- 1 Recycled stone mastic asphalt mixtures incorporating high rates of waste materials
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Recycled stone mastic asphalt mixtures incorporating high rates of waste materials

Abstract:

The new environmental targets set to save natural resources and recover waste materials have been the basis for several scientific studies in different research areas. Consequently, this work aims at developing recycled stone mastic asphalt mixtures with high rates of waste materials, including reclaimed asphalt pavements, waste engine oil products, waste polyethylene and crumb rubber. This new solution was compared with a conventional stone mastic asphalt mixture. Several blends of high penetration bitumens modified with waste materials and reclaimed aged bitumen were evaluated through basic and advanced tests, and the most promising solutions were selected to produce recycled stone mastic asphalt mixtures for further characterization. The water sensitivity, permanent deformation and fatigue cracking performance of those mixtures incorporating high rates of different waste materials were generally improved. The exceptional behaviour of these recycled mixtures together with the high incorporation rate of waste materials demonstrate the innovative character of these solutions for the road paving industry.

- **Keywords:** Stone mastic asphalt (SMA); Recycled mixtures; Reclaimed asphalt pavement; Aged
- 28 bitumen; Waste engine oil; Recycled engine oil bottoms; Polymer modification; Waste polymers;
- 29 Rheology; Mechanical performance

1. Introduction

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Stone mastic asphalt (SMA) mixtures were developed in Germany with the main purpose of resisting high loads from heavy traffic [1] and nailed tires providing a good macro texture. Due to their excellent permanent deformation resistance, several countries are applying these solutions in their road pavements. There is an increase of 20 to 30% in the stability of SMA mixtures when compared with conventional mixtures [2] due to the strong skeleton of interlocked aggregate particles, as well as the high coarse aggregate content [3]. Consequently, Miranda et al. [4] mentioned in their study that SMA mixtures are a very interesting economic and environmental alternative. In addition, SMA mixtures are also known for other advantages, namely their excellent macrotexture (decreasing the water spray on wet surfaces and reducing the noise) and also their increased fatigue life. However, SMA mixtures present some disadvantages such as high production cost when compared with conventional asphalt mixture (although it could be reduced throughout the pavement life cycle due to their durability), low initial skid resistance and risk of binder drainage during transport to the construction site (due to the high binder content) [2, 3]. SMA mixtures are characterized by a gap-graded particle size distribution, high content of coarse aggregates, filler and bitumen, as well as the use of stabilizers [3, 5], which result in a significant consumption of natural resources to produce this type of asphalt mixtures. Thus, the incorporation of different waste materials in SMA mixtures could be investigated in order to develop more sustainable solutions without compromising their recognised performance. The use of reclaimed asphalt pavements (RAP) in new asphalt mixtures brings environmental advantages, such as, a reduction on the volumes of waste sent to landfill as well as on the extraction of new natural resources, and also a reduction on the energy consumption. Moreover, recycled asphalt mixtures could be an economic solution in comparison to traditional asphalt overlay because it eliminates cracking, unevenness and/or other types of pavements distresses [6, 7]. As bitumen is one of the most valuable materials used in pavements [8], the study of recycled mixtures that incorporate a lower amount of new

bitumen is essential, both by reusing aged bitumen (from RAP material) and by modifying the new bitumen with some waste materials, due to the associated economic and environmental advantages.

In the recycling process, it may be necessary to restore the initial properties of the aged bitumen by adding a rejuvenating agent or a chemical additive [9]. Several studies mentioned the use of different types of rejuvenators such as high penetration bitumens, vegetable oils [10, 11], extender oils [12] and waste engine oil [13, 14]. Fernandes et al. [15] studied bitumens modified with waste engine oil products, namely waste engine oil and recycled engine oil bottoms, and polymers (waste polyethylene, crumb rubber and styrene-butadiene-styrene), which were evaluated through a basic characterization. From that study, it was possible to obtain high penetration bitumens modified with waste materials that could be used to restore the properties of the aged bitumen present in RAP material. In order to achieve that, higher contents of waste engine oil products (higher than 15%) were used together with a certain content of polymer.

Additionally, and according to Fernandes et al. [16] the eco-friendly bitumens produced with waste materials (waste engine oil, waste polyethylene and crumb rubber) show low thermal susceptibility, upraised values of high temperature performance grade and also low non-recoverable creep compliance values. When used in the production of SMA mixtures, those bitumens modified with waste materials revealed to be excellent solutions for road paving works due to their good mechanical and surface performance without affecting the environment or the human health.

Therefore, the main purpose of the present work was to develop recycled SMA mixtures produced with high rates of waste materials in order to increase the incorporation of end-of-life materials and minimize the extraction of new materials, according to the new circular economy paradigm. The high rates of waste materials should be achieved by using 50% RAP material, as well as, high penetration bitumens modified with waste engine oil products and waste or virgin polymers. According to Wang et al. [17], Noferini et al. [18] and EP [19], the common rates of RAP material used nowadays are limited to up to 30% and this works intends to go beyond this limit. Furthermore, these recycled mixtures produced with waste modified bitumens should present a performance similar to or even better than a conventional SMA mixture to assure an adequate performance when applied in road pavements.

2. Materials and Methods

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2.1. Materials used in this study

The materials used for partially replacing virgin bitumen, as well as the waste and virgin polymers used in this study were similar to those used in the work carried out by Fernandes et al. [15] and Fernandes et al. [16]. Thus, waste engine oil (EO), recycled engine oil bottoms (RB), waste high density polyethylene (HDPE), crumb rubber (CR) and styrene-butadiene-styrene (SBS) were used for bitumen modification. The EO and RB were supplied by a certified waste engine oil collection company (Sogilub, Lda), and their properties can be seen in the previously mentioned works. Additionally, the waste polymers (HDPE and CR) were supplied by Gintegral S.A. and Recipneu Lda, respectively, while SBS was supplied by Industrias Invicta, S.A. The bitumen used for the modification process was a conventional bitumen with a penetration between 35 and 50×10⁻¹mm, hereinafter referred to as B35/50. This conventional bitumen was supplied by Cepsa Portugal and showed a penetration value of 35×10⁻¹ mm and softening point temperature of 54 °C, according to EN 12591 standard. In addition, a softer conventional bitumen with a penetration grade between 160 and 220×10⁻¹mm (according EN 12591 standard) was also studied to rejuvenate the aged bitumen. These binders were also used for comparative purposes in both bitumen and asphalt mixtures characterization. Regarding the new aggregates used in this work for production of SMA mixtures, whose requirements fulfil those established by the EN 13043 standard, only one fraction (6/14 mm) of crushed granite supplied by Bezerras, Lda. was selected. Additionally, limestone filler supplied by Omya Comital S.A. was also used.

The acrylic fibres used in the control recycled SMA mixture presented a length between 6 and 12mm and

a nominal diameter of 14.4 µm. This control recycled SMA mixture was produced with a conventional

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B160/220 bitumen according to the results of a previous study [16], where 0.3% fibres (by mass of aggregates) were used in the control SMA mixture produced with a conventional B35/50 bitumen.

Since the main goal of this work is to study recycled SMA mixtures, a reclaimed asphalt pavement (RAP)
material obtained from an end-of-life highway surface course was also used and evaluated in this study.
The RAP material was divided into two main fractions (fine and coarse fraction), through an industrial
material classifier with an 8 mm mesh size, and then supplied by the company ELEVO Group. This
separation allowed a better adjustment of the grading curve because SMA mixtures are known for their
gap-graded particle size distribution.

112 2.2. *Methods*

2.2.1. Characterization of reclaimed asphalt pavement

Since both RAP fractions comprise particles of different dimensions, the proportion of aged bitumen and aggregates is different between them, as the smaller aggregate particles are usually covered by a higher amount of bitumen due to their higher specific surface area. Thus, it is necessary to determine the binder content (EN 12697-39) and aggregates gradation (EN 12697-2) of each RAP fraction. This information is crucial for the recycled asphalt mixtures design. In addition, the aged bitumen should also be recovered (according to EN 12697-3) in order to characterize its properties and to study the interaction between this binder and the new bitumen/modifiers.

To determine the binder content (EN 12697-39) a dry sample of RAP material was placed in a furnace and heated up to 540 °C until all the bitumen had been burned. The bitumen weight is obtained by the difference between the total weight of the sample and the weight of the sample after burning. The fine fraction of RAP presented a binder content of 6.0%, while the coarse fraction showed a binder content of 4.1%. The particle size distribution of the aggregate present in the fine and coarse fractions of the RAP material can be seen in Figure 1.

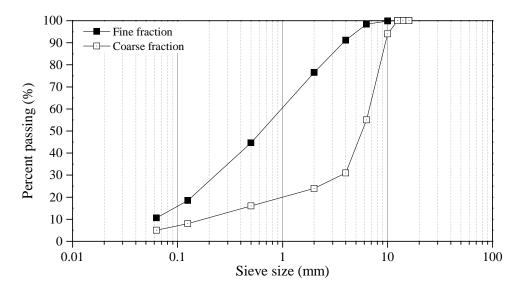


Figure 1. Particle size distribution of the aggregate present in the fine and coarse fraction of the RAP

Although the fraction separation has been made in an 8 mm sieve, the coarse fraction is essentially comprised of aggregates with a dimension between 4 and 10 mm, having yet 30% of material with a dimension smaller than 4 mm (related to fine material that is involving the coarser aggregates). On the other hand, the fine fraction showed a continuous grading with a higher percentage of thinner material (maximum dimension of 6 mm).

The RAP characterization also consists in recovering the aged bitumen through rotary evaporator method, according to EN 12697-3. Since both fractions were used in this study, each fraction was added in the respective content, according to the selected recycled SMA 14 mixture gradation (shown in Table 1). Within the RAP material incorporated in the final mixture (corresponding to 50% of the final recycled mixture), the proportion of the coarse fraction was 70%, while the proportion of the fine fraction was 30%.

The aged bitumen recovery is a three stage process. The first stage consists in placing the RAP material in a container together with a suitable solvent until all bitumen has visibly been dissolved, followed by a separation by centrifugation and filtration (to separate the coarse aggregates). In the second stage, the fine material is separated from the bitumen/solvent solution through an additional centrifugation process at a higher speed (3000 rpm), during 10 min. The last stage of the process includes a distillation process, where the solvent is separated from the aged bitumen in a rotary evaporator.

To characterize the aged bitumen, penetration at 25 °C (EN 1426 standard), softening point temperature (EN 1427 standard) and dynamic viscosity (EN 13302 standard) tests were carried out. The aged bitumen presents a penetration value of 8.8×10^{-1} mm and a softening point temperature of 74 °C, confirming the hardness of the bitumen present in the RAP material. In relation to dynamic viscosity, at the temperatures of 135 °C and 180 °C, the aged bitumen shows a dynamic viscosity of 3.8 Pa.s and 0.3 Pa.s, respectively. The mixing temperature of the aged bitumen is clearly higher than the mixing temperatures needed for the conventional bitumens most typically used for road paving in Portugal (between 150 to 165 °C). The properties of the aged bitumen are essential for further characterization and selection of the bitumens modified with waste materials.

2.2.2. Production of the recycled binders

For the production of the recycled binders, in order to rejuvenate the aged bitumen, it is necessary to produce high penetration bitumens modified with waste materials. The content of each bitumen substitute and virgin or waste polymer, as well as the production process were selected and the bitumens modified with waste materials were produced based on Fernandes et al. [15] and Fernandes et al. [16] works. The modified bitumens should present higher penetrations, in order to get a recycled binder with a penetration value situated within the range most commonly used for paving works (35 to 70×10^{-1} mm) [20].

Thus, the bitumen substitute content should be higher than 15%, allowing the use of higher amounts of RB than EO. Additionally, the waste HDPE should be used in a content of 6%, the SBS in a content of 5% and the CR in a content of 20%, in order to optimize their effect on the properties of the final binders produced with waste materials. These were produced by modifying a conventional bitumen B35/50 with the waste materials in a high shear mixer, at a temperature of 180 °C, during a period of 20 min [15, 16].

In order to compare the results of the present study with those of the control SMA mixture of Fernandes et al. [16] study (SMA-BF0.3), the binder content used to produce the recycled SMA mixtures was 5.8%. Therefore, the recycled binders were produced by adding 3.5% of bitumens modified with waste materials and 2.3% of aged bitumen (obtained according the respective proportion of each RAP fraction). The effect

of the production process (addition of the bitumens modified with waste materials to the RAP material and new aggregates and filler) on the properties of the final binder was simulated by using a low shear mixer, at a speed of 350 rpm and a temperature of 180 °C, for a period of 2 min (estimated time that would be used to mix the fine RAP fraction, introduced at ambient temperature, with the hot new aggregates and filler).

2.2.3. Characterization of recycled binders

- The bitumens modified with waste materials and the recycled binders were evaluated by a basic characterization, namely through penetration at 25 C (EN 1426 standard), softening point temperature (EN 1427 standard) and dynamic viscosity (EN 13302 standard) tests.
- Besides this basic characterization, the selected recycled binders (with the most promising properties) were also evaluated through a more advanced characterization, such as rheological tests. Thus, two additional tests were carried out, the first to obtain the complex modulus and phase angle (according to EN 14770 standard) and the second to obtain the non-recoverable creep compliance parameter (according to AASHTO TP 7011 standard). These parameters allow estimating the behaviour of the recycled binders at low and high temperatures, as well as, estimating their permanent deformation performance, when used in asphalt mixtures.
- The first rheological test was carried out at different temperatures (30, 45, 60, 70 and 80 °C) and frequencies (between 0.1 to 20 Hz), using a dynamic shear rheometer. The second test, known as Multiple Stress Creep Recovery (MSCR) test, was carried out at different stress levels (25, 50, 100, 200, 400, 800, 1600, 3200 Pa [21]), at the high performance grade temperature specified for the Portuguese conditions (64 °C).

191 2.2.4. Production of recycled SMA mixtures

Prior to the production of the recycled SMA mixtures, their aggregate gradation was selected, taking into accounting that this type of mixtures present a gap-graded particle size distribution. Consequently, in order

to fulfil the specified limits for SMA mixtures, 35% of coarse RAP fraction and 15% of fine RAP fraction were used. As previously mentioned, these proportions influenced the effective aged binder content (2.3%). In relation to the new aggregates and filler, it was necessary to add 47% of 6/14 fraction and 3% of filler. Table 1 shows the selected gradation of the recycled SMA 14 mixtures, as a result of the content of RAP material, aggregates and filler mentioned above, as well as the limits specified for SMA 14 mixtures in EN 13108-5 standard.

Table 1. Selected gradation of the recycled SMA 14 mixtures

Sieve size	Percent passing (%)		
(mm)	Selected gradation	Lower limit specified	Upper limit specified
20.0	100.0	100.0	100.0
14.0	95.6	90.0	100.0
10.0	70.9	Not required	Not required
6.3	38.9	Not required	Not required
2.0	23.7	15.0	30.0
0.063	6.4	5.0	12.0

The recycled SMA mixtures were produced based on a three stage process. The first comprises the addition of new aggregates, filler and coarse fraction from RAP material, at a temperature 30 °C above the mixing temperature of each bitumen, mixing them for about 1 minute, until a homogenous blend is observed. In the second stage, the fine fraction from the RAP material is added into the previous blend, at room temperature, and the materials are mixed for another 2 minutes period to guarantee that all materials acquire an evenly distributed temperature. Lastly, the conventional bitumen or the bitumen modified with waste materials is added, and mixed for an additional 2 minutes period, at the corresponding mixing temperature. This process is based on a previous work [22] where two RAP fractions were used to produce recycled asphalt mixtures, and it simulates the production process of a recycled mixture in a batch mixing plant. It should be noted that for comparison purposes, a control recycled mixture was produced, during which acrylic fibres were added in the second stage of the process, at room temperature.

2.2.5. Characterization of recycled SMA mixtures

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The recycled SMA mixtures were mechanically characterized with the aim of evaluating if these recycled

SMA mixtures produced with bitumens modified with waste materials have a better performance than a

conventional SMA mixture and a recycled SMA mixture both produced with a conventional bitumen.

Since higher contents of waste materials (RAP and modified bitumens with waste materials) were

incorporated in the mixtures developed in this work, in order to minimize the use of new aggregates and

conventional bitumen, it is crucial to guarantee an adequate mechanical performance of those mixtures.

Therefore, all the recycled mixtures were evaluated through the following tests: i) water sensitivity

(according to EN 12697-12 standard); ii) stiffness modulus in four point bending beam (according to EN

12697-26 standard); iii) fatigue cracking resistance in four point bending beam (in accordance with

AASTHO TP 8-94); and, iv) permanent deformation resistance at 60 °C (according to EN 12697-22). The

results obtained in those test allowed a comparison with the performance of the SMA 14 mixture produced

with a 35/50 bitumen and fibres from Fernandes et al. [16] work.

Besides the mechanical evaluation, the recycled asphalt mixtures were also evaluated through the binder

drainage test, using the basket method (EN 12697-18), since SMA mixtures could present some separation

between mastic and aggregates during transport to the construction site, also called binder drainage, due

to their high binder content and gap-graded aggregate gradation [3].

3. Results and Discussion

- 3.1. Characterization of bitumens modified with waste materials developed for production of recycled
- 232 SMA mixtures

One of the goals of this work is to produce recycled SMA mixtures with 50% RAP material. In order to

obtain a final recycled binder in those mixtures with a penetration value similar to that of the most

commonly used bitumens in the road paving industry in Portugal (B35/50 and B50/70), a new high

penetration bitumen must be added to balance the very low penetration at 25 °C of the aged bitumen from

RAP (8.8×10⁻¹ mm). A similar approach can be observed in the work carried out by Abreu [20], which used 2.5% of a high penetration bitumen (B160/220) to produce recycled asphalt concrete mixtures with 50% RAP (containing 2.6% bitumen with a penetration of 8.9×10⁻¹ mm), thus obtaining a final recycled binder with a penetration value typical of a B35/50 bitumen.

Thus, a conventional bitumen with a penetration value between 160 and 220×10⁻¹ mm was used as the control bitumen for production of a recycled SMA mixture. Then, different bitumens modified with waste materials were selected in order to present characteristics of a PMB75/130, and they were used as alternative materials for production of recycled SMA mixtures. These PMB75/130 bitumens should present a penetration ranging from 75 to 130×10⁻¹ mm and a softening point temperature equal or higher than 55 °C. The contents of the different waste materials (EO, RB, CR and HDPE) used to produce the modified bitumens was selected according to the study of Fernandes et al. [16], as previously mentioned. Higher amounts of RB and polymers could be used to obtain characteristics similar to those of a polymer modified bitumens produced with EO, as explained in Fernandes et al. [15].

The penetration, softening point and dynamic viscosity results of the conventional bitumen B160/220, as well as those of the bitumens modified with waste materials, are presented in Table 2. These bitumens were selected as being the most suitable for production of recycled SMA mixtures, while maximizing the use of waste materials The notation used in this study to classify the bitumens modified with waste materials was "aXbY", where: "a" is for EO or RB, depending on the type of partial bitumen substitute used; "X" stands for the percentage of the partial bitumen substitute used; "b" is for H (HDPE), S (SBS) or R (CR), according to the type of polymer added; and "Y" stands for the percentage of polymer added.

For each combination of polymer (HDPE, SBS or crumb rubber) and partial bitumen substitute, i.e. waste engine oil (EO) or recycled engine oil bottoms (RB), two different bitumens modified with wastes were selected and tested. Although higher amounts of RB than EO have been used as partial bitumen substitute, the penetration values of the polymer modified bitumens produced with EO or RB are analogous. Regarding the softening point temperatures, it should be noted that the bitumens modified with CR and RB are slightly below the minimum required limit of a PMB75/130, even though they are the best blends

produced with those specific materials. Nevertheless, all the bitumens modified with wastes have softening point temperatures clearly higher than that of the control bitumen B160/220. Thus, all these bitumens were combined with the aged bitumen recovered from RAP in the next phase of this work, in order to study the properties of the final binders in the recycled SMA mixtures.

Table 2. Basic properties of control bitumen and bitumens modified with waste materials developed for production of recycled SMA mixtures

Ditaman	Penetration at 25 °C	Softening point temperature	Mixing temperature
Bitumen	(0.1 mm)	(°C)	(°C)
B160/220	191	38	130
EO15H6	82	113	180
EO20H6	106	109	180
EO15S5	97	86	180
EO20S5	117	82	180
EO15R20	81	59	180
EO20R20	128	55	180
RB17.5H6	83	84	180
RB20H6	101	87	180
RB17.5S5	90	89	180
RB20S5	105	82	180
RB20R20	110	49	180
RB22.5R20	117	48	180

3.2. Selection of recycled binders through their basic characterization

The recycled binders were only produced after setting a binder content of 5.8% for the recycled SMA mixture, as mentioned previously. Thus, the recycled binders were obtained by mixing 3.5% of each bitumen modified with waste materials previously mentioned in Table 1, as well as the control bitumen B160/220, with 2.3% of aged bitumen recovered from RAP material. The binder content of RAP and the recycling rate of the SMA mixture (i.e. 50%) were also taken into account to define the previous

proportions. The notation used to classify these recycled binders is the same of the corresponding bitumens
 modified with waste materials, but preceded by "R" letter.

After production and characterization of the recycled binder used as reference (RB160/220), which was produced with the control bitumen B160/220, a penetration value of 63×10⁻¹ mm, a softening point temperature of 47 °C, and mixing and compaction temperatures respectively of 150 °C and 130 °C were obtained. Thus, the recycled binder RB160/220 shows characteristics of a conventional bitumen B50/70, as initially defined for this study.

The basic properties of the recycled binders incorporating 6% HDPE are presented in Figures 2 and 3 as a function of the partial bitumen substitute (EO or RB) contents. The penetration values of the recycled binders with EO are higher than those with RB. Moreover, the increase in the penetration values when increasing the partial bitumen substitute content is more evident in the recycled binders with EO than with RB. In fact, for an equivalent partial bitumen substitute content (20%), the penetration value of the recycled binder with EO (REO20H6) is 89×10⁻¹ mm, while it is only 71×10⁻¹ mm for the recycled binder with RB (RRB20H6).

The recycled binders with RB have equivalent softening point temperatures (50.0 and 50.4 °C) for different RB contents. However, the softening point temperatures of the recycled binders with EO decrease (from 52 °C to 47 °C) with the increase in the partial bitumen substitution content. Thus, the recycled binders produced with HDPE and RB are less susceptible to changes in the partial bitumen substitution content than the recycled binders with EO.

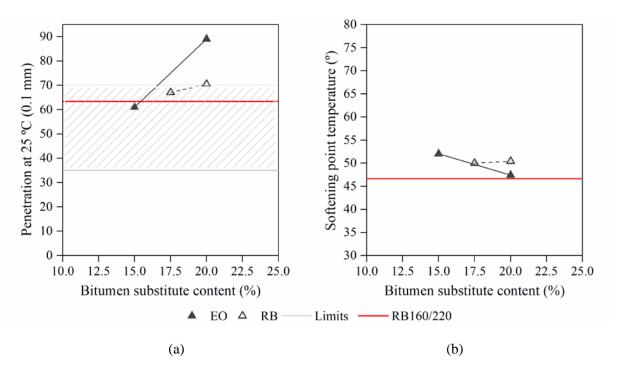


Figure 2. Basic properties of the recycled binders with EO or RB and 6% HDPE: (a) penetration and (b) softening point temperature

The recycled binders with HDPE that best fit both selection criteria, namely a penetration between 35 and 70×10^{-1} mm and a higher content of waste materials, are the recycled binders with 15% EO and 6% HDPE (REO15H6) and with 17.5% RB and 6% HDPE (RRB17.5H6).

The dynamic viscosity values of the recycled binders with HDPE are higher than those of the recycled binder RB160/220 (control binder), for all test temperatures (Figure 2). Besides, all recycled binders with HDPE have similar dynamic viscosities, regardless of the type and content of the partial bitumen substitute used. These similar dynamic viscosity results are mainly related with the effect of the aged bitumen in the final properties of the recycled binders. It should be highlighted that the dynamic viscosities used to define the mixing and compaction temperatures were respectively 0.3 Pa.s and 2 Pa.s. Based on these results, the mixing and compaction temperatures of the mixtures with these recycled binders are around 180 °C and 135 °C, respectively.

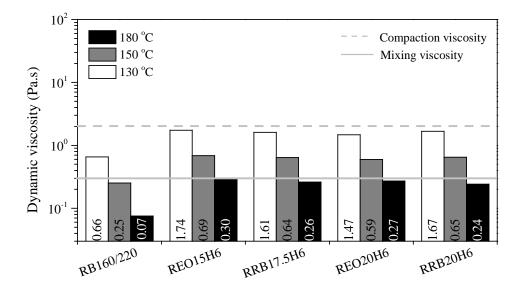


Figure 3. Dynamic viscosity of the recycled binders with EO and RB and 6% HDPE

A similar analysis was carried out for the recycled binders produced with the other polymers used in this study, namely SBS and CR. Thus, the basic properties of the recycled binders with 5% SBS are presented in Figures 4 and 5. The partial bitumen substitute contents are the same as for the recycled binders with HDPE, i.e. 15% and 20% EO and 17.5 and 20% RB.

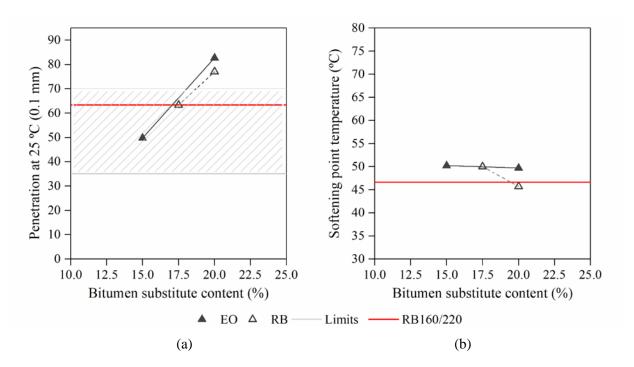


Figure 4. Basic properties of the recycled binders with EO or RB and 5% SBS: (a) penetration and (b) softening point temperature

When increasing the partial bitumen substitute content of the recycled binders with SBS, the penetration values also increase in a similar way, independently of using EO or RB. Nevertheless, the penetration values of the recycled binders with SBS and RB are slightly lower. The penetration values of the recycled binders with SBS are above the upper limit established in this work (70×10⁻¹ mm) for an amount of 20% bitumen substitute, both for EO or RB. Thus, lower contents of EO or RB must be used in the recycled binder with SBS in order to fulfil this selection criteria.

The softening point temperatures of the recycled binders with SBS (Figure 4b) decrease (from 50 °C to 46 °C) with the increase of RB content, while remaining nearly constant (around 50 °C) for different amounts of EO.

The recycled binders with SBS selected according to the previously mentioned criteria are the recycled binders REO15S5 (with 15% EO and 5% SBS) and RRB17.5S5 (with 17.5% RB and 5% SBS).

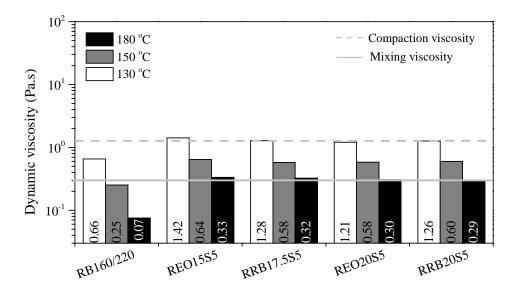


Figure 5. Dynamic viscosity of the recycled binders with EO and RB and 5% SBS

The dynamic viscosity results of the recycled binders with SBS are similar to those observed for HDPE. These binders also present higher dynamic viscosity values than those of the recycled binder RB160/220 (control), and have equivalent properties independently of the type or amount of bitumen substitute used (EO or RB). Furthermore, the dynamic viscosity results indicate mixing temperatures around 180 °C and compaction temperatures rounding 135 °C for mixtures produced with these binders with SBS.

Finally, the basic properties of the recycled binders with CR were also evaluated for two EO (15% and 20%) and RB (20% and 22.5%) contents, as shown in Figures 6 and 7. The penetration values of the recycled binders with EO and CR significantly increase with the increase of EO content. However, the change in penetration values of the recycled binders with RB and CR is negligible for different contents of RB. Thus, the amount of EO incorporated in recycled binders with CR must be limited, but higher amounts of RB can be used in order to obtain similar penetration values.

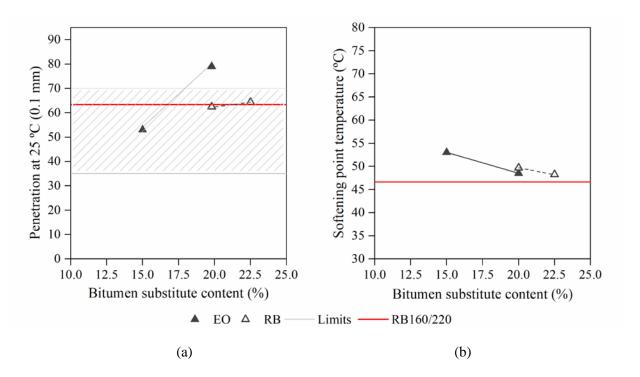


Figure 6. Basic properties of the recycled binders with EO or RB and 20% CR: (a) penetration and (b) softening point temperature

Regarding the softening point temperatures, all recycled binders with CR present higher softening point temperatures than RB160/220 (control recycled binder), especially when lower contents of partial bitumen substitute are used. Thus, although the modified bitumens with CR and RB had not fulfilled the softening point temperature requirements of a PMB75/130 (as observed previously), the corresponding recycled binders have an adequate behaviour after being added to the aged bitumen. This result is a consequence of using a very high amount of crumb rubber (20%) in the binder, which may be partially interacting with the aged bitumen.

Thus, the recycled binders with CR that that best fit both selection criteria previously mentioned are the recycled binder REO15R20 (15% EO and 20% CR) and the recycled binder RRB22.5R20 (22.5% RB and 20% CR). Although higher amounts of partial bitumen substitute may have been selected (especially for EO), these binders already have high amounts of waste incorporation and were selected to minimize the risk of inadequate performance during the evaluation of the corresponding mixtures.

The dynamic viscosity results (Figure 6) of the recycled binders with CR are different from those previously obtained for HDPE and SBS. In fact, even though the recycled binders with CR and EO have similar viscosities for different amounts of EO, the recycled binders with CR and RB present lower dynamic viscosities when the amount of RB increases, within the range of temperatures studied. This result could be related with the high RB contents used (20 and 22.5%), which could have a significant influence in the properties of the recycled binder at high temperatures.

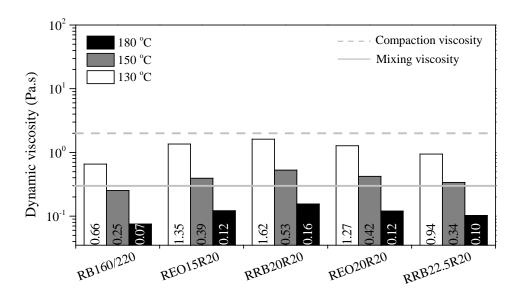


Figure 7. Dynamic viscosity of the recycled binders with EO and RB and 20% CR

It should be noted that all recycled binders with CR have higher dynamic viscosity values than those of the control recycled binder (RB160/220). Based on these dynamic viscosity results, the mixing and compaction temperatures of mixtures produced with the recycled binders with CR and EO should be 170 °C and 135 °C, respectively. Moreover, the mixing and compaction temperatures of mixtures incorporating recycled binders with CR and RB should be, respectively, 160 °C and 135 °C.

The six recycled binders selected in this phase of the work are the most promising for further evaluation in the next sections of this work. Next, those binders are characterized through advanced testing and compared with the control recycled binder (RB160/220) and with the conventional bitumen B35/50 used for production of SMA mixtures in the Fernandes et al. [15] work.

3.3. Advanced characterization of the selected recycled binders

First, the stiffness complex modulus and phase angle properties were used to evaluate the performance of the studied binders under different loading times and temperature settings [13, 23, 24]. Based on these two properties is possible to calculate the high temperature performance grade (PG) of the studied binders [25], which is used for their classification.

The complex modulus master curves of the studied binders are presented in Figure 8a. The master curves of the recycled binders with waste materials present a similar shape to those of the control recycled binder (RB160/220) and the conventional bitumen (B35/50), which suggests a similar rheological behaviour among all studied binders. Moreover, when compared with the control recycled binder (RB160/220), the recycled binders with waste materials present higher complex modulus, except for the recycled binder with CR and RB (due to the high amount of bitumen substitute used).

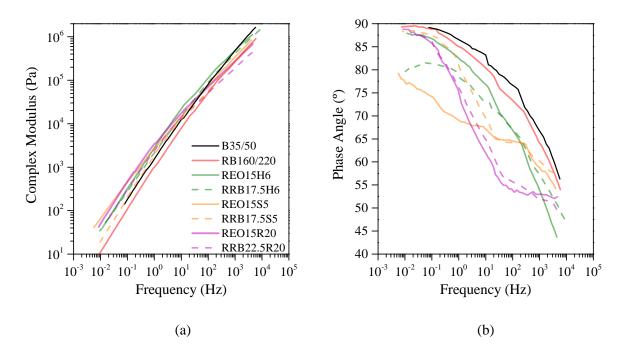


Figure 8. Master curves of the studied binders ($T_{ref} = 60$ °C): (a) complex modulus and (b) phase angle

In general, the recycled binders with waste materials have higher complex moduli at low frequencies (high temperatures) and lower or similar complex moduli at high frequencies (low temperatures) when compared with the conventional bitumen (B35/50) and control recycled binder (RB160/220), demonstrating their lower thermal susceptibility. These results could indicate an appropriate flexibility of those recycled binders at low service temperatures and, at the same time, a higher resistance to permanent deformation at high temperatures. Therefore, the operating temperature range of these new recycled binders with waste materials is higher than that of the reference binders, i.e. the control recycled binder (RB160/220) and the conventional bitumen (B35/50).

The phase angle master curves of the studied binders are presented in Figure 8b. Within the temperature/frequency range studied, the recycled binders with waste materials show lower phase angles than those of the reference binders (B35/50 and RB160/220), demonstrating a less viscous nature of the new binders with waste materials. The shape of the phase angle master curves of the recycled binders with SBS or CR is different from the remaining ones, mainly due to the elastomeric properties of SBS and CR polymers, which allows to increase the elastic component of the stiffness modulus of these binders (thus reducing the phase angle values over a wide range of frequencies).

Based on the previous rheological results, the high temperature performance grade (PG) of the studied binders was also assessed, as presented in Table 3.

Table 3. High temperature performance grades of the studied binders

Binder	High temperature Performance Grade	
B35/50	64	
RB160/220	64	
REO15H6	70	
REO15S5	70	
REO15R20	70	
RRB17.5H6	70	
RRB17.5S5	64	
RRB22.5R20	70	

The high temperature PG of the recycled binders with waste materials is higher than that of the control recycled binder (RB160/220) and the conventional bitumen (B35/50), except for the recycled binder with 17.5% RB and 5% SBS (RRB17.5S5) that have equivalent high temperature PG. The lower PG grade of binder RRB17.5S5 may have resulted from combining a high amount of bitumen substitute (17.5% RB) with a slightly lower amount of polymer (5%) in comparison with the remaining binders.

Binders with higher PG grades are expected to result in asphalt mixtures with better rutting resistance performance. Consequently, a good permanent deformation resistance of the recycled SMA mixtures produced with the binders modified with waste materials is expected to be obtained, in most cases, in the next phase of this work.

The multiple stress creep recovery test (MSCR) can also be used to estimate the permanent deformation performance of the studied binders, using the non-recoverable creep compliance parameter J_{nr} . The lower the value of this J_{nr} parameter, the higher the permanent deformation resistance. Figure 9 shows the

non-recoverable creep compliance parameter variation of the studied binders with the increase of stress applied in the MSCR test.

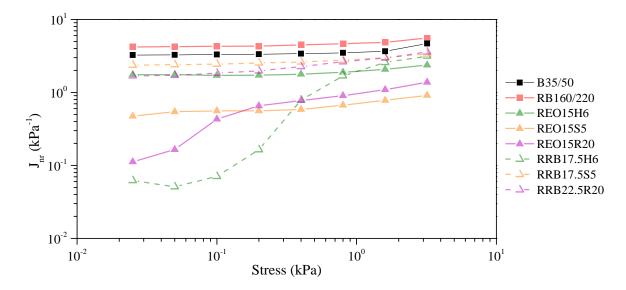


Figure 9. Non-recoverable creep compliance values of the studied binders at different stress levels

The recycled binders with waste materials have lower non-recoverable creep compliance values comparatively to both reference binders (control recycled binder RB160/220 and conventional bitumen B35/50). Moreover, the recycled binder with 17.5% RB and 5% SBS also presents high J_{nr} values similar to those of the reference binders, which is in agreement with the high temperature PG values previously presented for these specific binders.

It should be noted that the several recycled binders with EO, as well as the recycled binder with 17.5% RB and 6% HDPE, present lower J_{nr} values and therefore should have better rutting resistance performance. Furthermore, the recycled binders REO15R20 and RRB17.5H6 are more susceptible to changes in the stress applied during the MSCR test, and their use should be carefully evaluated depending on the expected traffic level.

After rheological characterization, it was concluded that the selected recycled binders with waste materials present, in general, an adequate performance to be used in the next phase of this work for recycled SMA mixture characterization. In particular, those binders have high permanent deformation resistance, which

- is usually higher than that of the control recycled binder (RB160/220) and the conventional bitumen
- 441 (B35/50).
- 442 3.4. Characterization of the SMA recycled asphalt mixtures with modified bitumens
- 443 After selecting the recycled binders and performing their advanced characterization, in the third phase of
- this work, different recycled SMA mixtures were produced with a selection of the modified bitumens and
- characterized in order to evaluate their performance through mechanical tests. Thus, seven recycled SMA
- 446 14 mixtures were produced with the selected modified bitumens (a control conventional bitumen, three
- bitumens modified with EO and each polymer and three bitumens modified with RB and each polymer).
- The corresponding recycled SMA mixtures were identified as follows:
- SMA-RBF0.3 (conventional 160/220 pen bitumen + 0.3% fibres);
- SMA-REO15H6 (bitumen modified with 15% EO and 6% HDPE);
- SMA-REO15S5 (bitumen modified with 15% EO and 5% SBS);
- SMA-REO15R20 (bitumen modified with 15% EO and 20% CR);
- SMA-RRB17.5H6 (bitumen modified with 17.5% RB and 6% HDPE);
- SMA-RRB17.5S5 (bitumen modified with 17.5% RB and 5% SBS); and,
- SMA-RRB22.5R20 (bitumen modified with 22.5% RB and 20% CR).
- 456 It should be highlighted that all these mixtures were also compared with the control mixture studied in
- 457 Fernandes et al. [16]. The SMA mixture in the mentioned work was produced with 5.8% of a conventional
- 458 B35/50 bitumen and 0.3% fibres. Thus, the recycled SMA mixture with the conventional B160/220
- bitumen was also produced with the addition of 0.3% fibres in order to reduce the binder drainage that
- 460 could occur. No fibres were added to the other recycled mixtures because the use of modified binders and
- the use of RAP material limit the binder drainage problems. However, all the mixtures were evaluated
- through binder drainage test to assure an adequate performance.

3.4.1. Binder drainage

According to Blazejowski [3] the higher binder content of SMA mixtures could lead to binder drainage. Consequently, all the SMA mixtures studied in this work were evaluated through the binder drainage test.

The results obtained for all SMA mixtures (including the SMA mixture produced without RAP material and used for comparison purpose - SMA-BF0.3) are lower than the limit recommended in the literature (0.2%), as can be observed in Figure 9. The drainage test values of the recycled SMA mixtures with modified binders are higher than the recycled SMA mixture with conventional bitumen and fibres (SMA-RBF0.3) and the SMA mixture with fibres (SMA-BF0.3). Nevertheless, all those recycled asphalt mixtures produced with modified binders have shown that the use of fibres is not necessary, as the introduction of polymers and the presence of RAP material are sufficient to prevent the binder drainage.

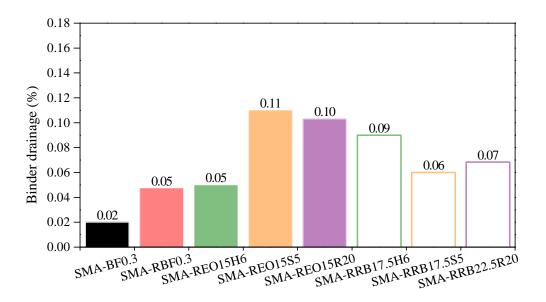


Figure 10. Binder drainage values of the studied SMA recycled asphalt mixtures

It should be highlighted that the recycled SMA mixtures with RB and elastomers (SBS and CR) exhibit lower binder drainage values in comparison to the recycled mixtures with EO and the same polymers. The higher amounts of RB added may ensure a better polymer dispersion in the recycled mixture, even after the interaction with the aged bitumen. However, the same does not happen in the recycled SMA mixtures with RB and HDPE because the HDPE is easily dispersed in bitumen, regardless of the amount of bitumen

substitute used. In this case, the high amount of RB (in comparison with EO) led to a decrease of modified bitumen's viscosity, slightly increasing its binder drainage.

3.4.2. Water sensitivity

Since the recycled SMA mixtures do not present binder drainage problems, they were tested to assess their water sensitivity. According to Sarang et al. [26] SMA mixtures should exhibit a minimum tensile strength ratio (TSR) of 80%, but NAPA [27] mentioned a minimum value of 70%. In Figure 10, it is possible to observe that all the recycled asphalt mixtures show TSR values above the minimum recommended value (80% - represented in the graph by the grey line). The same behaviour could be seen in the control mixture without RAP material (SMA-BF0.3), whose TSR value is 87%. Thus, all the SMA mixtures studied have reduced sensitivity to water. This good performance of all SMA mixtures can be partially associated with the use of limestone filler, which improves the adhesion between asphalt binders and aggregates, reducing their water sensitivity [28, 29].

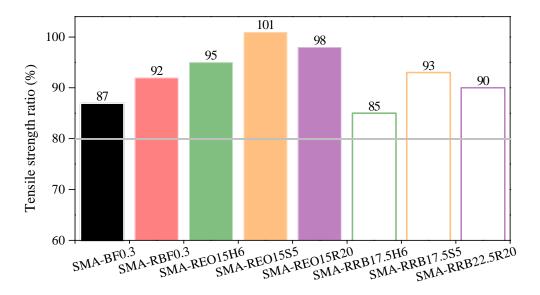


Figure 11. Tensile strength ratio values of the studied SMA mixtures

The SMA recycled asphalt mixtures produced with the bitumens modified with EO and polymers, namely SMA-REO15H6, SMA-REO15S5 and SMA-REO15R20, present the higher TSR values, respectively 95%, 101% and 98%. These values are also higher than the TSR value of the control recycled SMA mixture SMA-RBF0.3 (92%) and the control SMA mixture SMA-BF0.3 (87%).

On the other hand, the recycled SMA mixtures produced with modified bitumens with RB and polymers show slightly lower TSR values than the control recycled SMA mixture (with the exception of the recycled mixture SMA-RRB17.5S5). Thus, the addition of higher amounts of RB slightly reduces the resistance to water when compared with the addition of EO, but without compromising the limit specified for this mechanical property.

3.4.3. Stiffness modulus

From the analysis of the stiffness modulus and phase angle master curves (Figure 11), obtained for a reference temperature of 20 °C, it is clear that the recycled SMA mixture with CR and RB (SMA-RRB22.5R20) is less influenced by temperature variation (lower slope of master curve), while the recycled SMA mixture with RB and HDPE (SMA-RRB17.5H6) is more influenced by temperature changes (higher slope of master curve). In the first case, the lower interaction between the CR and the blend of bitumen and RB could contribute to the CR working as an elastic filler, turning the mixture less susceptible to temperature variations. In the second case, the higher temperature susceptibility could be related to the higher RB content that reduces the stiffness modulus at high temperatures (low frequencies). So, the SMA-RRB22.5R20 mixture presents low stiffness modulus and high phase angle values at high frequencies (low temperatures), as well as high stiffness modulus and low phase angles at low frequencies (high temperatures), showing the previously mentioned effect of CR acting as a filler.

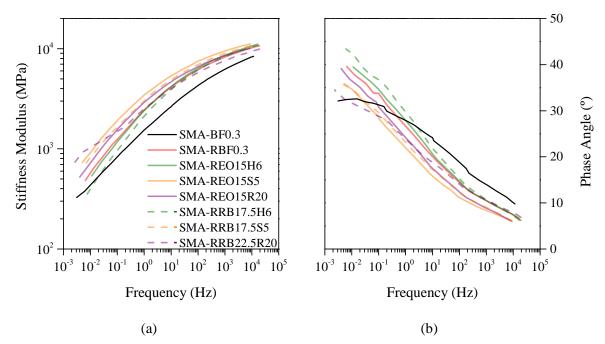


Figure 12. Stiffness modulus (a) and phase angle (b) master curves (T_{ref} =20°C) of the studied recycled SMA mixtures

At low temperatures (high frequencies), the other recycled SMA mixtures produced with modified binders present similar stiffness modulus and similar or slightly higher phase angles than the control recycled mixture (SMA-RBF0.3). At low frequencies (high temperatures), the recycled SMA mixtures with elastomers (SBS and CR) present higher stiffness modulus and lower phase angles when compared with the control recycled SMA mixture, while the recycled SMA mixtures with plastomers (HDPE) have similar or lower stiffness modulus and higher phase angles than control recycled mixture. In other words, in these recycled mixtures, the elastomers ensure lower temperature susceptibility than the plastomers. Nevertheless, all recycled SMA mixtures exhibit stiffness moduli higher than those of the SMA mixture produced with B35/50 and fibres. The aged bitumen present in RAP could contribute to the high stiffness moduli of these mixtures, although the penetration values of the corresponding recycled binders are higher than the penetration value of the conventional B35/50 bitumen.

Therefore, all the recycled SMA mixtures could be valid solutions for areas with mild weather conditions. On the other hand, for areas with hot weather, the recycled SMA mixtures produced with elastomers (with the exception of the SMA-RRB22.5R20, which showed a lower stiffness) are the best solutions.

3.4.4. Fatigue cracking resistance

The recycled SMA mixtures' durability should be associated to a higher resistance to fatigue cracking. As can be observed in Figure 12, the recycled SMA mixture with EO and HDPE (SMA-REO15H6) present the highest fatigue cracking resistance, followed by the recycled SMA mixture with EO and SBS (SMA-REO15S5), even though the fatigue cracking performance is similar. The high fatigue cracking resistance of the recycled SMA mixture with SBS could be related with elastic properties of the SBS polymer, while in the recycled SMA mixture with HDPE, it could have resulted from the interaction between the modified bitumen and the aged bitumen, contributing to a higher flexibility and a better fatigue cracking performance.

It must also be noted that the recycled SMA mixture with the worst fatigue cracking performance is the recycled mixture with RB and CR (SMA.RRB22.5R20), which could be related with the low interaction between the blend bitumen/RB and crumb rubber. The CR could have worked as a filler that absorbed part of bitumen needed for binding the components of mixture, decreasing the fatigue cracking resistance. Thus, the effective binder content available in the recycled SMA mixtures with CR is lower than that available in the other studied mixtures.

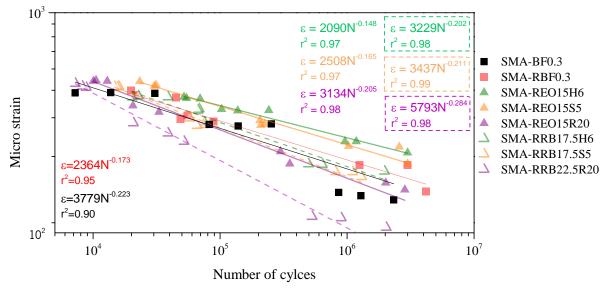


Figure 13. Fatigue test results of the studied SMA recycled asphalt mixtures

The other recycled SMA mixtures and the SMA mixture with B35/50 and fibres present a similar or slightly higher fatigue cracking performance when compared with the control recycled SMA mixture (SMA-RBF0.3) at high strain levels, but this behaviour is reversed for low strain levels (more usual in road paving). Therefore, it is essential to analyse the advantages and disadvantages of using these recycled SMA mixtures produced with bitumens modified with waste materials in order to guarantee an adequate fatigue cracking performance.

When comparing the recycled SMA mixtures produced with EO with the recycled SMA mixtures produced with RB, the recycled mixtures with EO show the best fatigue cracking resistance for the same kind of polymer. In this case, despite the addition of higher contents of RB, its effect on the fatigue cracking resistance is not as positive as that of EO.

3.4.5. Permanent deformation resistance

The permanent deformation resistance was evaluated through wheel tracking test which assesses the mechanical performance of an asphalt mixture at high service temperatures (60°C). Analysing the permanent deformation results, it can be observed that the recycled SMA mixtures with modified bitumens present a more promising performance than the control mixtures (SMA-RBF0.3 and SMA-BF0.3), except the recycled mixture with EO and HDPE (SMA-REO15H6) that show the lower permanent deformation resistance (Figure 13). Moreover, both recycled mixtures with CR (SMA-REO15R20 and SMA-RRB22.5R20, respectively) exhibit the best permanent deformation performances. These results confirm the importance of considering both fatigue cracking and permanent deformation performance to evaluate the advantages of each recycled mixtures for each particular case of application. When compared to the recycled mixtures with EO, the recycled mixtures with RB often display a higher permanent deformation resistance, despite the higher content of bitumen substitute used. Nonetheless, the recycled mixtures with CR present an equivalent behaviour when using either EO or RB.

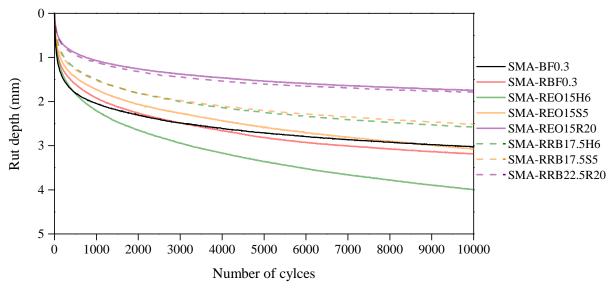


Figure 14. Permanent deformation test results of the studied recycled SMA mixtures

Subsequently, wheel tracking slope (WTS $_{AIR}$), proportional maximum rut depth (PRD $_{AIR}$) and maximum rut depth (RD $_{AIR}$) parameters specified in the EN 12697-22 standard were calculated and are presented in Table 3.

As mentioned above, the recycled mixture with CR and EO shows the best permanent deformation performance, since it presents the lowest WTS_{AIR} (0.04 mm/ 10^3 cycles) e PRD_{AIR} (4.34%) values, followed by the recycled SMA-RRB22.5R20 mixture with WTS_{AIR} and PRD_{AIR} values of, respectively, 0.04 mm/ 10^3 cycles and 4.36%. This excellent behaviour of both mixtures could be related to the significant amount of CR added (20%), in comparison to the recycled mixtures with SBS (5%) and HDPE (6%).

Table 4. Permanent deformation parameters of the studied SMA mixtures

Mixtures	WTS_{AIR}	$\mathrm{PRD}_{\mathrm{AIR}}$	RD_{AIR}
Mixtures	(mm/10 ³ cycles)	(%)	(mm)
SMA-BF0.3	0.06	7.38	3.03
SMA-RBF0.3	0.07	7.81	3.19
SMA-REO15H6	0.13	9.63	4.00
SMA-REO15S5	0.10	7.66	3.08
SMA-REO15R20	0.04	4.34	1.75
SMA-RRB17.5H6	0.07	6.26	2.58

SMA-RRB17.5S5	0.06	5.98	2.51
SMA-RRB20R20	0.04	4.36	1.79

Both control mixtures, recycled SMA mixture (SMA-RBF0.3) and SMA mixture without RAP material (SMA-BF0.3), as well as, the recycled mixtures with RB and HDPE or SBS present similar permanent deformation parameters (WTS_{AIR} values vary between 0.06 e 0.07 mm/10³ cycles). Although the recycled mixture SMA-REO15H6 present the less promising permanent deformation performance, the parameters WTS_{AIR} (0.13 mm/10³ cycles) e PRD_{AIR} (9.63%) are similar to those of mixtures commonly used in paving works.

The permanent deformation results obtained in this work showed that the addition of RB to recycled SMA mixtures lead to interesting permanent deformation resistances. The higher content of RB used as partial substitute of bitumen in these recycled mixtures does not compromise the permanent deformation performance, which is greater than that of the recycled mixtures with EO.

4. Conclusions

The main goal of this study was the development of recycled SMA mixtures with high rates of waste materials, such as, waste engine oil products (waste engine oil and recycled engine oil bottoms), polymers (waste HDPE, waste CR and SBS) and RAP material. High penetration bitumens modified with waste materials were used to produce recycled SMA mixtures with 50% of RAP material, which present, in general, a better mechanical performance than a conventional SMA mixture and a recycled SMA mixture produced with a straight run bitumen. The main conclusions of this study are outlined, as follows:

The characterization of the recycled binders with waste materials showed that, when aged bitumen is added, high contents of EO or RB could be used in the bitumens modified with waste materials to obtain the established penetration values. Dynamic viscosity and softening point temperatures of those recycled binders were generally higher when RB was used instead of EO;

- In rheological terms, the recycled binders incorporating waste materials were less susceptible to temperature variations, since they presented higher complex modulus at high temperatures and, lower complex modulus at low temperatures, when compared with the control recycled RB160/220 binder and the conventional B35/50 bitumen;
- Recycled binders incorporating waste materials generally present higher values of high temperature

 PG grade and also lower values of non-recoverable creep compliance than the control recycled binder

 and the conventional B35/50 bitumen, which could indicate an increased permanent deformation

 resistance;
- Recycled SMA mixtures produced with waste materials fulfilled the maximum recommended binder drainage value (0.2%), which means that they do not present any binder drainage problem, although high contents of bitumen substitutes were used;
- Regarding their mechanical properties, these recycled mixtures produced with bitumens modified with waste materials showed an excellent water sensitivity performance (TSR values higher than 80%), especially the recycled mixtures with bitumens modified with EO;

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- The recycled SMA mixtures produced with SBS and CR presented higher stiffness modulus and lower phase angles at high temperatures (low frequencies), which was not so evident for the recycled mixtures produced with HDPE;
- In terms of fatigue cracking performance, the recycled SMA mixtures produced with EO and HDPE or SBS (SMA-REO15H6 and SMA-REO15S5) showed the best fatigue cracking resistance, while the recycled SMA mixtures produced with CR exhibited the worst behaviour. The use of EO had a positive influence in the fatigue cracking resistance of recycled SMA mixtures;
 - Although all recycled mixtures have presented an interesting permanent deformation performance, the SMA-REO15H6 recycled mixture showed the worst performance, while the SMA-RRB22.5R20 recycled mixture had the best one. The permanent deformation resistance of these mixtures improved when RB was used instead of EO;

- The use of high rates of waste materials (RAP, EO, RB, waste HDPE and CR) contribute to obtain recycled SMA mixtures with enhanced mechanical performance, in comparison to conventional
- SMA mixtures, which can be seen as promising solutions at the environmental and economic levels.
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