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Characterization of binders recovered from RAP and recycling agents as a basis for evaluating the gree of mixing

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ABSTRACT

Given the need to improve the performance of asphalt pavements, the search for techniques that are durable and sustainable has become a reality in the paving industry. This includes the adoption of methodologies and new materials focused on reducing CO_2 emissions, with an emphasis on the use of RAP in recycled asphalt mixtures, specifically in analyzing the properties of the residual binder. In this context, the study evaluated the properties of binders recovered from RAP used in recycled asphalt mixtures. To this end, the binder was characterized with a focus on obtaining parameters that describe the interaction of the oxidized material with the other binders in the mixture. Laboratory analyses and performance tests revealed high stiffness in the recovered binder and in the blends containing higher proportions of that material, resulting in good rutting performance, although the fatigue resistance and integrity of both the isolated material and the binder blends were compromised; this can be explained by differences in viscosity and by the recovery observed when a recycling agent was incorporated. Based on this study, it can be concluded that complete blending of the binders does not depend solely on the characteristics of the blended materials, but also on boundary conditions, such as temperature, mixing time, and conditioning.

Keywords: RAP, pavements, recycled asphalt mixture, binder, blending.

1. INTRODUCTION

The transportation sector has been one of the main drivers of Brazil's economic growth. Associated with this expansion, there is a need to adopt strategies aimed at reducing GHG (greenhouse gas) emissions in the transportation sector [1]. Both system operation and infrastructure interventions must aim to reduce the carbon levels generated during implementation, as well as adopt new dosage methodologies and use new pavement materials.

It is known that pavement maintenance, rehabilitation, and reconstruction activities not only result in high emission rates but also require large amounts of natural materials and generate significant waste [2]. One such residue is Reclaimed Asphalt Pavement (RAP), a by-product used as a component in new asphalt mixtures [3-6].

A large body of literature on the subject demonstrates that mixtures containing RAP tend to exhibit high stiffness, and this property may be related to the composition of the binder remaining in the RAP, the degree of aging of this binder, and its interaction with new asphalt materials [7-9].

Regarding asphalt mixtures with RAP characteristics, conducted a study that evaluated the properties of binders used in these recycled mixtures [6]. While existing research shows that the fatigue life of asphalt mixtures is significantly affected by the amount of RAP used, the author noted that there is not a linear relationship between the percentage of RAP incorporated and the reduction in the mixture's fatigue life. Interestingly, the study found that the mixture with 45% RAP performed better in terms of fatigue life compared to the mixture containing 25% [10].

To better understand the behavior of these mixtures, it was necessary to study the remaining binder in more detail to understand its behavior and interaction with other binders. Research indicates that temperature, mixing time, the degree of aging of the recovered binder, and its interaction with other materials are crucial factors for the performance of the mixture [6].

Thus, the investigation into new technologies and methodologies for the recovery and reuse of materials from pavement restoration services has become increasingly relevant, especially due to the rising demand for higher percentages of RAP utilization. In Brazil, plants currently have the capacity to recycle 40% by mass; however, behavioral changes start to become apparent at percentages above 25% [11]. To work with higher contents, it is essential to carry out more detailed analyses. This includes verifying the degree of oxidation of the remaining binder in the RAP, determining the amount and quality of the aged binder, and assessing how it interacts with the virgin binder. To resolve uncertainties regarding the use of recycling agents and new virgin binder fractions, studies have concentrated on the advanced characterization of binders in recycled asphalt mixtures [6]. This is important because when an oxidized binder mixes with virgin material, it can initiate an accelerated oxidation process [12].

About material properties, certain processes and techniques for binder recovery are known, involving solvent extraction for subsequent recovery of the solution and evaluation of the properties of the material. Studies demonstrate that among the common solvents used, chlorinated ones tend to affect the material's properties, such as accelerating binder aging, increasing viscosity, and altering the softening point. These changes can result in a gain in stiffness, increased consistency and density, and reduced penetration, resulting in higher

resistance to diffusion [13-19].

Such changes in the physical properties of the recovered binder may indicate the need for a recycling agent (RA) to adjust and balance the material's characteristics, ensuring suitability for use in new asphalt mixtures. During the balancing process, a recycling agent is added to the matrix as an additive for recycling asphalt mixtures, both in a plant and on-site. This agent can help replace maltene fractions in aged binders [20]. This occurs because many RAs contain a high number of neutral oils, enabling the chemical balance of the aged binder and modifying the structure of the materials [21-22].

Consequently, to assign a recycling agent, it is necessary to know the properties of both the recovered binder and the RA. These agents can increase the ductility of asphalt mixtures with high RAP content, showing favorable results in resistance to premature cracking, longer fatigue life, and better performance against rutting [23].

However, because this material is relatively new to the pavement industry and given its variety, which can be petroleum-based, plant-based, or even derived from waste, there is no consensus on determining the ideal content of the recycling agent or the specific timing of its addition during the production process for recycled asphalt mixtures [24]. Therefore, it is necessary to understand the mechanisms involved in the interaction between binders.

Among the mechanisms involved, one key mechanism is the degree of binder activity (DoA), which is an intrinsic property of each type of RAP material and corresponds to the minimum amount of aged binder that can be considered “active” for the formulation of new mixtures, regardless of the addition of recycling agents [25]. The Degree of Blending (DoB) refers to the point where the RAP binder contributes to the rheological, chemical, or conventional properties of a homogenized binder blend. The functionality of these indicators is related to the RAP type and processing conditions, such as mixing time and temperature [26]. Binder blending or activation is not a straightforward process; difficulties in blending directly influence the final performance of the asphalt mixture [27].

Analyzing this blending, three hypotheses on interaction conditions were defined: the binder behaving as an inert material (black aggregate), complete blending, and partial blending [28-31]. In this process, it is more accurate to say that partial blending occurs in blends with high RAP contents [32-33].

Regarding DoA and DoB, effective results are influenced by the properties of the mixture components and processing conditions. Interaction is affected by external factors related to material diffusion, such as mixing temperature, time, and material viscosity [34].

To further correlate performance with the degree of blending, Fick's laws have been used to describe mass transfer fluxes and concentration gradients in molecular diffusion [35]; Fick's second law is most applicable, though models must adopt considerable thicknesses of virgin binders over RAP aggregates since the actual diffusion coefficient is related to the asphalt binder film thickness [27].

Diffusion between binders is governed by time, film thickness, the proportion of virgin binder, and the diffusion coefficient [36]. The diffusion coefficient is the thermodynamic parameter governing the speed at which oils and asphalt molecules mix, migrate, or age [37].

To evaluate the degree of mixing between virgin and oxidized binders and to develop testing methodologies that would better identify whether mixing had occurred, researchers sought to divide the process into five interaction hypotheses, namely: indirect measurement methods, identifiable difference methods, staged extraction methods, tracer labeling methods, and in situ microscopic observation methods [36-41], as listed in Table 1.

Table 1- Main methods for evaluating the degree of binder blending.

Influencing Factors	Evaluation Method	Main Results
Conditioning time after mixing	Indirect measurement	Extended conditioning time can improve the blending degree between virgin and RAP binders and alter the PG [41].
Virgin binder properties	Identifiable differences	Lower asphalt content in RAP leads to a higher blending degree. Less viscous virgin binders result in a higher blending degree [37].
Mixing time	Identifiable differences	The blending degree improved as a result of increased mixing time [40].

Conditioning time after mixing	Staged extraction	Increasing the temperature and extending the curing time can improve the degree of mixing between virgin asphalt binders and RAP [36].
Mixing time	*In situ* microscopic observation	The blending degree improved as a result of increased mixing time [39].
Presence of recycling agents	Indicator marking	The addition of recycling agents contributed to the blending degree [38].

Considering that the distribution concentration of virgin asphalt binders varies over time, variability exists for the remaining RAP binder; thus, the diffusion system tends to be dynamic and operates in a non-steady state.

2. METHODOLOGY

The experimental program was organized in stages: material acquisition, RAP processing, extraction and recovery of the remaining binder, execution of blends, and material characterization through conventional, rheological, and performance tests. Figure 1 describes the stages of the experimental program and the analyses performed.

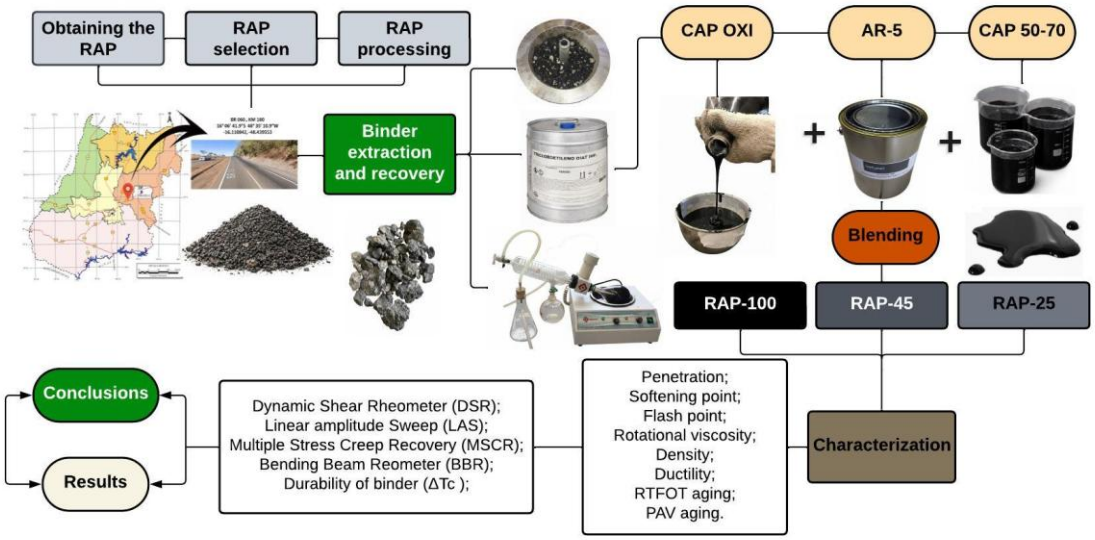


Figure 1- Research methodology flowchart.

In addition to the recovered binder, a CAP 50-70 virgin binder (a Brazilian penetration-grade asphalt binder) and a commercial recycling agent known as CICLOPAV 100 (classified as AR-5) were evaluated. This AR-5 is common in hot-mix recycled asphalt services and has shown improvements in workability [10, 42], reduction in the viscosity of oxidized binder, and lower processing temperatures due to the chemical rebalancing of the aged binder, which makes it more suitable for use in asphalt mixture recycling operations.

3. EXPERIMENTAL ANALYSIS

The experimental analysis consisted of tests aimed at obtaining the materials and subsequently characterizing their physical and performance properties, both isolated and combined.

3.1 BINDER ACQUISITION PROCESS

To obtain the oxidized binder, extraction was performed using trichloroethylene solvent and a Rotarex extractor, followed by recovery from the RAP through solution filtering and processing in a rotary evaporator with CO_2 application to enhance recovery effectiveness.

The RAP was obtained from the milling of the asphalt surface of a section of BR 060, km 180, in the state of Goiás, Brazil. The residue underwent processing involving selection, removal of organic elements, and other debris. After processing, the material was heated in an oven and pulverized using a crucible and mortar for grain size distribution.

Only the coarse matrix of the RAP (retained on the #4.8 sieve) was used for extraction to ensure better binder recovery and more effective filtration and sedimentation. Trichloroethylene was used as the solvent for the extraction of the solution, employing the centrifugal extraction method using Rotarex equipment. The choice of method was based on a statistical analysis that considered the percentages of residual material that may be present in the sample when using three distinct determination methods (centrifugation—Rotarex, muffle furnace combustion, and Soxhlet extraction) [10].

Regarding solvents, it is well known that they have a significant influence on the characteristics of recovered asphalt binders and can change their physical, chemical, and rheological properties. In view of this, trichloroethylene was chosen for its dissolving capacity and its ability to alter the material's characteristics; among chlorinated solvents, it is the one that causes the least change in properties [13].

Following extraction, the resulting solution was filtered and decanted to remove any residual fines from the sample, after which the ligand was recovered by rotary evaporation, with CO_2 added to accelerate the process.

Once recovered, the "CAP OXI" was conditioned in an oven at 160°C until the blends with virgin binder and AR-5 were prepared.

4. PROCESS FOR PRODUCING COMBINED BINDERS

Given that this analysis is intended to characterize asphalt binders and evaluate changes in the structure of the recovered material when mixed with virgin binders and recycling agents, a virgin CAP 50-70 binder and an AR-5 class recycling agent were also used as test materials in this study.

CAP 50-70 was chosen due to its wide use in hot-mix projects in Brazil, and also based on existing research on this material, such as [43-44]. The recycling agent used in this study was the same one used in the studies of [42, 45].

This recycling agent consists of a shale oil-based product classified as an AR-5, commonly used in the hot mixing of recycled asphalt mixtures, improving workability, reducing the viscosity of the oxidized binder, and potentially lowering the processing temperature, with the potential to restore the chemical balance of the aged binder, making it more suitable for use in asphalt mixture recycling applications in pavement construction.

Three additional binder types were studied, obtained by mixing virgin binder, CAP OXI, and AR-5. The alphanumeric nomenclature corresponds to the percentage of remaining CAP OXI in the sample.

For blending, the three base binders, CAP OXI, CAP 50-70, and AR-5, underwent conditioning (heating), with CAP OXI heated to 160°C, CAP 50-70 to 145°C, and the recycling agent to 120°C. The mixing was performed mechanically using a rotating dispersing rod on a heating plate set to 140°C, resulting in the RAP-100, RAP-45, and RAP-25 blends.

The samples of the RAP-100, RAP-45, and RAP-25 received 20% recycling agent (AR-5), based on the oxidized binder content (CAP OXI) incorporated into the binder mixture. The 20% AR-5 content was determined based on studies [10, 42, 45] that used AR-5 and adopted this percentage in relation to the residual binder content present in the RAP. In this study, however, the 20% AR-5 content was employed based on the recovered residual binder content that would be present in the blended samples under evaluation.

For illustration purposes, considering a 100 g sample of RAP-45 binder, the material was composed of 45 g of recovered binder, 9 g of AR-5 (corresponding to 20% of the 45 g of recovered binder), and 46 g of virgin CAP 50-70 binder.

5. RESULTS

5.1 EMPIRICAL AND CONVENTIONAL CHARACTERIZATION OF BINDERS

Binder characterization was conducted in two stages. First, empirical characterization was performed using conventional tests widely accepted in Brazil. Tables 2 and 3 present the results for the virgin binders, RAP binders, blending, and RA.

Table 2- Conventional characterization of binders.

Tests	ANP Resolution 897/22 Limits	CAP 50-70	CAP OXI	RAP 100	RAP 45	RAP 25
Penetration (mm)	50-70	53.7	29.4	34.8	43.3	48.2
Softening point (°C)	min. 46 °C	48.3	64.1	60.7	55.2	52.1

Flash point (°C)	min. 235 °C	302.4	194.6	219.4	257.13	278.69
Rotational Viscosity Brookfield (cP)	min. 274 cP	342	600	488.39	398.23	346.71
	min. 112 cP	171	528	426.38	301.43	230.03
	min. 57 cP	58	417	335.7	210.05	138.25
Susceptibility index	(-1.5) to 0.7	-1.47	0.59	0.33	-0.31	-0.77
Density (g/cm³)	_	1.0117	1.1417	1.15	1.1044	1.0784
Ductility (cm)	min. 60 cm	> 100	18.6	35.8	64.8	81.6
Elastic recovery (%)	_	10	19.3	17.4	14.2	11.8
RTFOT Aging 163°C and 85 minutes ASTM D2872/22	max. 0.5	0.287%	–	–	–	–
	min. 55	59.96% **(32.2 mm)**				
	max. 8	2.6 °C **(50.9 °C)**				

Table 3- Characterization of the recycling agent.

Test	Method		Limits	AR-5
Flash point (°C)	NBR 11341		min. 218 °C	318.6 °C
Rotational viscosity Brookfield (cP)	ASTM D 4402M/23	135°C	-	41.96 cP
		150°C	-	19.92 cP
		177°C	-	10.5 cP
	ASTM D 2171M/22	60°C	176-900 cP	409.78 cP
Density 20/4°C (g/cm³)	NBR 6296		-	1.1826 g/cm³

Based on Tables 2 and 3, the recovered material exhibited significant stiffening and low penetration due to oxidation. All binders met the limits set forth in Resolution No. 897/2022 [46], except for the CAP OXI and RAP-100 binders, which had penetration and ductility values below the minimum required by the regulation, which also affected the rotational viscosity of these materials. This reduction in penetration may be related to the amount of CAP OXI in the sample, since these samples exhibited high consistency, possibly due to other materials introduced during the extraction and recovery process.

Since viscosity is directly related to material aging, materials with higher levels of residual binder exhibit increased viscosity due to a reduction in the percentage of neutral oils in the sample. In the context of asphalt mixture formulations, this increase can affect the aging of the mixture and, in the absence of temperature, make homogenization more difficult. Increases in density and viscosity have also been reported to reduce the workability of the material [2, 42].

.It should also be noted that high viscosities may require higher temperatures and longer mixing times to allow proper diffusion between the materials. This is consistent with the ductility results, which vary with the amount of remaining binder in the sample. The elastic recovery data, however, were disregarded, as they did not yield satisfactory values.

Regarding the AR-5, it met the standards established by Resolution No. 897/2022 [46] and by the manufacturer. When evaluated separately, it shows low viscosity under the same shear conditions used for the asphalt binders during the test. In practice, this implies increased fluidity of the material, making it flow more easily and improving the workability and lubricity of the mixture.

Materials with higher CAP OXI contents exhibited higher softening points temperature and lower flash points temperatures. This is consistent with the fact that the solvent used to extract and recover the binder is non-flammable, so any aromatic compounds detected in the samples come exclusively from the AR-5 and CAP 50-70 fractions — which are present in relatively low proportions in the CAP OXI and RAP-100 binders evaluated.

Among the binders studied, AR-5 had the highest density, with the others having very similar values. It should also be noted that, since the recycling agent in question is not petroleum-based, it required higher temperatures to remove the volatile gases enclosed in the sample during the flash point test.

Therefore, only the CAP 50-70 binder — both virgin and after short-term aging — met the defined limits for all conventional empirical tests. CAP OXI and the blends did not meet the specifications [46], failing to satisfy the limits due to the material's high stiffness.

However, it can be assumed that binders containing residual RAP in their composition tend to exhibit increased viscosity, associated with reduced penetration, requiring higher temperatures to achieve workability. Furthermore, these same binders tend to lose their binding and recovery properties, which can result in asphalt

mixtures with low fatigue life when processed with these materials. This process is influenced by boundary conditions.

5.2 MECHANICAL CHARACTERIZATION OF BINDERS

The mechanical performance characterization evaluated resistance to rutting, fatigue life, and integrity, considering three scenarios: binder that is recently milled (virgin), fully aged binder (CAP OXI), and binder in a blended state — the condition that begins the moment the asphalt mixture is prepared, with the addition of RAP, virgin binder, and a recycling agent.

About rheological and performance characterization, analyses were conducted on the CAP 50-70, AR-5, CAP OXI, RAP-100, RAP-45, and RAP-25 binders. To obtain the dynamic shear modulus (|G*|) values and phase angle (δ) used in the sigmoidal fit, and to calculate the non-recoverable compliance, three repeat tests (samples) were conducted for each experimental condition, based on the frequency and temperature sweep test [47].

The master curves of the dynamic shear modulus (|G*|) and phase angle (φ) of the binders were plotted for a reference temperature of 28°C, using the sigmoidal fit model for asphalt binders — the same reference temperature adopted by [43] who analyzed the same CAP 50-70 binder in their research.

The master curves are presented in Figure 2. For the CAP 50-70 binder, two scenarios were considered: the virgin condition and short-term aging by RTFOT.

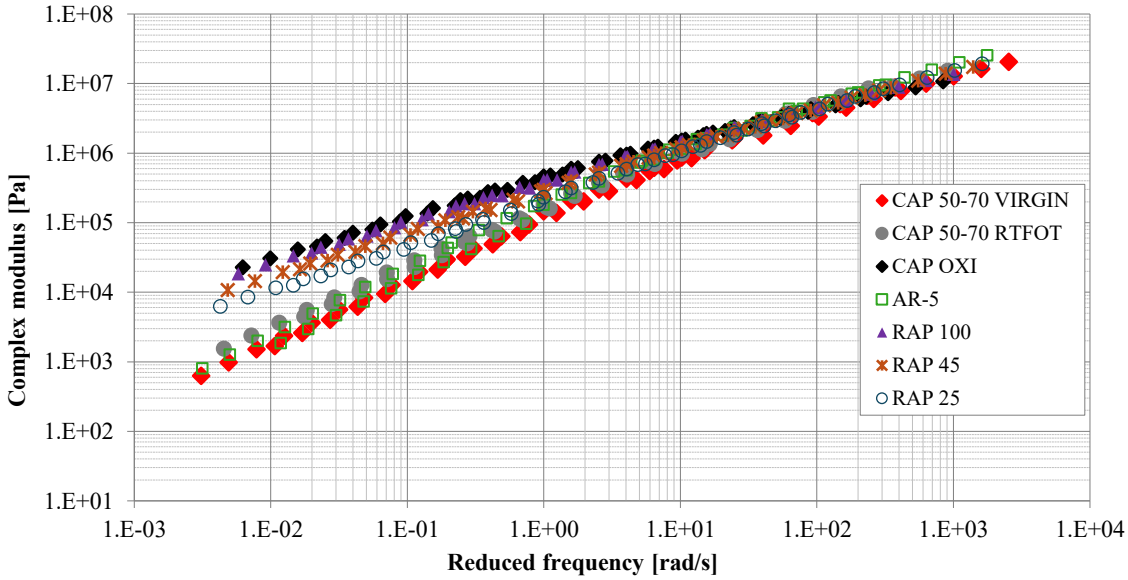


Figure 2- Master curve of the binders in the 28°C TR |G*|.

Looking at the master curves of the CAP 50-70 binder in its virgin and short-term aged conditions, the dynamic shear modulus |G*| increases gradually with frequency, as also reported by [43]. This behavior is characteristic of the material at low temperatures, where it tends to behave as an elastic solid. Short-term aging did not cause major changes in the material's viscoelastic response, which is not the case when low frequencies and high temperatures are considered.

The AR-5 curve fell between the virgin and aged CAP 50-70 curves, which was expected given the large amount of aromatics in its composition — a fraction that tends to be lost during aging. An increase in the shear modulus was observed as frequency increased. At higher frequencies, corresponding to low temperatures, AR-5 showed higher |G*| values than all the other materials analyzed, demonstrating its usefulness in restoring the workability of stiffer materials.

CAP OXI showed the highest |G*| values at low frequencies, corresponding to high temperatures, which may translate into greater rutting resistance but a shorter fatigue life due to the increased stiffness. RAP-100 and RAP-45 followed the same trend; accordingly, [48-49] report that the oxidation and aging process leads to stiffness gains that can be inversely proportional to thermal cracking performance.

RAP-25, in turn, contains more virgin than oxidized material and accordingly showed intermediate behavior between the virgin and oxidized binders, which may translate into good performance at both high and low temperatures, in terms of both fatigue and rutting.

To characterize the recoverable elastic response versus the non-recoverable viscous response to an applied load, the phase angle was determined for the binder samples, as detailed in Figure 3.

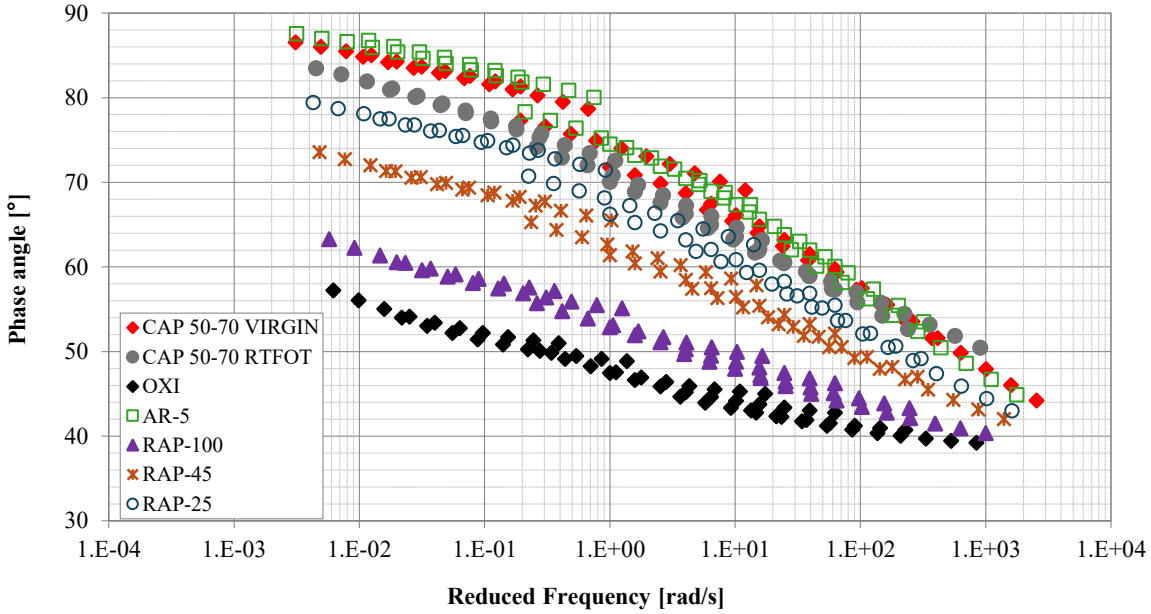


Figure 3- Master curve of the binders in the 28°C TR | δ |.

As shown in Figure 3, the materials with a higher proportion of recovered RAP binder exhibited lower phase angles, indicating more elastic behavior, whereas materials with phase angles close to 90° indicate purely viscous behavior.

This behavior is somewhat questionable for the CAP OXI materials, since RAP binders tend to make the material stiffer, and given its viscosity it was expected that CAP OXI and the *blending* would have difficulty showing an elastic, recoverable response — yet the opposite was observed.

This reversal shows that the mechanical behavior also needs to be evaluated using other methods, since the test methodology adopted, while effective at describing and indicating the material's performance trend, may not be deterministic.

It can also be observed that oxidation and aging make the binders less sensitive to temperature variation compared with virgin materials, [50-52] as is the case with the virgin CAP 50-70 binder and the recycling agent.

To evaluate the binders' rutting behavior, the Multiple Stress Creep and Recovery (MSCR) test [53] was performed over a temperature range of 22 to 76°C in 6°C increments, with compliance evaluated at 58°C using samples 25 mm in diameter with a 1 mm gap. The results for the non-recoverable compliance parameter (Jnr), weighted for stresses of 100 Pa and 3200 Pa, are shown in Figure 4.

Looking at Figure 4, which shows the compliance values at 100 Pa and 3200 Pa, it can be seen that AR-5, when evaluated in isolation, does not meet the criterion defined in [54], which classifies it as a material with low rutting resistance, unable to withstand standard (S) traffic loading. However, evaluating it in isolation is not really appropriate, since the agent is a component used to balance the properties of the binders in the blend.

It can be observed that the CAP OXI binder is classified for extremely heavy traffic (E), RAP-100 for heavy traffic (V), and RAP-45, RAP-25, and CAP 50-70 for standard traffic (S).

Regarding the non-recoverable compliance values at the high PG temperature — adopted as 58°C for this study — this is the temperature threshold above which the material has difficulty showing an elastic return, owing to its viscosity under that condition [55]. Figure 5 shows that all the materials met the limit established in [54], highlighting the change in behavior of CAP 50-70, whose Jnr dropped considerably, and of AR-5, which becomes classified for standard traffic (S), while RAP-25 is classified for heavy traffic (H), RAP-45 for very heavy traffic (V), and RAP-100 and CAP OXI for extremely heavy traffic (E).

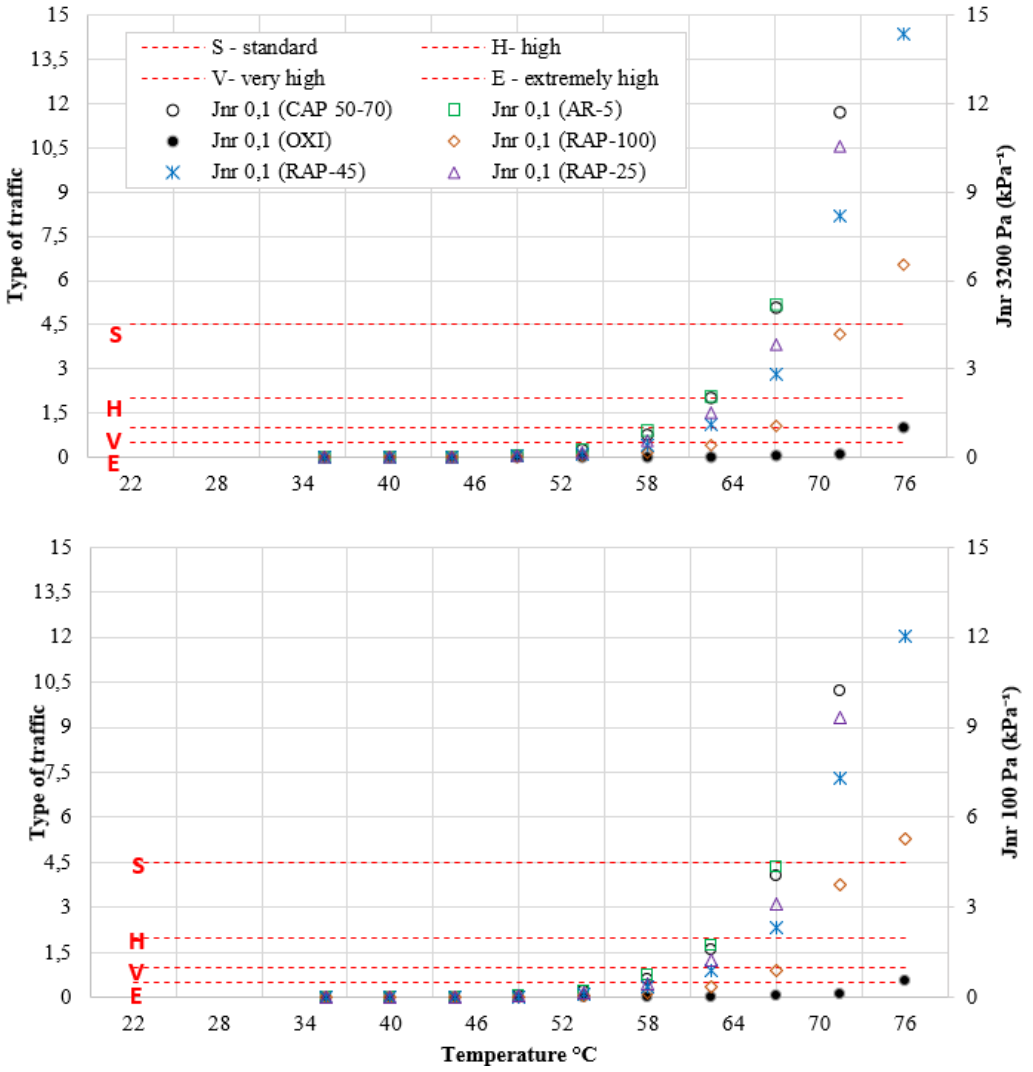


Figure 4- Non-recoverable compliance at 100 and 3200 Pa.

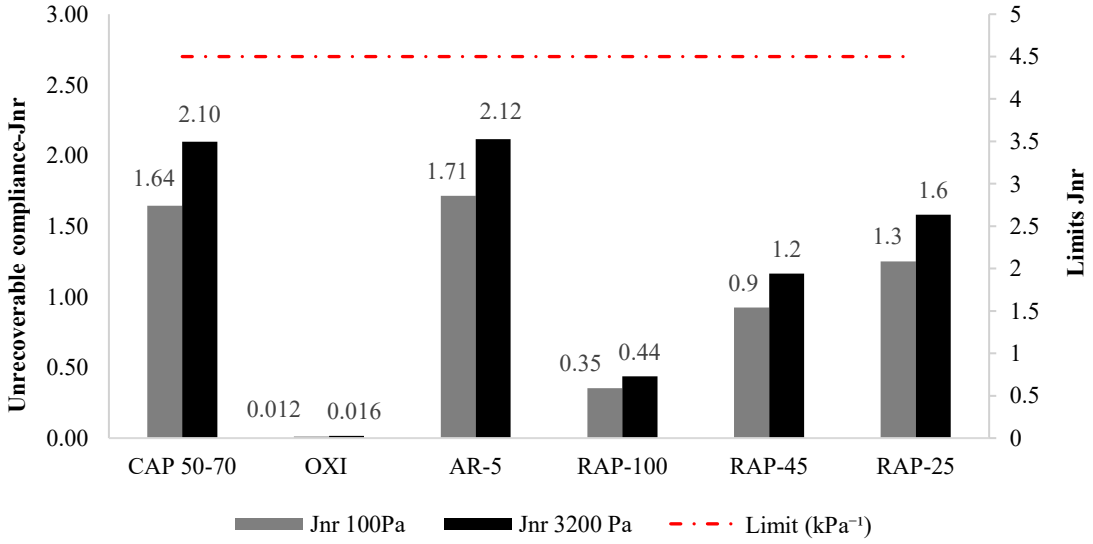


Figure 5- Non-recoverable compliance at 100 and 3200 Pa at 58°C.

It can therefore be assumed that the proportion of aged material in the sample reduces Jnr, which in turn implies greater rutting resistance — a situation quite similar to what occurs in polymer-modified binders [56].

In addition, to evaluate the material's sensitivity to changes in the applied stress level, the difference

between the non-recoverable compliance values was calculated. Under the adopted criterion, a material is considered non-stress-sensitive if this difference does not exceed 75%; as detailed in Figure 6, all the materials met this limit, with compliance differences below 37%.

Another parameter evaluated was the recovery of the samples after stress cycles of 100 Pa and 3200 Pa, which indicates the material's capacity for elastic return after being subjected to stress and relaxation events. The analysis was carried out over a temperature range of 22 to 76°C, with particular attention to the high PG temperature of 58°C. This type of assessment is common for elastomeric binders; however, since the RAP-containing materials showed small phase angles, the recovery evaluation was also carried out here to assess the elastic behavior of these binders.

The results shown in Figure 6 are consistent with those obtained from the frequency sweep, with the stiffer binders showing higher recovery values — which may be related to the role of the recycling agent and to structural changes that may have occurred during the recovery and *blending* processes of these binders. Relating the recovery percentage to the recoverable compliance (Jnr at 3.2 kPa) and to the recommended limits [57], it was observed that CAP OXI showed the lowest compliance, with values below 0.25 kPa^{-1}, meeting the recommended recovery requirement of above 50% — it in fact reached 74%. For RAP-100, compliance was below 0.50 kPa^{-1}, so the required recovery is above 45%, and the material showed values above 61%. For RAP-45 and RAP-25, compliance ranged from 1.2 to 1.6 kPa^{-1}; these materials are required to show recovery above 30%, and they reached values above 33%. As for CAP 50-70 and AR-5, their recoverable compliance exceeded 2 kPa^{-1}, which is characteristic of unmodified material.

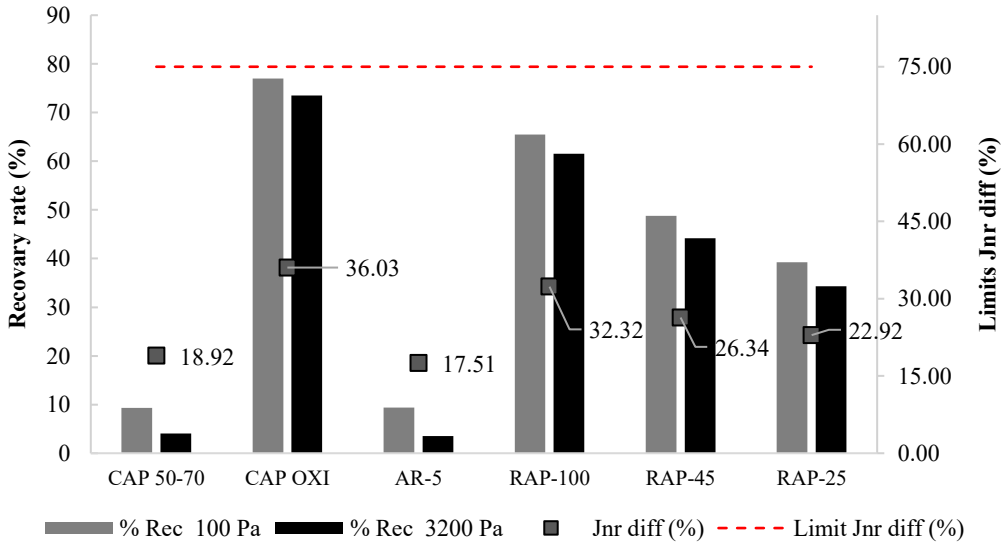


Figure 6- Recovery and Jnr diff at 58°C.

Based on the recovery results obtained, it can be assumed that the binders evaluated could be used in the mix design of asphalt mixtures intended for standard-to-heavy traffic, since they meet the requirements for good in-service performance.

Researchers report that higher recovery percentages indicate that the binder can recover a larger share of its total deformation at the end of each loading-unloading cycle, favoring the material's resistance to rutting [50, 56, 58-59].

To evaluate the binders' resistance to fatigue damage, the Linear Amplitude Sweep (LAS) test was performed on the DSR at an intermediate temperature of 19°C (20 ± 0.5°C), using parallel-plate samples 8 mm in diameter with a 2 mm gap. The test was run in two stages, performed in sequence: a frequency sweep to characterize the binders' viscoelastic properties, and an amplitude sweep applying shear at a frequency of 10 Hz to determine the damage to the material [60]. The virgin binder, recycling agent, and *blends* underwent the aging step [61]. Figure 7 shows the shear stress versus strain curves for the binders evaluated in this study.

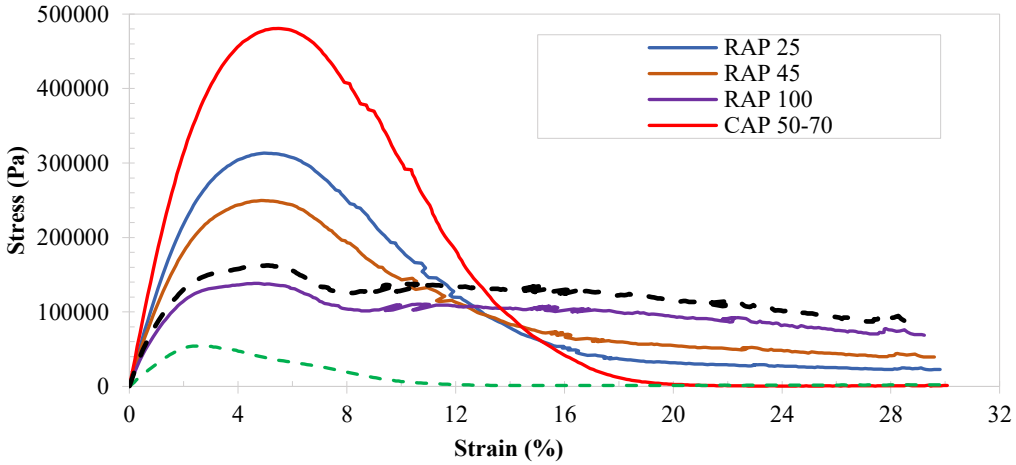


Figure 7- Stress-strain curve of the binders.

The curves show that, in the initial phase, shear stress increases linearly with the applied strain. Once the materials enter the non-linear region, stress peaks are reached at different strain levels. The shear stress applied to CAP 50-70 was the highest, increasing as the strain amplitude rose to about 6%, after which the stress declined beyond the peak.

Binders with higher proportions of RAP-derived material, such as RAP-100 and CAP OXI, showed lower stress peaks and slightly lower strain amplitudes than CAP 50-70, indicating lower damage resistance associated with the amount of oxidized material in the sample. The recycling agent showed the lowest shear stress value, at a small strain, which may be related to its composition and viscosity. RAP-25 and RAP-45, in turn, showed higher stress peaks at strain amplitudes similar to those of CAP 50-70.

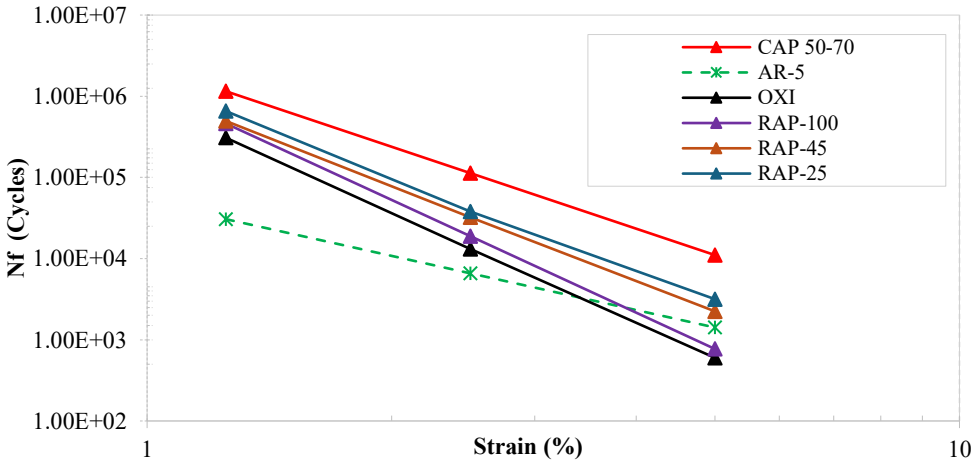


Figure 8- Fatigue life as a function of shear strain of the binders.

To evaluate the fatigue behavior of the binders, three strain levels (1.25%, 2.5%, and 5.0%) within the linear viscoelastic range were adopted, and the binder fatigue factor (BFF) was calculated. Looking at each material in isolation, CAP OXI showed the shortest fatigue life, and the fatigue life decreased in proportion to the percentage of RAP added.

However, some caution is warranted here: based on the initial modulus values, |G*|ini, this parameter is not believed to be very reliable, since the binder's initial stiffness evaluated in isolation is not a good predictor of its fatigue performance. This is confirmed by the study of [62] which details this non-linear behavior: when evaluating the fatigue performance of recycled asphalt mixtures with 100%, 45%, and 25% RAP, the mixture with 100% RAP by mass performed better than the one with 45%, also considering that the boundary conditions involved in the mixing process can influence material performance.

Therefore, even though the binders with higher CAP OXI contents showed lower resistance at higher numbers of cycles in the present study, it is necessary to look further and interpret the material's behavior across its different strain ranges within the linear viscoelastic range, rather than relying solely on the numerical value

of the BFF.

To this end, future studies could propose other indices derived from the LAS test to quantify different aspects of the material's fatigue resistance, such as the average reduction in integrity (I^R), strain tolerance (εT), strain energy tolerance (Ef), the Fatigue Resistance Energy Index (FREI), and the reduction in peak modulus (Δ|G*|peak τ) [63-66].

To predict the durability and integrity of the binders, ΔTc was calculated, as shown in Figure 9. This parameter evaluates the difference between the critical temperatures Tc,s and Tc,m, which refer respectively to the temperature (°C) at which stiffness (S) reaches the maximum limit of 300 MPa, and the temperature at which the relaxation parameter (m) reaches the minimum limit of 0.3 [41, 67]. In conventional PG terms, the more critical of these two temperatures determines the low-temperature PG grade [33], which was -10°C for this study.

The data used to calculate ΔTc were obtained from the BBR (Bending Beam Rheometer) creep test, from which the stiffness S(60) and the relaxation property m(60) were measured [68]. It is worth noting that that as the binder ages, ΔTc tends to decrease, becoming smaller, which points to a loss of relaxation capacity and an increased susceptibility to cracking. Cracking itself is defined as the point at which the material undergoes a considerable loss of its ability to relax stresses without cracking. Accordingly, positive values of ΔTc indicate that the material shows good integrity, while negative values already signal warning signs of cracking; -2.5°C is defined as the value indicating the binder is close to cracking, and -5°C describes a binder that has already begun cracking [65-67].

Looking at Figure 9, which details the temperature variation at which the binders reach maximum stiffness and minimum relaxation, CAP OXI shows the lowest durability. With a ΔTc of -10°C, the material can already be considered to have started cracking, which indicates that as the material's aging and oxidation increase, its low-temperature performance declines, since the ΔTc value becomes more negative [69].

As for the other materials, they also showed a risk of cracking, with RAP-25 identified as the material with the best low-temperature performance. Since none of the materials in this study showed a positive ΔTc value, it is believed that the relaxation properties of these binders are not in good condition, and thermal cracking is a real possibility, as the ΔTc of CAP 50-70, AR-5, RAP-100, and RAP-45 all came close to the warning threshold.

A trend observed in this study, then, is that an increase in the amount of remaining RAP binder is associated with a numerical reduction in ΔTc values, resulting in a loss of the binders' relaxation properties [70].

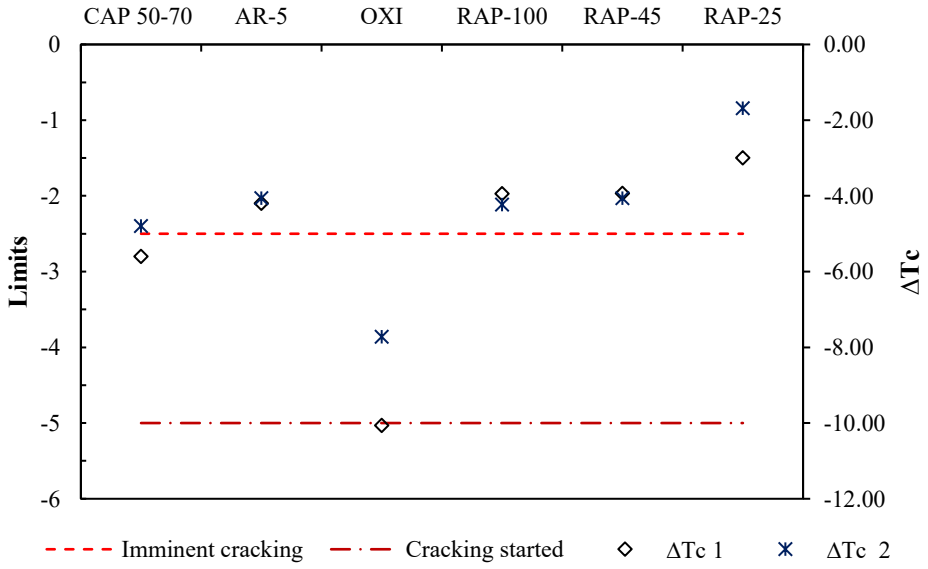


Figure 9- ΔTc of the binders.

It can therefore be assumed that the binders' relaxation rate (m-value) is related to their aging and oxidation, so binders containing residual RAP material in their composition will tend to show lower ΔTc values.

In this sense, it is not only the loss of relaxation that relates to ΔTc — other factors, such as the origin of the material, the production process, and the type of modifier or recycling agent used, may also be intrinsically related to it, as [71-75] showed in their study, finding that the ΔTc parameter is controlled mainly by the rheological index R, while other rheological parameters — such as activation energy, crossover frequency, and the glassy modulus — play a secondary role in the analysis.

Moreover, although ΔTc is obtained from a test designed to predict low-temperature failure, while fatigue cracking is related to intermediate temperatures, both phenomena are assumed to be linked to the binder's stress-relaxation potential [73].

5 CONCLUSIONS

Based on the study carried out — focused on characterizing the binder remaining in RAP and the binders used in recycled asphalt mixtures, with emphasis on the blends of virgin binder, oxidized binder, and recycling agent — the following conclusions can be drawn:

- The CAP OXI and recycling agent contents in the sample influence its fatigue resistance and, consequently, its integrity. The stiffness gain observed in asphalt mixtures produced with high RAP contents is due not only to the mineral matrix (aggregates) but mainly to the binders in the composition, so there is a relationship between the binder fatigue factor and the mixture fatigue factor.
- Binders whose blends contain higher amounts of CAP OXI tend to show an increase in the dynamic shear modulus |G*| at low frequencies (high temperatures), resulting in greater rutting resistance. This reinforces the need to adjust the mixing temperature and the mixing and conditioning time of these materials so that, once incorporated into an asphalt mixture, more effective homogenization is achieved, improving the performance of the resulting material.
- Analyzing each material in isolation, based on the known properties of each binder under a specific condition, shows that once materials with similar behavior but different aging and oxidation states are blended, a structural change occurs in the blend, leading to a performance improvement — reflected in the change in viscosity and the gain in recovery that the recycling agent produces in the binder blends.
- Temperature is a crucial factor in achieving complete blending between the binders, making it a boundary condition for assessing the integrity of the blends: the lower the mixing, conditioning, and in-service temperature, the greater the risk of premature cracking and the lower the resulting integrity.

More detailed analyses involving different types and contents of recycling agents, as well as different aging stages of the remaining RAP binder, can therefore be considered topics for future studies. It is believed, however, that the characterization of binders for use in recycled asphalt mixtures should take into account the blending parameters, and not just those of the incorporated virgin binder alone.

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