended.

When the encapsulated rejuvenator is added to the mixture, the unaged asphalt mixtures (or even after short-term ageing) with vermiculite capsule show slightly lower DT values than the sepiolite capsule. According to the RT values, similar conclusions can be drawn, RT values for vermiculite capsules are lower than for sepiolites at initial stages; although these differences are less pronounced than that of *DT*. This suggests that vermiculite capsules are able to retain a bit more rejuvenator agent at early stages of the pavement service life than the sepiolite ones. However, after long-term ageing (9 or 19 days), the differences observed between the two type of capsules are minimal, showing a similar behaviour.

By comparing these results to the ones of rejuvenator directly added to the mixture, it is observed that using capsules, the asphalt mixtures show comparable (or slightly lower) resistance (*RT*) and ductility (*DT*) values than the mixture without rejuvenator. This means that the rejuvenator does not work during the manufacturing stage and in the first years in service. However, the effect of rejuvenator is noticed as the ageing occurs, since RT values are lower than the mixture without rejuvenator and the DT values are higher. In other words, the effect of rejuvenator is evident since the mixture ages as a lower rate than the reference sample.

If we also compare the results obtained with the capsules with those of the mixture in which the rejuvenator was directly added, it is observed that after the mixture manufacturing process (without ageing) with a short-term ageing, the asphalt mixture, either with vermiculites or sepiolites, shows a higher resistance and lower ductility. This confirms that the rejuvenator is not working during these initial stages. In contrast, after long-term ageing (especially after 19 days conditioning), the RT and DT values are similar, showing that the rejuvenator in the capsules has a similar effect as if it had been added directly to the mixture. Actually, these values are not exactly the same to those of the mixture with rejuvenator, which suggests that part of the rejuvenator has not yet worked.

4. Conclusions

In this paper, the effect of ageing on the stiffness behaviour and the cracking resistance on asphalt mixes were studied. Dynamic modulus and Fénix test were the test methods used. The obtained results show some general trends regarding the ageing and the use of rejuvenators, both directly added to the mixture as well as incorporated in capsules:

- Ageing caused a significant increase in asphalt mixture modulus, a slightly decrease in ductility (DT) and an increase in resistance (RT) of the mixture. This effect of ageing can be compensated with the addition of a rejuvenator agent; however, its effectiveness was reduced with ageing, especially after long-term ageing.
- The addition of rejuvenator could considerably reduce the stiffness of asphalt mixtures and increase their ductility, resulting in mixtures more susceptible to rutting. Therefore, the incorporation of rejuvenator to new unaged mixtures is not recommended.
- The addition of encapsulated rejuvenator is an alternative that can solve this problem. This technology helps to release the rejuvenator from the pores of the aggregates as the mixtures age, while maintaining the stiffness of unaged and short-term aged mixtures to similar levels as the reference mixtures.
- The asphalt mixtures with capsules containing rejuvenator showed a more moderate ageing than the samples with rejuvenator directly added to the mix at equivalent dosages and the reference mixtures. This demonstrates the benefit of using encapsulated rejuvenator over its use directly to the asphalt mix.
- Additionally, when the rejuvenator was incorporated in capsules, similar values (slightly lower) of resistance and ductility than the reference mix (without rejuvenator) were shown for the unaged asphalt mixtures. This indicates that the rejuvenator did not work; however, it did as the mixture ages, since the resistance was lower and the ductility higher than that of the mixture without rejuvenator, but these values were not as close as those of the mixtures with rejuvenator, which suggests that part of the rejuvenator had not yet worked.
- Comparing the results obtained with both types of capsules, slightly lower ductility values were obtained with the vermiculite capsule. This likely indicates that these capsules are able to retain more rejuvenator agent in the initial stages, but after long-term ageing (9

or 19 days) the differences between both types of capsules were minimal, since they experienced very similar behavior patterns.

CRediT authorship contribution statement

Raquel Casado-Barrasa: Conceptualization, Methodology, Investigation, Validation, Writing – original draft. **Teresa López-Montero:** Conceptualization, Investigation, Writing – review & editing. **Daniel Castro-Fresno:** Validation, Writing – review & editing, Visualization, Supervision. **Rodrigo Miró:** Methodology, Formal analysis, 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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