Pushing the Asphalt Recycling Technology to the Limit

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Abstract: The environmental and economic benefits of using Reclaimed Asphalt Pavement (RAP) material in hot mix asphalt (HMA) applications could be pushed up to the limit, by producing totally recycled HMAs (100% RAP), but the performance of this alternative must be satisfactory. In the present study, the utilization of a used motor oil as a rejuvenator was evaluated. This would allow the aged binder to restore some of its original properties, thus promoting an adequate performance of the mixture. After studying the RAP moisture content, the optimal amount of oil was determined by conventional bitumen tests, using the penetration grade as the selection criterion. Then, the binder was evaluated through rheological testing, and laboratory specimens were prepared and tested for water sensitivity, permanent deformation, stiffness and fatigue, in order to confirm that the totally recycled mixture will perform as good as a conventional mixture used for comparison purposes.

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Introduction

In order to contribute towards a sustainable development, Road Administrations are beginning to adopt alternative materials to be applied on their infrastructures. Furthermore, the rehabilitation of the existing pavements is also being more often considered in a closed cycle, where the used material (usually know as Reclaimed Asphalt Pavement, RAP) is seen as a product for the new layers, rather than a waste material [1-3].

Recycling of asphalt mixtures is increasing due to higher costs of bitumen, scarcity of quality aggregates and environmental issues related to the disposal of aged asphalt mixtures [4-8]. Several studies have been carried out in the past [9-11] with high content of recycled asphalt mixtures (up to 60%), which is mainly limited by each country's material specifications and by practical issues related to the production of the mixtures in the asphalt plant.

In Portugal, the production of hot recycled mixtures with high RAP content in asphalt plants is not yet a reality, partially due to the National Asphalt Specifications [12], which do not encourage the use of mixtures with more than 50% RAP (in binder or base courses) and 10% RAP (in surface courses). In contrast, the study of recycled mixtures with high RAP content is already a reality in other countries which have begun to produce mixtures with 100% RAP [13-15], showing that it is possible to successfully introduce 100% recycled HMA mixtures in the paving industry. However, in order to assure that this type of mixture can be seen by the Road Administrations as a true alternative, its performance must be as good as that of conventional mixtures. In fact, these mixtures may

present a lower performance due to the loss of the lighter fractions of the aged binder. Taking that into account, a rejuvenator is normally used in order to improve the mixture properties.

Rejuvenation of bitumen is simple in principle, consisting on the replacement of the oils lost during the aging process, and on the rebalancing of the bitumen composition. However, this is not generally possible, as it would require sophisticated extraction, testing and remodelling of the binder in the road pavement [16]. Although some commercial rejuvenators or warm mix asphalt (WMA) technologies have been used by other authors in previous asphalt recycling works [17-20], the rejuvenator selected for this study was a used motor oil because a similar material has already been used as binder modifier [21-23] or rejuvenator [24, 25] in previous studies. In addition, it would allow the recycling of asphalt mixtures with exclusive use of reclaimed materials, which is the main objective of this study. Thus, this paper tries to push up to the limit the use of Reclaimed Asphalt Pavement (RAP) material, focusing on hot mix asphalt (HMA) recycling applications.

Material Characterisation

RAP Characteristics

The RAP material is the main constituent of the new recycled asphalt mixtures that were produced in this work. The characteristics of this material must be in agreement with National and European specifications (EN 13108-8), namely for the following properties:

- Amount of coarse foreign matter in RAP (EN 12697-42);
- Particle size distribution of RAP (EN 933-1);
- Moisture content of RAP in a ventilated oven (EN 1097-5);
- Binder content of RAP by ignition (EN 12697-39);
- Characteristics of the recovered binder (EN 12697-3 and then EN 1426 and EN 1427 for binder characterisation).

One of the characteristics of RAP material is its heterogeneity. Thus, the RAP material used in the laboratory was reduced by

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Fig. 1. RAP and its Aggregates Grading Curves Outside AC 14 Surf Envelope.

quartering, according to EN 932-1 and EN 932-2 standards, in order to obtain samples that correctly represent the studied RAP.

Amount of Coarse Foreign Matter in RAP

The studied RAP was originally obtained by milling a distressed surface course of a main national highway, and this material was stored in the open air at a construction company yard before being collected for this study. The presence of coarse foreign matter in the RAP was quantified according to EN 12697-42 standard, by making a visual inspection of a sample. It was concluded that foreign matter was absent in the examined sample of RAP.

Particle Size Distribution of RAP

The particle size distribution of the RAP material was evaluated according to the EN 933-1 standard. The RAP material was also incinerated, according to the EN 12697-39 standard, in order to burn the bitumen and to evaluate the particle size distribution of the aggregates constituting the RAP (EN 12697-2), so as to determine whether it fits within the grading envelope of a conventional asphalt concrete surface course mixture (AC14 surf), as illustrated in Fig. 1.

The RAP aggregates have an excess of fines and a low percentage of coarse material. This is due to the milling process of the surface layer from which the RAP was extracted. An excessive amount of fines may cause permanent deformation problems, although this is not expected in the present study because the RAP binder is very hard, increasing the rutting resistance. Thus, it is important to use performance based tests to verify if the mixture will behave adequately even if it does not fully meet the aggregates grading specifications.

The results of the particle size distribution of RAP should also be used to classify or identify the RAP material, according to EN 13108-8 standard. This material was classified as 31.5 RA 0/22.5, where 31.5 is the maximum dimension of the RAP material, in mm, RA is the symbol for "reclaimed asphalt", and 0/22.5 are the minimum and maximum dimensions of the aggregates present in the RAP, in mm.

Moisture Content of RAP in a Ventilated Oven

Besides the objective of quantifying the RAP moisture content, this section also evaluates the effect of the water in the RAP material. In fact, it is known that a key factor in the success of RAP applications is related to its storage conditions, namely the control of the moisture, dust and other pollutants.

In Portugal, the storage of this type of material (RAP) is still not generally carried out in an appropriate manner, i.e., local contractors do not have adequate storage facilities. However, the lack of investment in this type of recycled mixtures leads to inefficient knowledge implementation in this field [26], with most of the RAP stocks being kept uncovered, which compromises the performance of the recycled mixtures.

In this section, the time needed to attain the loss of moisture from the RAP was determined up to the achievement of the mass equilibrium, at different temperatures. This was made in order to guarantee that the RAP is free from moisture without compromising the performance of the final mixtures. By studying different production temperatures (RAP heating) it is intended to select the minimum temperature (in order to limit the ageing of the binder in the RAP) that allows a significant reduction of the time necessary for the RAP to dry.

One of the criteria that need to be met in order to use a specific RAP material in the production of asphalt mixtures is that its moisture content should not exceed approximately 5% (EN 13108-8). Taking the additional objectives of this study into account, the moisture content was determined according to the EN 1097-5 standard, which recommends the use of a ventilated oven. Thus, in addition to the standard test, the (water) mass loss from the RAP sample was measured with a monitoring equipment, including



Fig. 2. Results of the Moisture Content Monitoring at Different Conditioning Temperatures (Left Side) Obtained in the Water Loss Monitoring System (Right Side).

temperature probes and a balance, which allowed the continuum data acquisition regarding the sample mass, as illustrated in Fig. 2.

In the mentioned tests, the temperature and the mass of the sample were measured continuously, which allowed to calculate the moisture content loss by evaporation during the heating of the RAP samples. The temperatures used in this particular study were 110, 125 and 150 °C and the results obtained are presented in Fig. 2.

The results obtained in the different tests, carried out at different temperatures, show that the moisture content of the various samples analysed is generally identical (between 3.1 and 3.2%), which indicates that the material used in the present study comprises the same moisture characteristics. Furthermore, it is possible to observe that the values are within the specification limits for RAP application.

According to the results shown in Fig. 2, it is possible to conclude that for the temperatures of 110 and 125°C, the moisture loss only stabilised after a period of approximately 5 hours, while for the temperature of 150°C that occurred for a smaller period (approximately 3.5 hours, corresponding to a reduction of 30% on the drying period), even though a static drying procedure was used. A similar reduction would be expected for a plant production situation, where the RAP would be dynamically exposed to temperature and air flow, increasing the drying capability. Thus, a production temperature of 150°C should be selected to increase the productivity of the asphalt plant, assuring an adequate drying of the RAP material, in order to reduce the negative influence of the moisture content in the final properties of the recycled mixture.

These tests show, in practical terms, how important the production time and temperature in the asphalt plant can be to obtain recycled mixtures with adequate in service performance. In fact, if the RAP material is not totally dry, some significant performance problems may appear as a result of the use of 100% RAP in the final mixtures.

Binder Content of RAP by Ignition

There are several methods for determining the binder content of asphalt mixtures (or RAP). In the present study the ignition methodology (EN 12697-39) was selected, since it does not demand the use of solvents.

In order to consider the material variability in the determination of the binder content, three samples were used to characterise the studied RAP. As can be observed in Fig. 3, it was decided to monitor the weight loss during the test, which would allow establishing the minimum time that is necessary to obtain the final results.

Based on the results presented in Fig. 3, it can be concluded that the ignition time is approximately 45 minutes for the three samples tested. However, these results depend on the amount of material that is placed in the oven, on the sample characteristics and may also be different for other similar pieces of equipment used.

The average binder content attained in the present study (5.3%) is within the normal range of values used for conventional surface course mixtures, as that from where the RAP was obtained. It was also observed that the different samples analysed did not show significant oscillations. Thus, the RAP homogeneity necessary to assure that the results obtained throughout the study are comparable



Fig. 3. Percentage of Mass Loss in the RAP Samples During the Ignition Test.

could be confirmed.

Characteristics of the Recovered Aged Binder

The characterization of the binder of the recycled mixture is a very important information to assess the expected performance of the mixture [27]. In order to characterize the aged binder present in the RAP, it was separated from the aggregates sample by dissolving it in toluene and, after removing all solid particles from the bitumen solution, using filter and tube centrifuge, the bitumen was recovered by vacuum distillation using a rotary evaporator, following the procedures specified in the EN 12697-3 standard.

Later, the recovered bitumen was characterized through penetration (EN 1426) and softening point (EN 1427) tests, in order to obtain its basic properties. The results revealed a very hard bitumen with low penetration value (10 dmm) and high softening point (73°C), with a great consistency between the different samples tested.

Dynamic viscosity tests (EN 13302) are not mandatory to study aged binders recovered from RAP, but they were also performed at a range of high temperatures (120 - 180°C), in order to study the mixing/compaction properties (Table 1) of the recovered binder, using a rotating spindle apparatus, according to a predefined procedure [28].

These results confirm that it is not viable to produce recycled mixtures at temperatures below 150°C, due to the high viscosity of the binder at those temperatures. The viscosity of the binder should not be higher than 0.5 Pa.s if an adequate coating of the aggregate is to be obtained [29]. Taking into consideration that the RAP aggregates are already coated with bitumen as well as the previous results observed during the moisture content evaluation, it was confirmed that a mixing temperature of 150°C would result in a satisfactory workability of the mixture and, therefore, that temperature should be used to continue the study. This also prevents the RAP binder from suffering additional ageing at higher temperatures.

Study of Binder Rejuvenation

Selection of Rejuvenator Content Based on Basic Characterization of Binders

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Table 1. Dynamic viseosity of the Recovered Dinder and of the Dinder Samples Trepared with Different Amounts of Rejuvenator.								
Test Temperature (°C)		120	130	140	150	160	170	180
Dynamic Viscosity (Pa.s)	RAP recovered Binder	6.41	2.98	1.50	0.83	0.48	0.30	0.18
	10/20 Bitumen	6.45	3.16	1.69	0.96	0.60	0.40	0.28
	Oil	0.007	0.006	0.005	0.004	0.004	0.003	0.003
	10/20 + 3% Oil	4.85	2.39	1.29	0.76	0.46	0.30	0.20
	10/20 + 6% Oil	3.19	1.65	0.93	0.56	0.36	0.24	0.16
	10/20 + 12% Oil	1.98	1.06	0.63	0.39	0.24	0.16	0.11

Table 1. Dynamic Viscosity of the Recovered Binder and of the Binder Samples Prepared with Different Amounts of Rejuvenator.



Fig. 4. Penetration and Softening Point (R&B Temperature) Test Results of Bitumen with Different Percentages of Rejuvenator.

In the present study, RAP material was used as the main component of the mixture. However, since the binder usually present in this type of material is too hard for a conventional bituminous mixture, an aliphatic-aromatic used motor oil was also used to rejuvenate the binder and improve its properties. This material is very difficult to characterize, but for the scope of the present paper, a dynamic viscosity characterization was carried out in order to understand its influence on the softening of the recovered binder, the results of which are presented in Table 1. In order to determine the amount of additive that should be added to the mixture (to improve its properties), a new binder (10/20 pen grade), with properties similar to those of the RAP binder (penetration of 13 dmm and softening point of 68°C) was used in this particular part of the study, as it would be impracticable to extract the necessary amount of aged binder from the RAP to run the series of tests.

The rejuvenation study included the addition of three rejuvenator percentages (3, 6 and 12%, by mass of binder) to samples of the previously mentioned 10/20 pen bitumen, which were then characterized through penetration (EN 1426), softening point (EN 1427) and dynamic viscosity (EN 13302) tests. The latter were carried out at various temperatures, as described previously.

The optimum amount of additive that should be used was determined by means of conventional bitumen tests. The criterion used was the obtainment of a bitumen of a higher penetration grade (in this case, the bitumen should reach a 20/30 pen grade, since it was previously classified as a 10/20 pen bitumen). The penetration and ring and ball test results are shown in Fig. 4.

The minimum amount of rejuvenator necessary to modify the aged binder and to achieve a 20/30 penetration grade (corresponding to the shaded areas, as specified in EN 12591) was 5%. This was the additive content used in the remainder of the study.

The recycled binder viscosity is normally achieved by means of mixing the aged binder with different proportions of rejuvenator until the desired viscosity is obtained [30]. The dynamic viscosity test results of the 10/20 pen bitumen and binder samples with different amounts of oil rejuvenator, determined at different temperatures, are shown in Table 1.

As can be seen in Table 1, the rejuvenator has a significant effect on the reduction of viscosity at high temperatures, typically used during the production/compaction stages, which is proportional to the amount of additive added to the 10/20 pen grade bitumen. By observing the viscosity reduction after adding the rejuvenators, it could be confirmed that the quantity of additive previously selected to continue the study (5%) allows reducing the mixing temperature of the recycled HMA mixture by approximately 10 to 15°C in comparison with the same mixture without rejuvenators.

Rheological Comparison between Aged and Rejuvenated Binders

After selecting the rejuvenator content to be used with the RAP in order to produce the asphalt recycled mixtures, the mechanical properties of the recovered aged binder were evaluated, by means of rheological tests, before and after its rejuvenation with 5% of oil.

The rheological properties were assessed using a stress controlled rotational rheometer (Stresstech-HR, Reologica) equipped with 40 mm parallel plates and a gap of 1 mm. Small amplitude oscillatory shear (SAOS) frequency sweep tests were performed at various temperatures (30, 40, 50, 60, 70, and 80 °C), in the linear range (confirmed by using two different strain levels) for each temperature and sample, in order to obtain the mechanical spectra of each sample at the corresponding temperatures. Prior to the record of the mechanical spectra, both thermal and structural equilibrium conditions were checked. The experimental procedures for the rheological characterization of the bitumens with the dynamic shear rheometer (DSR) were based on EN 14770 standard.



Fig. 5. Master Curves (TREF = 30 °C) of Complex Modulus (G^*), tan(δ), Elastic Modulus (G') and Viscous Modulus (G'') Obtained in the DSR for Aged and Rejuvenated Binders.

The procedure of superposing curves at different times (frequencies) and temperatures is known as the time-temperature superposition (TTS) and the resulting curves (master curves) cover large time or frequency domains well beyond the frequency range experimentally accessed by the rheometer.

The master curves of the recovered aged bitumen before and after rejuvenation (Fig. 5) were constructed using the IRIS software – Innovative Rheological Interface Software (version 9.0, copyright 1990-2005) of Rheo-Hub Innovative Rheology Information Systems, IRIS development LLC.

The mechanical spectra (G^* , $\tan(\delta)$, G' and G'') of both binders obtained in the DSR test, at temperatures between 30-80 °C and adjusted for a reference temperature of 30°C, are different over the whole range of frequencies, thus confirming the significant influence of the rejuvenator in the mechanical behaviour of the binder at the tested temperatures. It can be observed that the complex modulus of the rejuvenated binder is nearly 10 times lower than that of the aged bitumen almost for all frequencies and temperatures, especially due to the reduction of the elastic modulus, although the viscous modulus has also decreased. Thus, the values of $\tan(\delta)$ of the rejuvenated binder increase for all frequencies due to the higher decrease of its elastic modulus.

A characteristic relaxation time corresponding to the inverse of the frequency where $tan(\delta) = 1$ [31] can be extracted from the master curve for both bitumens. The relaxation time of the bitumen decreases (corresponding to a higher frequency for G' = G'') after the addition of the rejuvenator, since it is known that the relaxation time is proportional to the bitumen viscosity and the cube of the size of the asphaltenes/resins micelles. This means that the rejuvenator effectively reduced the size of the micelles of the binder, thus causing a change in the behaviour of the binder at higher frequencies.

Performance of Studied Mixtures

The characteristics of the RAP material does not allow to fulfill the mixtures specifications defined by the Road Administrations, since it contains an excessive amount of fines. Since a conventional HMA mixture cannot have a composition similar to the 100% RAP mixture, a different approach had to be used. Therefore, the performance of the 100% RAP mixture was compared to that of a conventional HMA mixture (necessarily with different composition) so as to validate the possibility of direct substitution of such mixtures by 100% recycled materials.

Production of the Studied Mixtures

The studied mixtures were produced in a laboratory mixer, being compacted into slabs with a roller compactor according to the EN 12697-33. The slabs were then cut into prismatic and cylindrical specimens with dimensions specified in the European standards for each test to be carried out.

The conventional HMA mixture was produced with a 35/50 penetration grade bitumen (the main type of asphalt binder presently used in Portugal) and its mix design was carried out using the Marshall methodology. By using a grading curve for the conventional HMA mixture within the envelope presented in Fig. 1 (AC14 surf), an optimum binder content of 5.1% was obtained. The mixing temperature used (165°C) was specified by EN 12697-35 according to the type of binder used.

The 100% recycled mixture was produced in accordance with the conclusions obtained in Section 2, namely using 5% of OIL and a mixing temperature of 150°C.

In order to confirm that the 100% recycled mixture will perform as good as a conventional HMA mixture, water sensitivity, permanent deformation, stiffness modulus and fatigue tests were performed on both mixtures and the results are presented below.

Water Sensitivity

The evaluation of the water sensitivity is essential when studying asphalt recycled mixtures, since this property is directly related to the performance and durability of these materials during the road pavement life. The evaluation of this property is determined in Europe by the EN 12697-12 standard. According to this standard, two groups of three specimens are tested for the indirect tensile strength (ITS) after a specific period, in different conditions. In that period, one group is kept dry and the other is immersed in water, in order to determine the influence of the water on the weakening of the bond between aggregates and binder and, consequently, on the strength of the mixture.

Following the determination of the ITS of each specimen, it is possible to calculate the average value of each group and the indirect tensile strength ratio (ITSR), which corresponds to the ratio between the ITS of the wet group (ITSW) and the dry group (ITSd) of specimens. In the present study, the indirect tensile test was carried out according to the EN 12697-23 standard, after a



Fig. 6. Results Water Sensitivity Tests (ITSR vs. Air Voids Content).



Fig.7. Wheel Tracking Tests Results of Both Mixtures Carried Out in Air, at 50 °C.



Fig. 8. Stiffness Modulus and Phase Angle for Different Frequencies at 20 °C.



Fig. 9. Fatigue Cracking Resistance of Both Mixtures Assessed at 20 °C.

volumetric characterization of the specimens (to determine the voids content, which significantly influences the results), as presented in Fig. 6.

Overall, it was found that both mixtures had very good water sensitivity results, although the conventional mixture (produced with a 35/50 pen grade bitumen) was slightly more sensitive to the presence of water (lower ITSR due to its higher voids content). The recycled mixture with incorporation of a rejuvenator (RAP - OIL) showed a better performance (durability), mainly due to the low volume of voids, which in turn is related to the high amount of fines previously mentioned.

Permanent Deformation

The rut resistance of asphalt mixtures may be assessed, in comparative terms, by the analysis of wheel tracking test (WTT) results, plotted in a graph deformation vs. number of cycles. Fig. 7 represents the results of the recycled mixture and a conventional mixture produced with a 35/50 pen bitumen.

The permanent deformation performance of both mixtures is similar, as can be observed from Fig. 7, although the 100% recycled mixture has shown a slightly better behaviour. One of the main parameters used to assess the rut resistance of bituminous mixtures is the wheel tracking slope in air (WTS_{air}) measured between the 5000th and the 10000th cycles. In this study, the recycled asphalt mixture presented a WTS_{air} value of 0.14 mm/10³ cycles while the conventional mixture used for comparison presented a WTS_{air} value of 0.16 mm/10³ cycles.

Stiffness Modulus

The stiffness modulus and phase angle were obtained using the four-point bending beam test, with a repetitive sinusoidal loading configuration, as specified in the EN 12697-26 standard. A frequency sweep (0.1 to 10 Hz) test was used to determine the response of the material to different loading frequencies. The tests were carried out in controlled strain, at 20°C, and the results are presented in Fig. 8.

As can be observed in Fig. 8, the recycled mixture is less susceptible to the loading frequency which is typical of mixtures with aged binders, since the stiffness modulus variation is smaller than in the case of the conventional mixture and the phase angle is significantly lower than that of the conventional mixture, for any frequency tested. These results are in accordance with those obtained in the rut resistance tests, where the recycled mixture has shown a good performance, even at a high temperature (which is equivalent to lower frequencies in the stiffness modulus tests).

Fatigue Cracking Resistance

The fatigue life equations at 20 °C of the conventional AC 14 Surf 35/50 (CONV) and the 100% recycled (RAP - OIL) mixtures are presented in Fig. 9, in order to evaluate the difference between the fatigue resistance of 100% recycled and conventional HMA mixtures.

Based on the results presented in Fig. 9, it was possible to conclude that the recycled mixture presented a better performance than the conventional mixture.

The high fatigue resistance of the 100% recycled mixture can result from its high content of fines (the quantity of mastic filling material increases), which are present in the RAP due to the milling operation of the bituminous mixture from the road pavement. This fact could also have led to rutting problems if the hardened bitumen of the recycled mixtures was not so stiff. This unexpected high fatigue resistance result has already been noted by other authors [32], who concluded that the aged binder in RAP formed a stiffer layer coating the RAP aggregate particles. This layered system helped to reduce the stress concentration within the mixtures and the aged binder mastic layer was actually serving as a cushion layer in between the hard aggregate and the soft binder mastic.

The parameters specified in EN 12697-24 (number of loading cycles obtained for 100 microstrain - N₁₀₀ and the strain level that corresponds to a fatigue failure at 1 million loading cycles - ε_6), which are normally used to evaluate the fatigue performance of bituminous mixtures, were estimated from the fatigue life equations of Fig. 9. The N₁₀₀ values obtained for RAP-OIL and CONV mixtures were, respectively, 3.8×10^8 and 1.3×10^6 , while the ε_6 values obtained for the same RAP-OIL and CONV mixtures were, respectively, 222×10^{-6} and 107×10^{-6} .

As previously mentioned, the rejuvenators increase the fatigue life of recycled mixtures due to the combined effect of the reduction on the penetration grade of the binder and the slight increase on the binder content of such mixtures. In this case, those factors associated with a higher content of fines greatly enhanced the flexibility of the recycled mixture, thus increasing its fatigue cracking resistance.

Conclusions

The main conclusions that can be drawn from this study with a mixture 100% recycled from old and distressed road pavement materials are the following:

- In order to reduce the negative influence of the moisture content in the final properties of the recycled mixture, a production temperature of 150 °C was selected, which proved to reduce the time to release all the RAP moisture by 30%, in comparison to the temperatures of 110°C and 125°C.
- The content of rejuvenator (OIL) was defined by the minimum amount that could change the grade of the aged binder from 10/20 to 20/30 pen grade, resulting in an optimum content of 5%.
- The rheological test results confirmed the influence of the rejuvenator in the modification of the binder properties, reducing the size of the asphaltenes/resins micelles.
- The improved performance of the 100% recycled mixture, in comparison to the conventional mixture can be explained by the higher stiffness of the binder, for the mixture stiffness and rutting resistance, and by the higher content of fines associated with the increased flexibility given by the rejuvenator, for the water sensitivity and fatigue resistance.
- In summary, the incorporation of 100% RAP and used motor oil (as a binder rejuvenator) in the production of asphalt mixtures can be a paving solution with a performance as good as conventional asphalt mixtures, as long as adequate storing (moisture) and production (temperature) conditions are assured.

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