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## **Influence of ageing on the properties of bitumen from asphalt mixtures with recycled concrete aggregates**

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### **Abstract**

The reuse of recycled concrete aggregates in new hot-mix asphalt can be a more sustainable method of production, but these mixtures may need a heat treatment before compaction to improve their water sensitivity performance. A direct consequence of this treatment is an increase in the hot-mix asphalt resilient modulus. The aim of this paper is to analyse the effect of ageing on the stiffness of asphalt mixtures with different amounts of recycled concrete aggregates, before and after a heat treatment, which was analysed through the assessment of its bitumen properties. Moreover, this paper also aims to analyse whether the rolling thin-film oven test is able to simulate the ageing effect of the heat treatment. In the laboratory work, a paving grade bitumen B50/70 has been used to produce asphalt mixtures with 0% and 30% recycled concrete aggregates, and the bitumen was later characterised (using penetration, softening point, dynamic viscosity and dynamic shear rheometer tests) in various situations, such as when using virgin bitumen, short-term aged bitumen, aged bitumen after heat treatment (simulated with 4 hours of rolling thin-film oven test) and bitumen samples recovered from asphalt mixtures with different production mixes (0% and 30% recycled concrete aggregate) and heat treatment conditions (0 and 4 hours of curing time in the oven). Based on the results obtained, it could be concluded that the ageing resulting from the heat treatment is the primary cause of the hot-mix asphalt's increased stiffness, while recycled concrete aggregate content has a small influence. Moreover, it could be concluded that when there is no curing time, the recycled concrete

aggregate protects the bitumen against ageing. Additionally, it could be stated that the rolling thin-film test is able to adequately simulate the ageing effect of the heat treatment. Thus, this test is useful for determining the ageing suffered by the bitumen when the recycled concrete aggregate mixture is manufactured using a heat treatment.

*Keywords: hot-mix asphalt; recycled concrete aggregates; bitumen ageing; rheology, viscosity; bitumen properties*

## **1. Introduction**

There are some critical issues in sustainable development, such as energy consumption, generation of emissions during production (Gadja and VanGeem, 2001) and waste management (Oliveira et al., 2013). Construction and demolition waste (C&DW) has great potential for recycling within the construction industry (Symonds et al., 1999). In particular, the development of new construction materials that reuse recycled concrete aggregates (RCA) from C&DW has numerous environmental benefits, such as the mitigation of natural resource depletion and the reduction of environmental effects derived from quarry extraction (Blankendaal et al., 2014).

The use of RCA as aggregate for concrete (Rodrigues et al., 2013; Guo et al., 2014; Medina et al., 2014) and unbound pavement layers (Poon and Dixon, 2006; Vegas et al., 2011) is widely accepted. Nevertheless, several authors have stated that the use of RCA as aggregate for hot-mix asphalt (HMA) leads to mixtures with a lower moisture damage resistance (Paranavithana and Mohajerani, 2006; Pérez et al., 2012; Zhu et al., 2012), which prevents its generalized use.

However, in recent years, Pasandín and Pérez (2013, 2014) have stated that the heat treatment of HMA mixtures with RCA in the oven for a curing time of at least four hours before compaction improves the water sensitivity performance of this type of mixture. The same authors observed a great increase in the stiffness of the mixture (resilient modulus of such mixtures with RCA at 0, 10 and 20°C) as a consequence of the heat treatment in the oven. Two phenomena, which can

occur simultaneously, were hypothesized to be the main causes of the notable increase in stiffness during the heat treatment of the mixtures with RCA: i) bitumen ageing due to oxidation and loss of volatile compounds at high temperatures; and ii) absorption of a great amount of bitumen by the porous mortar attached to the RCA surface.

The heat treatment affects other properties of the mixture, such as permanent deformation and fatigue. In this regard, Pasandín and Pérez (2013, 2014) concluded that mixtures made using a heat treatment with up to 30% RCA exhibit good rutting performance. Nevertheless, they also stated that permanent deformation increases with increasing RCA percentages, so mixtures with higher RCA content should be expected to be more susceptible to rutting. Moreover, Pasandín and Pérez (2014) stated that higher initial densifications of HMA were obtained for greater heat treatment lengths due to their higher air void content. Pasandín and Pérez (2013) also stated that HMA with up to 20% RCA exhibit a fatigue life similar to that of a conventional mixture. However, the fatigue life of a HMA made with 30% RCA was slightly lower than that of a conventional mixture. Thus, again, the maximum RCA percentage to be included in a HMA should be lower than 30%.

The aim of this work is to evaluate in more detail the phenomena occurring during the production and heat treatment of asphalt mixtures with RCA, which cause the stiffening of those mixtures. Another objective is to assess whether the bitumen absorption by the RCA could protect the bitumen against ageing. To conduct that analysis, an advanced characterization of the bitumen (penetration grade, softening point, dynamic viscosity and rheological properties) was performed in the following situations:

1. virgin bitumen;
2. bitumen after standard Rolling Thin-Film Oven Test;
3. bitumen after extended Rolling Thin-Film Oven Test of 4 hours of ageing time;
4. bitumen recovered from samples of HMA made with 0% RCA manufactured with 4 hours of curing time in the oven (recovery 1);

5. bitumen recovered from samples of HMA made with 30% RCA manufactured with 4 hours of curing time in the oven (recovery 2);
6. bitumen recovered from samples of HMA made with 0% RCA without curing time (recovery 3);
7. bitumen recovered from samples of HMA made with 30% RCA without curing time (recovery 4).

The comparison between the bitumen properties in the different situations was the main method used to understand the effect of ageing during production (situations 6 and 7 vs. 1) and during the heat treatment (situations 4 and 5 vs. 6 and 7), as well as the effect of using more porous RCA aggregates instead of natural aggregates (situation 5 vs. 4 and 7 vs. 6). The properties obtained in situations 2 and 3, with the rolling thin-film oven test (RTFOT) ageing test, were used to understand if this test is able to adequately simulate the ageing of the studied mixtures after production and after heat treatment.

## **2. Materials**

A commercial paving grade bitumen B50/70 has been used in this investigation, and plays a leading role in the results of this paper. The evolution of the properties of this bitumen was assessed in different phases: before its use, after short-term ageing and after four hours of ageing in the RTFOT (simulating the expected ageing caused by the heat treatment). This bitumen has also been used to produce asphalt mixtures with 0% and 30% of RCA, using two conditioning procedures (0 and 4 hours of heat treatment after production). The bitumen used in these mixtures was then recovered and characterized.

Two types of aggregates have been used to produce HMA samples: natural and RCA. A hornfels rock, typically applied in paved road construction in Spain, has been used as natural aggregate. The RCA came from C&DW from residential buildings and was supplied by a

Spanish recycling site. The RCA is mainly composed of petrous materials (89.3%) and other materials such as asphalt materials (6.5%), ceramics (3.6%) and impurities (0.6%). It must be noted that RCA aggregates mainly differ from natural ones because they have old mortar attached to their surface. That mortar is a porous material that reduces the RCA aggregates' resistance to fragmentation (Sánchez de Juan and Alaejos Gutiérrez, 2009) and greatly increases their absorption of water (Paranavithana and Mohajerani, 2006; Pérez et al., 2012) and bitumen (Lee et al., 2012; Pasandín and Pérez, 2013). Finally, it should be mentioned that Grey Portland cement (CEM II/B-M (V-L) 32.5 N) was used as filler during the production of asphalt mixtures.

### **3. Experimental Methods**

The main tests performed as part of the laboratory investigation are described below.

#### **3.1. Marshall mix design method**

The asphalt mixture AC 22 base B50/70 G was designed following the Marshall procedure according to NLT-159/86 (MOPT, 2002). In this test, four series of five cylindrical Marshall samples were analysed to obtain the optimum bitumen content (OBC). The Marshall series were manufactured with bitumen contents ranging from 3.5% to 5.0%. The cylindrical samples were compacted with 75 blows per face using the Marshall hammer. Four series of this mixture were independently studied using this method: with or without RCA and with or without heat treatment (corresponding to the four mixtures used to recover the bitumen for situations 4, 5, 6 and 7 presented previously). The results were obtained by taking the average values of the five samples of each series.

#### **3.2. Bitumen recovery**

UNE-EN 12697-1 (AENOR, 2013a) and UNE-EN 12697-3 (AENOR, 2013b) were followed to recover the bitumen from the HMA samples. As shown in Figure 1, the UNE-EN 12697-1 indicates that the loose (uncompacted) bituminous mixture (Figure 1a) must first be introduced

into an asphalt centrifuge extractor with 1 L of toluene, as shown in Figure 1b. After five centrifugation cycles, 500 mL of clean toluene must be added and the loose mixture must be subjected to five new centrifugation cycles. As a result of this first step, the bitumen, the fines and the solvent (toluene) were separated from the rest of the aggregates. A Rotofix 32 centrifuge was then used to separate the fines from the bitumen and the solvent (Figure 1c). In this second step, the cups with bitumen, toluene and fines rotate at 3000 rpm for 10 minutes and the sediments remained in the bottom of the cups. Finally, in a third step, UNE-EN 12697-3 indicates that a rotary evaporator (Büchi Rotavapor R-205) ensures the evaporation and condensation of the toluene and its separation from the bitumen. The rotating distillation glass balloon of the rotary evaporator device is partially immersed in a heating oil bath while the solution of bitumen and toluene is subjected to a vacuum with controlled pressure (Figure 1d). As a result, bitumen and toluene are totally separated. Toluene is a highly polluting organic solvent that must be used only by qualified personnel using adequate personal protective equipment. After the test, the clean toluene must be carefully stored to be reused in other tests.

Bitumen was recovered from four different situations: HMA Marshall samples with optimum bitumen content made with 4 hours of curing time in the oven, with 0% (recovery 1) and 30% (recovery 2) of RCA, and HMA Marshall samples with optimum bitumen content manufactured with no curing time in the oven (control mixture), with 0% (recovery 3) and 30% (recovery 4) of RCA.

### **3.3. Rolling Thin-Film Oven Test**

The UNE-EN 12607-1 (AENOR, 2007c) standard has been followed to perform the RTFOT ageing procedure. This procedure simulates the short-term ageing (Jamshidi et al., 2012) that occurs during the mixing process (AENOR, 2007c) and pavement construction phase (Michalica et al., 2008). In this test (Figure 2), eight glass bitumen containers, each with  $35 \pm 0.5$  g of bitumen, were introduced into an oven with a rolling carriage at the speed of  $15 \pm 0.2$  rpm for 75 minutes. The test temperature was  $163 \pm 0.5$  °C. During the test, the samples

were subjected to an air flow of 4 L/min. Two bottles were used to determine the mass loss, while the other six were used to prepare the samples for the bitumen characterization tests. In addition to the standard RTFOT procedure, a modified RTFOT procedure extended to 240 minutes was also conducted to simulate the additional ageing caused by the heat treatment applied to the studied mixtures.

### **3.4. Bitumen basic characterization**

Two tests were carried out to analyse the basic properties of bitumen, in accordance with the EN 12591 (CEN, 1999) standard. In this regard, penetration testing according to UNE-EN 1426 (AENOR, 2007a) and ring and ball softening point testing according to UNE-EN 1427 (AENOR, 2007b) were conducted on the seven bitumen samples: B50/70, B50/70 after standard RTFOT, B50/70 after 4 hours RTFOT and the four samples obtained after bitumen recovery.

The penetration test consists of measuring the penetration, in tenths of millimetres, of a  $100\pm 0.10$  g standard needle which penetrates a bitumen sample for 5 seconds at the temperature of 25 °C. At least 3 repetitions must be carried out on each bitumen sample, with a 10 mm separation from the limits of the container and between the samples.

In the ring and ball softening point test, as the temperature rises, the bitumen becomes softer and less viscous. In this test, two horizontal discs of bituminous binder, cast in brass shouldered rings, are heated at a controlled rate of 5 °C per minute in a liquid bath while each supports a steel ball of  $3.5\pm 0.05$  g. The softening point is reported as the mean of the temperatures at which the two discs soften enough to allow each ball, enveloped in bituminous binder, to fall a distance of  $25.0\pm 0.4$  mm.

### **3.5. Bitumen rheological characterization**

A Dynamic Shear Rheometer (DSR) test (Figure 3) was conducted according to UNE-EN 14770 (AENOR, 2012) to determine the shear complex modulus ( $G^*$ ) and the phase angle ( $\delta$ ) of

the different samples of bitumen under different testing conditions. During the test, different levels of a known oscillatory shear strain are applied to the test specimen and the resulting shear stresses are measured. The adopted procedure must ensure that the test specimens are tested in the linear region over each temperature and frequency chosen (10 rad/s). To remain in the linear range, the value of  $G^*$  must not differ by more than 5% of its value over the strain levels chosen. Different configurations were used according to the testing temperatures. A plate with a 25 mm diameter and a 1 mm gap was used to conduct the test at the temperatures of 46, 52, 58, 64, 70, 76, 82 and 88 °C. At the temperatures of 15, 21, 27, 33 and 39°C, the DSR test was carried out using an 8 mm diameter plate and a 2 mm gap. Rheological characterization was also conducted for the seven situations mentioned above.

### **3.6. Bitumen dynamic viscosity**

A dynamic viscosity test (Figure 4) was conducted according to UNE-EN 13302 (AENOR, 2010) at the temperatures of 100, 110, 120, 130, 140, 150, 160, 170 and 180°C, according to a procedure presented by Silva et al. (2009), using a Brookfield viscometer and a Thermosel temperature control system. In this test, a torque is applied to a spindle rotating in a special sample container containing the bitumen sample. The relative resistance of the spindle to rotation is determined, which provides a measure of the dynamic viscosity of the sample. Bitumen dynamic viscosity tests were also performed for the seven situations mentioned above.

## **4. Results and Discussion**

The main results obtained from the laboratory investigation are described and discussed below.

### **4.1. Marshall mix design method**

The OBC and the volumetric properties of the mixtures, namely the air voids ( $V_m$ ) and voids in mineral aggregates (VMA), can be seen in Table 1.



As expected, mixtures manufactured using a curing time of 4 hours in the oven present higher OBC than the mixtures made without curing time in the oven. The higher bitumen absorption that takes place during the time that the mixture remains in the oven is mainly responsible for this performance (Pasandín and Pérez, 2013). Moreover, mixtures made with 30% RCA have higher OBC than mixtures made with 0% RCA due to the porous nature of the mortar attached to the RCA surface (Pasandín and Pérez, 2013).

The air voids and voids in mineral aggregates are also higher for the mixtures manufactured with 4 hours of curing time. Again, the higher bitumen absorption that takes place during the time that the mixture is in the oven is mainly responsible for this performance (Pasandín and Pérez, 2013). Additionally, it can be observed that the mixture with 30% RCA presents higher  $V_m$  and VMA values due to the higher difficulty of compacting these mixtures as a consequence of the roughness of the RCA surface (Pasandín and Pérez, 2013).

#### **4.2. Bitumen basic characterization**

Figure 5 shows the results of the penetration tests (Figure 5a) and of the ring and ball (R&B) softening point or R&B temperature tests (Figure 5b) carried out on the bitumen samples studied.

First, the basic characterization results of virgin bitumen B50/70 (equivalent to 0 minutes of RTFOT), B50/70 after standard RTFOT (75 minutes) and B50/70 after 4 hours (240 minutes) of extended RTFOT have been plotted against the ageing time. This allowed us to calculate the regression lines presented in Figure 5a and Figure 5b. These regression lines were then used to estimate the equivalent RTFOT ageing time needed to obtain the penetration and softening point properties of the bitumen samples recovered from the four asphalt mixtures studied in this work.

The superposition of the penetration and ring and ball test results for the recovered bitumen samples in the regression line equations (Figure 5a and Figure 5b) shows that the basic

properties of the bitumen samples from recoveries 1 and 2 (after heat treatment) are very similar to those of the bitumen after four hours of extended RTFOT ageing time. Moreover, the bitumen samples from recoveries 3 and 4 (after production, without heat treatment) have properties similar to those of the bitumen after 75 minutes of standard RTFOT ageing time (short-term ageing during production). Thus, it was concluded that RTFOT can be a useful ageing method to estimate the basic properties of aged bitumen contained in asphalt mixtures (with or without RCA aggregates), both after production (short-term ageing) and after heat treatment for a defined curing time in the oven.

Figure 5a and Figure 5b also show the great differences between the performance of the bitumen recovered from HMA produced without heat treatment and the performance of the bitumen recovered from HMA manufactured with 4 hours of curing time in the oven. In this regard, the bitumen recovered from asphalt mixtures conditioned with 4 hours of curing time in the oven is much stiffer (i.e., lower penetration values and higher R&B softening temperatures for recovery 1 and recovery 2) than the bitumen recovered from mixtures produced without heat treatment (recovery 3 and recovery 4). Thus, it is also clear that the basic properties of the recovered bitumen are very dependent on the ageing phenomena associated with the heat treatment of the asphalt mixtures in the oven prior to compaction (independently of using RCA aggregates), which greatly increases the stiffness of the bitumen.

Looking into the stiffening effect of using RCA aggregates in asphalt mixtures in more detail, different conclusions can be obtained before and after using the heat treatment. In fact, when analysing the basic properties of the bitumen recovered from mixtures produced without curing time in the oven, it was observed that the mixture with RCA shows a slightly higher penetration value and lower softening point temperature than the mixture produced with natural aggregates, thus resulting in lower RTFOT equivalent ageing times. This trend seems to indicate that the porous mortar attached to the RCA surface protects the bitumen and limits its short-term ageing during production. However, when mixtures are treated with an additional four hours of curing

time in the oven, the bitumen recovered from the mixture manufactured with RCA presents similar penetration values and slightly higher ring and ball temperatures than that recovered from the mixture produced with natural aggregates, thus resulting in similar or slightly higher RTFOT equivalent ageing times. Thus, when applying the heat treatment, the use of RCA aggregates seems to be detrimental for the bitumen, increasing its ageing. Finally, the stiffening effect of using RCA instead of natural aggregates is not as pronounced as that caused by bitumen ageing during the additional four hours of heat treatment.

### **4.3. Bitumen rheological characterization**

The DSR results for the 25 mm plate and for the 8 mm plate of the bitumen samples studied in this work are presented in Figure 6.

The initial variation in the complex shear modulus versus the test temperature is presented in Figure 6a for lower temperatures (15 °C to 39 °C) and in Figure 6b for higher temperatures (46 °C to 88 °C). As expected, higher ageing times lead to a higher complex shear modulus. As is well known, thermal ageing stiffens the bitumen, which becomes more brittle and, as a result, reduces the durability of the pavement (Cong et al., 2012), although it can also increase the high temperature deformation resistance. Moreover, the use of higher DSR test temperatures leads to a lower complex shear modulus due to the thermoplastic nature of the bitumen (Firoozifar et al., 2011).

It can be observed that bitumen samples from recoveries 1 and 2 (after heat treatment) have complex shear moduli similar to those obtained for the bitumen after an extended RTFOT of 4 hours of ageing time, both at lower (Figure 6a) and higher (Figure 6b) temperatures. Thus, it was confirmed that the extended RTFOT procedure with four hours of ageing time closely represents the ageing of the bitumen recovered from asphalt mixtures after the heat treatment. Moreover, some rheological differences between the bitumen samples recovered from mixtures with natural (recovery 1) and RCA (recovery 2) aggregates were also observed after the heat

treatment. At lower temperatures (Figure 6a), the bitumen from recovery 2 (RCA) shows a slightly higher shear complex modulus, due to the reduced bitumen film thickness around the aggregates (as a consequence of the porous nature of the RCA-attached mortar) in comparison to that of the mixture without RCA. The reduced bitumen film thickness is detrimental to its ageing resistance. At higher temperatures (Figure 6b), the difference between using natural and RCA aggregates is barely noticed. Bitumen ageing is associated with an increase in the molecular size due to molecular structuring of heavy bitumen components, which increases the complex modulus of the bitumen. However, at higher temperatures, these new molecular structures can be dispersed more easily, disguising the stiffening effect of ageing. This may justify the small differences observed between the shear complex moduli of aged bitumen recovered from mixtures with natural and RCA aggregates at higher DSR test temperatures.

In the case of bitumen samples obtained from recoveries 3 and 4 (short-term ageing after production, without heat treatment), the complex shear modulus results are quite different. In this regard, bitumen from recovery 3 (natural aggregates) shows a behaviour closer to that of the bitumen obtained after the standard RTFOT, while the bitumen from recovery 4 (RCA) behaves closer to the original bitumen. The lower complex modulus observed in the bitumen of the mixture with RCA is probably due to the porous nature of attached mortar, which absorbs part of the bitumen, protecting it from short-term ageing. In this case, even though the bitumen film thickness of the RCA mixture is thinner than that of the mixture with natural aggregates, the effect of thermal ageing is negligible and significantly lower than that observed after 4 hours of curing time.

Figures 6c and 6d show the variation of the phase angle versus the test temperature. As seen, the bitumen shows lower phase angle values for higher ageing times, which indicates that the bitumen is more elastic. Moreover, as was expected, due to the thermoplastic nature of the binder (Firoozifar et al., 2011), higher temperatures make the bitumen more viscous and it therefore shows higher phase angles. In general, the range of temperatures in which the elastic

component is higher than the viscous component (phase angles lower than  $45^\circ$ ) depends on the ageing degree of the bitumen. In general, it is observed that the ageing process increases the elastic component and reduces the viscous component of the bitumen.

Bitumen samples from recoveries 1 and 2 (after heat treatment) show phase angle values lower than those obtained for the bitumen after extended RTFOT of 4 hours of ageing time, both at lower (Figure 6c) and higher (Figure 6d) temperatures. This result demonstrates that the ageing process which occurred during the heat treatment can be slightly more intense than that of the extended RTFOT procedure, increasing the elastic behaviour of the bitumen. The phase angle differences observed between the bitumen samples recovered from mixtures with natural (recovery 1) and RCA (recovery 2) aggregates after heat treatment were in agreement with the previous observations of the complex modulus. At lower temperatures (Figure 6c), the bitumens from the mixture with RCA show slightly lower phase angles than those of the mixture without RCA. Nevertheless, at higher temperatures (Figure 6d), there are no differences between the bitumen recovered from mixtures made with and without RCA.

Phase angles of bitumens from recoveries 3 and 4 (short-term ageing after production, without heat treatment) are higher than those obtained for the bitumen aged using the standard RTFOT. For these recoveries, the bitumen from the mixture with RCA presents phase angle values higher than those obtained from the mixture without RCA, both at lower (Figure 6c) and higher (Figure 6d) temperatures. Again, this can be explained by the porous nature of the RCA-attached mortar, the pores of which protect the bitumen against the short-term ageing effect of the mixing and compaction operations.

#### **4.4. Dynamic viscosity**

Figure 7 represents the dynamic viscosity versus the temperature. As expected, higher RTFOT ageing times resulted in higher dynamic viscosity of the bitumens. Moreover, the heat treatment of asphalt mixtures in the oven also results in higher dynamic viscosity of the recovered

bitumen. It must be noted that bitumens from recoveries 1 and 2 (samples manufactured with 4 hours of curing time) show viscosities slightly higher than those of the bitumen from the extended RTFOT of 4 hours, while bitumens from recoveries 3 and 4 (samples manufactured without curing time) present viscosity values just below those of the bitumen from standard RTFOT. Thus, again, RTFOT appears to be an adequate test to simulate the ageing that occurs during the heat treatment in the oven as well as the short-term ageing during production.

The viscosity of the bitumens obtained from both mixtures subjected to heat treatment is similar. However, the bitumen recovered from the mixture with RCA shows slightly higher viscosity values than those of the bitumen recovered from the mixture without RCA, due to the thinner bitumen film thickness, which results in a higher ageing effect from the heat treatment. Moreover, in mixtures manufactured without heat treatment, the porous nature of the RCA-attached mortar protects the bitumen from short-term ageing during mixing and compaction operations, leading to bitumens with slightly lower viscosity values in comparison with the bitumen of the mixture produced with natural aggregates.

## **5. Conclusions**

Based on the bitumen properties evaluated (penetration, softening point, complex modulus, phase angle and dynamic viscosity), it can be confirmed that the ageing effect of the heat treatment significantly increases the stiffness of the mixtures subjected to a curing time of 4 hours in the oven. The effect of the amount of RCA is much smaller and acts differently on mixtures subjected to the heat treatment when compared with those produced without curing time.

Taking that into account, it can be concluded that when there is no curing time, the RCA protects the bitumen against ageing. The pores of the mortar attached to the RCA surface protect the bitumen from short-term ageing (during mixing and compaction operations).

On the contrary, when using a heat treatment (4 hours of curing time in the oven), the use of RCA may have a detrimental effect on the bitumen ageing. The higher bitumen absorption of RCA leads to a thinner bitumen film thickness around the aggregates, thereby making the bitumen more susceptible to thermal and oxidative ageing. In this case, the protective action of the attached mortar pores is overtaken by the greater influence of the lower binder film thickness.

Moreover, based on the basic properties of the bitumens, it was observed that the ring and ball test was able to reflect the effect of ageing more accurately than the penetration test. This conclusion may reflect on the common laboratory practices when using RCA.

It also must be taken into account that when analysing the rheological properties of bitumen, the effect of using RCA was more pronounced at lower temperatures, when the bitumen molecular structures created during ageing control its behaviour.

Additionally, an important conclusion that can be drawn from this study is that RTFOT appears to be an adequate tool to simulate the ageing that occurs during the curing time of asphalt mixtures made with RCA, when the mixtures are kept in the oven for a certain period of time. Thus, this test is very useful for determining the ageing that the bitumen will suffer when the mixture with RCA is manufactured using a heat treatment.

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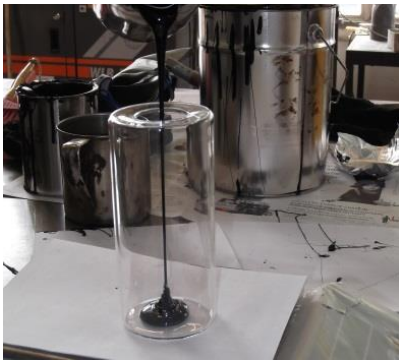
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Figure 1. Bitumen recovery procedure: a) Loose asphalt mixture; b) Centrifuge extractor; c) Centrifuge used to decant fines from the solution of toluene, bitumen and fines and d) Rotary evaporator used to separate bitumen from toluene



Figure 2. Rolling Thin-Film Oven Test procedure



$35 \pm 0.5$  g of bitumen were introduced into each glass container.



In the test, eight containers were used. Two of them were used to determine the loss of mass.



The containers with bitumen were rolling at a temperature of  $163 \pm 0.5$  °C while subjected to an air flow of 4 L/min.



After the RTFOT, samples of aged bitumen were prepared for DSR, dynamic viscosity, penetration and softening point tests.

Figure 3. Dynamic Shear Rheometer



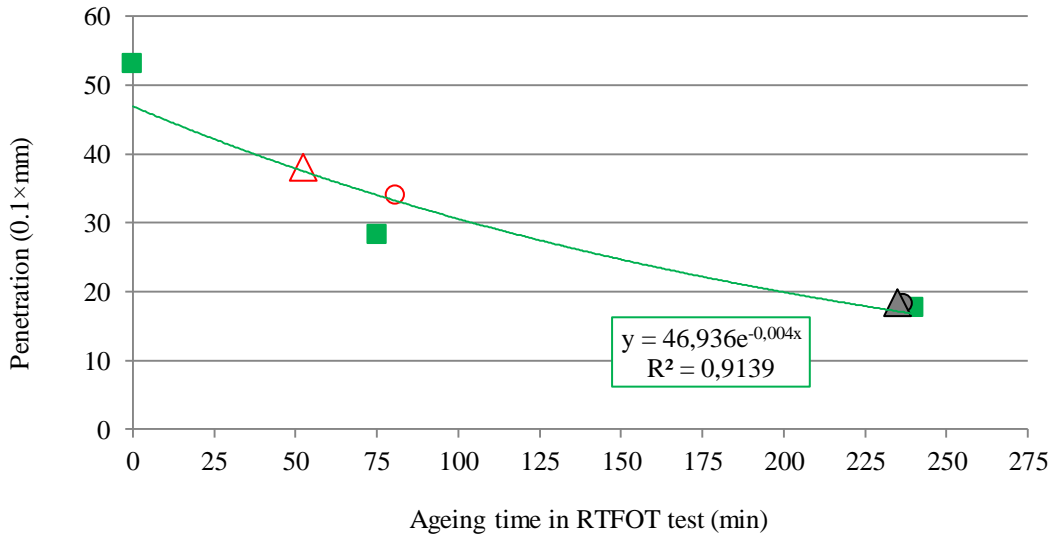
Figure 4. Rotating spindle apparatus used for the dynamic viscosity tests





Figure 5. Bitumen basic characterization results: a) Penetration; b) Ring and ball softening point.

a)



b)

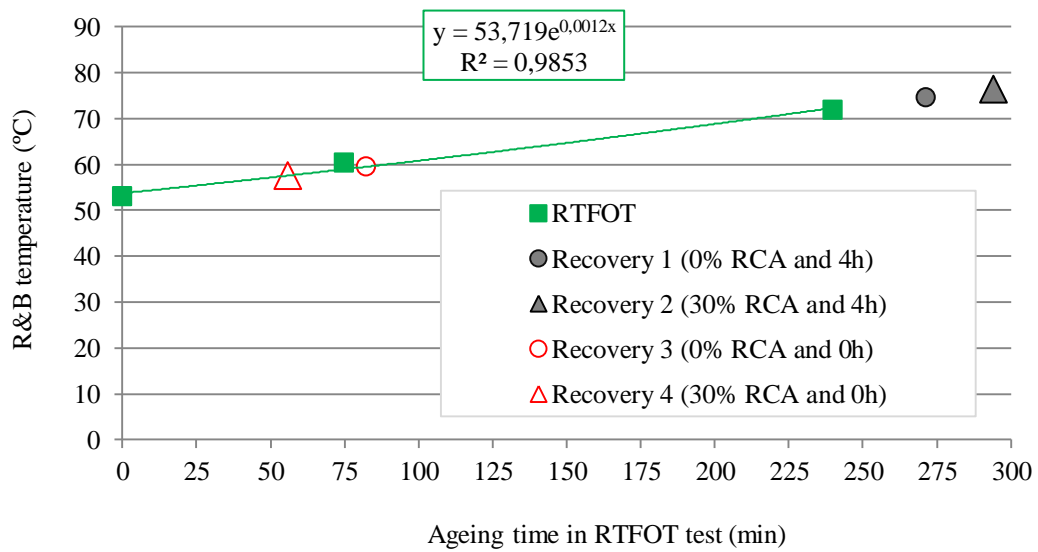


Figure 6. DSR results: a) Complex modulus for the 8 mm plate; b) Complex modulus for the 25 mm plate; c) Phase angle for the 8 mm plate and d) Phase angle for the 25 mm plate

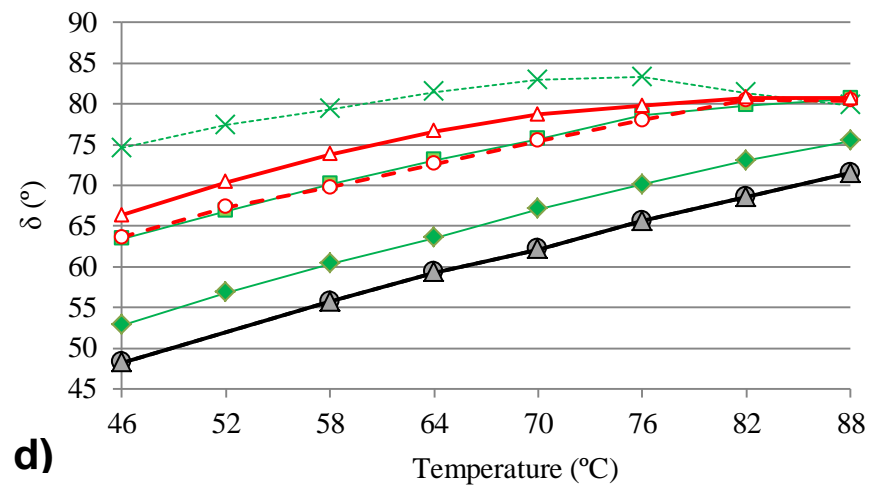
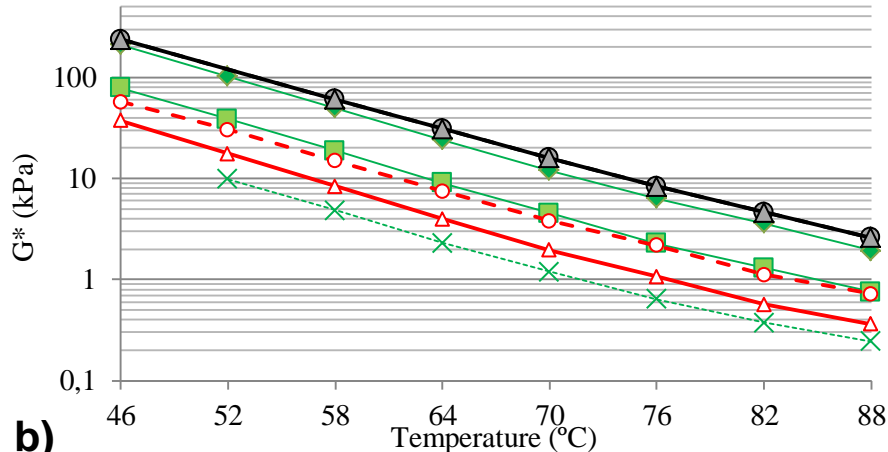
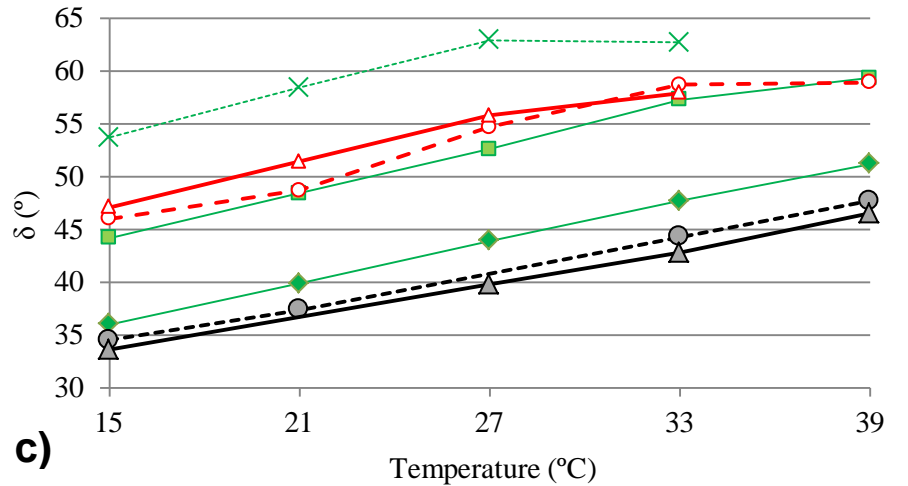
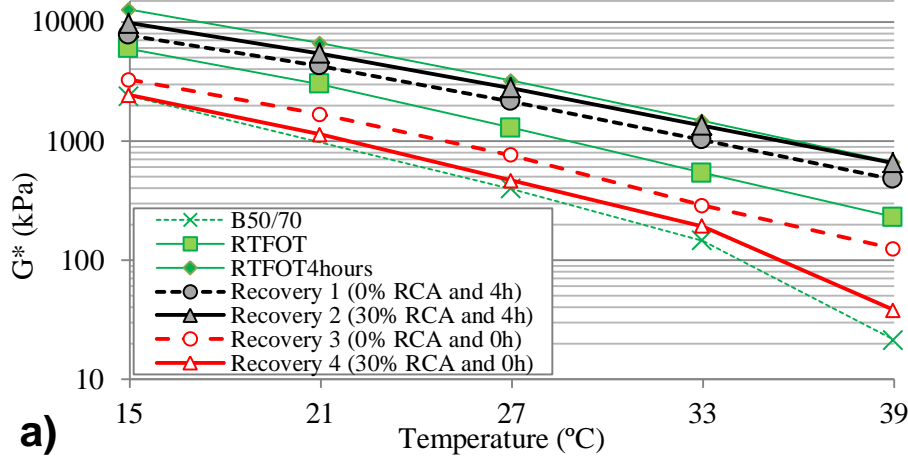




Figure 7. Dynamic viscosity results

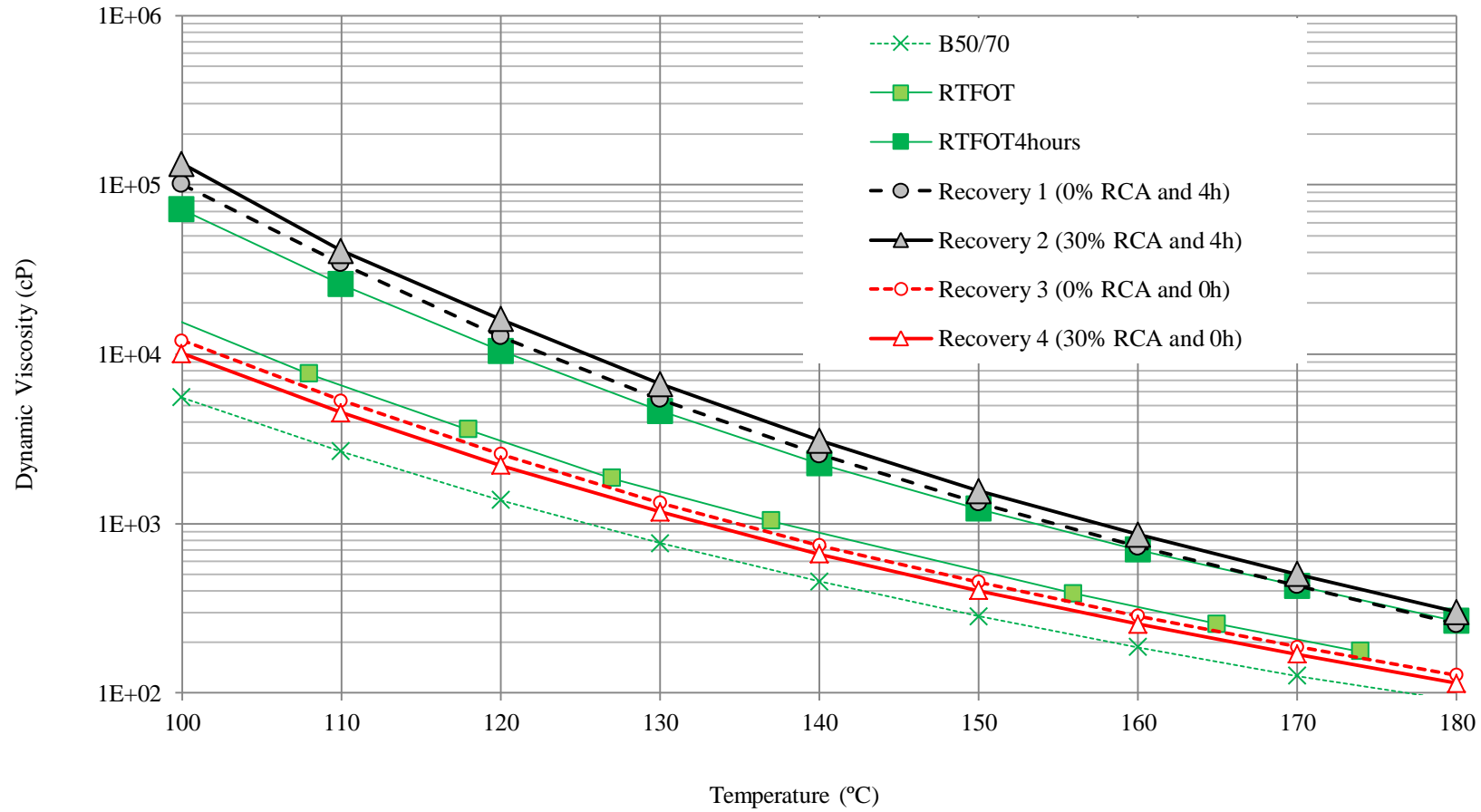


Table 1. Marshall mix design results

Curing time in the oven	RCA (%)	OBC (%)	Vm (%)	PG-3 specification for T00 to T0 (*)	VMA (%)	PG-3 specification
4 hours	0	4.0	7.25		16.84	
4 hours	30	4.3	7.71	5-8	17.71	≥14
0 hours	0	3.7	6.68		15.60	
0 hours	30	4.0	7.27		16.65	

(\*) Traffic category T00 refers to AADHT (Annual Average Daily Heavy Traffic) ≥ 4,000  
 Traffic category T0 refers to 4,000 > AADHT ≥ 2,000