Civil Engineering Infrastructures Journal xx,xx
DOI: 10.22059/ceij.2025.380627.2185

Rheological Study of Asphalt Modified with Recycled Tire Rubber Under Different Aging Conditions

#	Name	Email Address	Degree	Position	Phone	Mobile	Country	Affiliation	
1	Zapata Ferrero, Ignacio	izapata@frlp.utn.edu.ar	Ph.D.	Professor	2215552540	2215552540	Argentina	LEMaC Centro de Investigaciones Viales UTN FRLP CIC PBA	
2	Gelsi, Agustina	agelsi@alu.frlp.utn.edu.ar	Other	Assistant Professor	+542217777777		Argentina	LEMaC Centro de Investigaciones Viales UTN FRLP - CIC PBA	
3	Cabrera, Pablo	pcabrera@frlp.utn.edu.ar	Ph.D. Candidate	Assistant Professor	+542214951990		Argentina	LEMaC Centro de Investigaciones Viales UTN FRLP - CIC PBA	

#	Name	Email Address	Degree	Position	Phone	Mobile	Country	Affiliation	
4	Rivera, Julián	jrivera@frlp.utn.edu.ar	Ph.D.	Professor	+542215555555		Argentina	LEMaC Centro de Investigaciones Viales UTN FRLP - CIC PBA	
5	Botasso, Gerardo	gbotasso@frlp.utn.edu.ar	Ph.D.	Professor	+542216666666		Argentina	LEMaC Centro de Investigaciones Viales UTN FRLP - CIC PBA	

Received: 20/11/2024

Revised: 19/03/2025

Accepted: 07/05/2025

ABSTRACT

The asphalt used in the road industry must possess the necessary properties to absorb the deformations generated by the pavement under traffic. In this regard, the major issue observed is oxidation caused by the loss of volatile compounds. This results from exposure to high temperatures during the mixing and production of the asphalt mixture and, on the other hand, from progressive deterioration due to environmental conditions while in service. The reduction of its lighter compounds leads to cracking and deterioration of the asphalt layer.

Furthermore, waste generation remains an unresolved issue. Recycled Tire Rubber (RTR) from the automotive industry, which has reached the end of its life, has become common as an asphalt modifier in high proportions (percentages exceeding 10 %).
This study aims to evaluate asphalt modified with RTR in increasing proportions and analyze the samples' behavior under short-term and long-term aging conditions to consider the material's evolution. In the results obtained, the influence of the aging of the samples can be observed as the proportion of RTR is increased according to the results of FTIR and complex shear modulus. An increase in the softening point and the fatigue parameter is also observed with the addition of the residue.

Keywords
Rheology, RTR, Asphalt, Aging, Enviromental conditions

1. Introduction

Hot mix asphalt is composed of a combination of different materials, such as conventional or modified asphalt, aggregates, and fillers. The properties of these mixes are directly related to the rheological behavior of the asphalt binder. To optimize certain physical, chemical, and rheological properties of conventional asphalt binders, additives are used to create modified asphalt binders (Botasso & Segura, 2013; Gallego, 2018). Various modifiers for base asphalt include hydrocarbons, mineral fillers, and polymers. Polymers are more frequently used because they reduce the thermal susceptibility of the binder, increase internal cohesion, adhesion, and elasticity, resulting in mixes with greater resistance to fatigue and permanent deformation (Chavarria Salas, 2005, Panda & Mazumdar, 1999).

1.1 Recycled Tire Rubber

Additionally, due to the large amount of waste generated, it is necessary to include waste materials. One such material is Recycled Tire Rubber (RTR), which is suitable for use in the asphalt paving industry as a modifier for the base asphalt in the mix. This material can be incorporated into asphalt to achieve the performance of asphalt modified with virgin polymers or, at the very least, to closely match those characteristics (Botasso & Segura, 2013; SIGNUS, 2017).

Tires are composed of various materials such as rubber, chemicals, textiles, steel, and other metals. Currently, used tires represent only between 0.5% and 1% of global waste, but their components have a long degradation time, taking years to centuries to decompose. In Argentina alone, more than 150,000 tons of tires are discarded every year. Consequently, reusing these materials is crucial. Adding RTR to asphalt mixes not only improves their properties but also provides environmental benefits by reintegrating RTR into the production of another material, substituting virgin materials with those that have already been used. The main advantages of RTR modified asphalt mixtures are the benefits against permanent deformations, cracking at low temperatures and improvement against aging (Bahia & Davies, 1994, Guo et al., 2024, Khadim & Al-Mosawe, 2024, Kök & Çolak, 2011, Nanjegowda & Biligiri, 2023, Lee et al., 2008, Soleymani et al., 2004, Wang et al., 2020, Wang et al., 2022.)

The incorporation of RTR powder into asphalt mixes is carried out using three methodologies: dry technology, used tire powder is incorporated into the asphalt mix as an aggregate. In this way, the digestion times considered appropriate for the asphalt modification to occur must be met. The process was initially patented in Sweden and consists of adding RTR particles between 0 and 2 mm to replace between 1 and 3% of the mass of the fine aggregate (Chavez et al., 2019).

For the modification of asphalt mixes with rubber by dry process, RTR powder in a size smaller than 0.5 mm is used. Special care must be taken with the incorporation of used tire powder considering the digestion times in the mixing drum of the asphalt plant, the total mixing time and the waiting time in the asphalt mix transport truck. In recent years, this technology has fallen into disuse. Some results below the requirements of asphalt mixtures and excessive rubber digestion times to achieve the modification of asphalt cement make this methodology not very recommendable (Botasso & Segura, 2013).

The wet technology consists of modifying the asphalt cement instead of adding RTR powder to the asphalt mixture. In this case, the process consists of dispersing the asphalt and rubber at a temperature between 185 and 195 ºC, for a time that ensures digestion for at least 60 minutes. The most important factors in this process are temperature, energy and mixing time, and the rubber particle size. By using modified asphalt, the beneficial properties obtained are transferred to the behavior of the asphalt mixture. Like the dry process, the higher viscosity of asphalt with the rubber aggregate requires higher mixing temperatures than conventional asphalts to ensure the correct wrapping of the aggregates. The greatest disadvantage of the wet process is the possible separation of the phases between rubber and asphalt. This problem is intensified about the higher rates of rubber incorporated into the asphalt. Faced with these problems, one of the solutions is to reduce the distances for transporting asphalt cement to the asphalt mixing plant, allowing for a time for the trucks to arrive so that there is no separation between the materials. Another alternative is the production of modified asphalt cement directly at the plant. (Bahia & Davies, 1994, Gallego, 2018).

The latest trend in the incorporation of RTR powder as an additive in asphalt mixes is in the form of pre-digested material. This technology consists of dispersing and grinding the RTR powder with additives and generating a type of filler that can be incorporated directly into the asphalt concrete mix. One of the most relevant benefits of this technology lies in the shorter digestion times required by this additive, having been previously treated. On the other hand, having the fineness and appearance of a filler, the handling and incorporation into the asphalt mixes is similar to the latter, being able to opt for a pneumatic type of addition. Properties of asphalts modified with RTR powder show superior performance compared to conventional unmodified asphalts, and competitive behavior compared to asphalts modified with virgin polymers (Gallego, 2018).

The proportions of incorporation of these materials always refer to the percentage by weight of 100% of the asphalt. Various studies have shown that asphalts improved with RTR, in low proportions (around 4%) or medium (around 12%), improve both penetration and softening point compared to the original asphalt, and also show a significant increase in viscosity. This results in a material that is less susceptible to climatic factors, such as high temperatures experienced in the summer (Botasso & Segura, 2013). Studies are also being conducted on SMA (Stone Mastic Asphalt) type mixtures with the incorporation of RTR and glass fibers (Morea et al., 2023).

Yu et al. (2023) studied the application of RTR in micro and nanometric sizes to avoid the problems associated with adding large proportions, which lead to storage stability issues. Through fragility tests and scanning calorimetry, it was determined that the incorporation of this addition offers benefits concerning cracking at low temperatures. Additionally, measurements performed using a dynamic shear rheometer showed that the material becomes more elastic at high temperatures.

Alsheyab et al. (2023) compiled studies on the incorporation of RTR into asphalt and asphalt mixtures. Their analysis showed that the addition of this material increases the softening point and viscosity. Furthermore, reductions in penetration, ductility, and flash point were observed.

In Spain, the SIGNUS guide presents the necessary parameters for incorporating RTR into asphalt. Specifications were also developed to classify asphalt cements modified with RTR according to the percentage of waste added. This guide proposes, in a novel way, the classification of improved, modified, and high-viscosity asphalts with used tire dust. In a second stage, details the requirements for preparing asphalt mixtures of different types, made with modified asphalt (SIGNUS, 2017).

Apaza et al. (2024a) incorporated used tires into an asphalt mixture to evaluate the sound absorption of these pavements. Mixtures with 0, 0.75, and 1.50% RTR in the mix were prepared using the dry process. An influence of incorporation was found at temperatures ranging from 10 to 60 °C when the mix contains high void contents. Soengas et al. (2022) studied discontinuous asphalt concrete with RTR incorporation and determined higher sound absorption values compared to a standard sample across a range of 15 analyzed frequencies. On the other hand, the incorporation of RTR in asphalt mixtures provides improved macro- and microtexture to the considered pavement. In a continuation of the aforementioned work, Apaza et al. (2024b) studied the influence of RTR in different types of asphalt mixtures with increasing RTR contents. It was found that residue incorporation had a considerable influence on SMA and open-type mixtures, but not on semi-dense mixtures. Botasso & Segura (2013) studied modification with 8% RTR in discontinuous surface mixtures, evaluating the macro- and microtexture of the samples before and after abrasive testing using the wheel-following test. Favorable results were obtained in the evaluation of the macrotexture of the mixtures with RTR compared to those modified with polymer.

On the other hand, when incorporating other additives, Rodríguez Alloza et al. (2014) evaluated the incorporation of viscosity-reducing additives to reduce the temperature in asphalt mixtures containing 20% RTR based on the weight of the asphalt. The mixtures obtained through this process experienced a decrease in the viscosity of the binder used, but the stiffness of the material increased, resulting in a mixture susceptible to fatigue failure. On the other hand, Gallego et al. (2020) evaluated different forms of compaction of SMA-type mixtures with the incorporation of RTR and an additive to reduce the temperature of the mixtures. They found that the best results were found when evaluating using a rotary compactor versus static compaction. Sol-Sanchez et al. (2020) studied the incorporation of RTR in asphalt mixtures by applying high-penetration asphalt using two temperature-reducing additives. Acceptable results were obtained at temperatures of 135°C without compromising the mechanical properties of the asphalt mixture. The rheological and environmental properties of the mixtures were improved with 15% to 20% RTR combined with two viscosity-reducing additives. The addition of the temperature-reducing additive was observed to slightly reduce the fatigue strength of the mixtures (Pouranian et al., 2020). Another waste material that can be incorporated into the asphalt mix is blast furnace slag. A study showed that fine aggregate replacement percentages of 0 to 25% yielded the best results in terms of permanent deformation, indirect tensile strength, and stability. Based on the results obtained, it was found that a 20% replacement rate yielded the best results (Chaubey et al., 2024).

1.2. Aging in Asphalt Binder

The oxidation process in asphalt mixtures occurs at both high and low temperatures, but with distinct processes. At high temperatures, asphalt behaves like a viscous material, making it more susceptible to permanent structural deformations. The internal energy the material endures to resist loads during each cycle can be dissipated in the form of molecular friction, i.e., heating, or through the deformation of molecular bonds (Tauste et al., 2018).

In the case of the process at low temperatures, asphalt behaves like a solid, in which, when loads are applied, the resulting deformations manifest themselves in the short term. At these temperatures, the maltene fraction of asphalt exhibits less molecular motion and forms more rigid networks. The mechanism by which these fractions function is manifested as stretching and bending of intermolecular bonds until fracture occurs. Due to these circumstances, in contrast to high-temperature behavior, considerable permanent deformations are not generated, and therefore, in each load application cycle, the energy is dissipated through material fatigue (Moreno Navarro et al., 2018).

Other issues also affect aging, such as the ratio of asphaltenes and maltenes in the chemical composition of asphalt. Generally speaking, the aging process of asphalt manifests itself through the interaction of oxygen and ultraviolet radiation during the asphalt mixture production process and its service life. In both stages, a loss of volatile occurs, leading to hardening and oxidation (Read & Whiteoak, 2003).

Regarding chemical composition, the asphaltene fraction undergoes a small increase during the production of asphalt concrete and a smaller increase over time. On the other hand, the resin and aromatic fractions undergo a noticeable decrease during the manufacturing and mixing process. As with asphaltenes, the resins and aromatics undergo slight changes after the placement and finishing processes, and their serviceability after several years (Read & Whiteoak, 2003).

The presence of RTR within the asphalt implies a material with more elastic rheological behavior, for which reason it is expected that the behavior with large additions will improve the fatigue resistance performance of the asphalt. The present investigation aims to characterize asphalts with 15 %, 18 %, 20 %, 22 % and 25% RTR by weight of asphalt combined with the base asphalt, through rheological tests such as the master curve and SUPERPAVE parameters to assess fatigue. Various conditioning methods, including short-term, medium-term, and long-term aging, are also proposed.

2. Materiales

The Recycled Tire Rubber (RTR) samples observed under the microscope exhibit a heterogeneity in particle sizes and shapes within the analyzed sample can be seen in Figu. Elongated and rounded particles are observed in roughly equal proportions. Additionally, the sample shows a completely irregular surface with torn material features, typical of the crushing process that the material undergoes. These characteristics suggest better interaction between the rubber particles and the asphalt, due to the larger specific surface area of the particles.

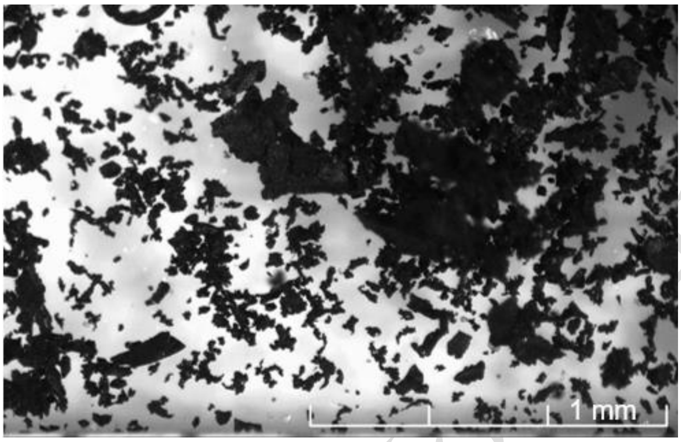

Fig. 1. Recycled Tire Rubber in microscope

The source material is found in the city of Buenos Aires, which is collected from neighboring cities. It is subjected to a mechanical crushing process to obtain tire dust. For the characterization of the RTR powder's particle size, a sample is subjected to a series of sieves to find the cumulative percentage of each material fraction that passes through. The particle size distributions used correspond to the C1 curves from the CEDEX manual (CEDEX, 2013), as shown in Table 1.

Table 1. Particle size distribution of Recycled Tire Rubber

	Amunt passing (%)		
Sieve	**C1 Sup.**	**Curve**	**C1 Inf.**
2000 µm (N°10)	100	100.00	100
1680 µm (N°12)	100	100.00	100
1410 µm (N°14)	100	100.00	100
707 µm (N°25)	50	84.00	85
500 µm (N°35)	10	48.61	80
250 µm (N°60)	5	8.22	70
125 µm (N°120)	0	0.47	30
63 µm (N°230)	0	0.45	15
53 µm (N°270)	0	0.31	7

The RTR used in asphalt mixtures requires the most extensive grinding process to obtain the finest possible material. In summary, the sample analyzed has more than 85% of the material passing the N° 25 sieve and 80% is between 0.15 and 0.80 mm in average diameter. Figure 2 shows the granulometric curve of the sample.

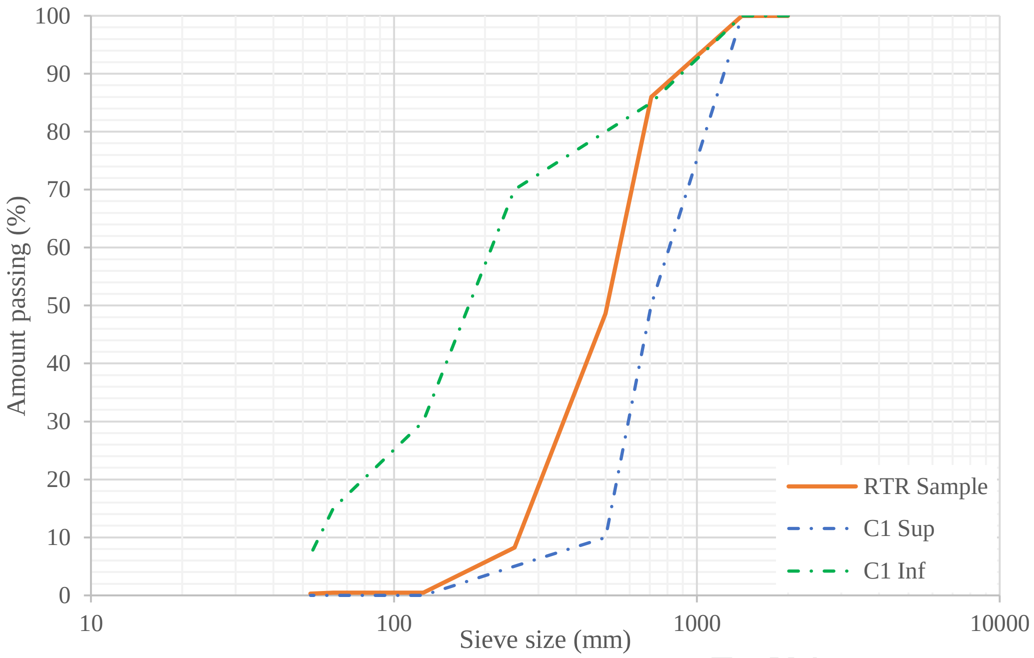

Fig. 2. Recycled Tire Rubber granulometric curve

An asphalt binder denominated as CA-30 is used as the base. Increasing amounts of Recycled Tire Rubber (RTR) are added to the samples for evaluation, at 15%, 18%, 20%, 22%, and 25% by weight of the asphalt whose designation is CA30, 15RTR, 18RTR, 20RTR, 22RTR, and 25RTR respectively. RTR percentages are selected based on the consulted bibliography, adding large quantities of recycled tires. The properties of the samples can be seen in Table 2.

Table 2. Characterization of samples

Property	Standard	CA30	15NFU	18NFU	20NFU	22NFU	25NFU
ORIGINAL							
Softening point (°C)	ASTM D36-20	53	59	61	62	65	68
Penetration (25°C, 0.1 mm)	ASTM D5-20	39	38	34	32	23	21
Torsional elastic recovery (%)	ATM-122-22	-	47.8	56.1	63.3	71.7	78.7
Rotational Viscosity (cP) [135 °C-0,9RPM S21]	ASTM D4402-23	-	4111	5166	7611	11277	16777
Rotational Viscosity (cP) [170 °C-0,9RPM S21]	ASTM D4402-23	-	575	804	1212	1304	2617
After RTFOT							
Variation of weight (%)	ASTM D2872-22	0.20	0.18	0.15	0.15	0.20	0.21
Softening point (°C)	ASTM D36-20	58	64	67	67	70	71

For the preparation of the different samples, the dispersion of RTR powder in the asphalt is carried out. The dispersions follow the LEMaC-A02/16 test methodology (LEMaC, 2019). The procedure involves heating the asphalt to a temperature between 160°C and 170°C, using a double-walled container filled with oil according to Segura et al. (2023). Once the sample reaches the desired temperature, it is poured into the container, and the disperser is lowered, ensuring it is 2 cm above the bottom of the container. The disperser is then turned on, and the revolutions are progressively increased to the maximum speed (approximately 5,000 rpm). The calculated amount of RTR is added within a maximum time of 20 minutes. Care must be taken to avoid saturating the surface of the container and to allow sufficient time for the equipment to disperse the RTR. Oscillatory movements of the disperser are made to ensure the RTR is thoroughly dispersed throughout the asphalt mass. The dispersion process continues for 60 to 75 minutes, during which the asphalt temperature is progressively monitored to ensure it does not exceed 190°C.

3. Methods

3.1 Aging in Rolling Thin Film Oven (RTFOT)

The first conditioning of the asphalt binder samples is performed in a Rolling Thin Film Oven (RTFOT). This aging simulates the conditions that asphalt undergoes during the production of asphalt mixes, aiming to replicate the loss of volatiles and oxidation during the mixing of aggregates. For the test, 38 g of each sample is prepared in glass jars, placed on a horizontal surface for one hour. After this time, the jars are placed in an oven previously heated to 163°C. Simultaneously, air is blown into the oven inside each container. The total test duration is 83 minutes. After the test, the glass containers are emptied for further characterization tests (AASHTO, 2022).

3.2 Aging in Ultraviolet (UV) Chamber

Following the RTFOT aging, the samples are prepared for testing under UV aging conditions according to ASTM G154, as shown in Figure 2. This type of aging simulates the radiation conditions that the pavement experiences during its service life. The asphalt aging is carried out for 810 hours. To calculate the simulation time, a UV radiation of 50 W/m² from the lamps inside the chamber is used. The average radiation per day in La Plata is 4 kW/m². The conditioning process is similar to that proposed by Liu et al. (2022) and Briliak & Remisova (2022). Data are obtained from the Department of Seismology and Meteorological Information, La Plata Observatory FCAG.

Fig. 2. Ultraviolet test chamber

If ultraviolet radiation represents 1% of the total emitted radiation, the UV chamber aging time corresponds to 2.5 years. It is important to note that aging during this stage represents medium-term aging.

After conditioning, the samples show notable surface differences. The base CA30 sample appears the most deteriorated, showing streaks across the surface. The 20RTR sample shows a significant improvement compared to the base sample, with marks only visible at the edges. Finally, the 25RTR sample is the least affected, with a surface similar to that before conditioning. Although this is a visual assessment, it highlights the benefit of incorporating RTR in reducing aging effects. The visual appearance shown in Figure 3 of the samples after conditioning is consistent with that observed by Song et al. (2023).

Fig. 3. Samples after UV aging. CA30 (left), 20RTR, (center), and 25RTR (right)

3.3 Aging in Pressure Aging Vessel (PAV)

As an alternative to UV chamber aging, aging is carried out in a Pressure Aging Vessel (PAV). This aging simulates the conditions that the pavement is subjected to during its service life over a period of 5 to 10 years. The test involves placing 50 grams of each asphalt sample in trays inside the PAV. Once placed, a temperature of 100°C and a pressure of 2.1 MPa are applied. The total test duration ranges from 20 to 22 hours, depending on the equipment's pressure drop requirements (AASHTO, 2022).

3.4 Softening Point

This test is mainly used to determine the susceptibility of a material to temperature variations. The method consists of filling two rings with the sample to be evaluated, and once placed in a container of water, the temperature is progressively raised. The final test temperature is recorded when the balls inside the rings with the sample make contact with the lower plate. (ASTM, 2020).

3.5 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) with Attenuated Total Reflectance (ATR) using a diamond tip is employed to identify the wavelengths of functional groups in each sample. The equipment used is the IRAffinity-1 by SHIMADZU. The detection range covers wavelengths from 3.500 cm^{-1} to 500 cm^{-1}. For the analysis, a small portion of each prepared sample is extracted and placed in the equipment for study. Finally, the peaks corresponding to the functional groups detected in the spectrum are analyzed.

3.6 Frequency Sweep – Master Curve

For rheological characterization and analysis of the influence of loading frequency, temperature, and RTR content, a frequency sweep is performed on a dynamic shear rheometer. The samples are subjected to dynamic oscillatory stress while the equipment records the resulting deformations and applied torque. A frequency range of 0.15 Hz to 15 Hz and a temperature range of 35°C to 75°C are applied. The rheological characterization of the asphalts is performed using a Discovery HR-2 Hybrid Rheometer DSR from TA Instruments. The 25 mm geometry is used in the parallel plate configuration, as shown in the figure below. Using the software provided by the manufacturer (TRIOS), the complex shear modulus and phase angle are recorded according to AASHTO T315.

Using the time-temperature superposition principle, the master curves of the complex shear modulus are obtained as a function of the reduced frequency for the samples studied. Obtaining the master curves allows for the analysis of test frequencies that are difficult to measure in practice due to the technological limitations of existing measurement equipment. To construct the master curves, after the frequency sweep test, one of the test temperatures is chosen, with the average temperature being recommended. The modulus data are then shifted horizontally until the results align in a single sinusoidal curve. Analytically, the frequency shift factor can be obtained. The correctly aligned data are then fitted to a Sigmoidal equation, as represented by Equation 1.

$$log|G^*| = \alpha + \frac{\beta}{1 + e^{[\gamma + \delta * \log(f_R)]}} \qquad 1$$

Where:
f_R = Reduced frequency (Hz)
α, β, δ, γ = parameters of the sigmoidal model

To obtain the shift factor (aT) for the complex shear modulus data and the master curve, the William-Landel-Ferry model is applied. The equation of William Landel Ferry is shown in Equation 2.

$$log(aT) = \frac{-C_1(T - T_R)}{C_2 + T - T_R} \qquad 2$$

Where:
C_1, C_2 = Material constants (adimensional)
T = Test temperature (°C)
T_R = Reference temperature (°C)

3.7 SHRP – SUPERPAVE Parameter

The SUPERPAVE parameter, G*sinδ, is estimated to use the dynamic shear rheometer. This parameter is not determined in this study using a temperature scale until the failure point proposed by the specifications (corresponding to 5 MPa) is reached, but instead is evaluated at a defined temperature of 20°C. This allows for a comparative basis between the different modified asphalts and their corresponding aging states. For the test, an oscillatory stress of 10 rad/sec, equivalent to 1.59 Hz, is applied, and both the complex shear modulus (G*) and phase angle (δ) are recorded (AASHTO, 2010).

4. Results and discussion

4.1 Softening point

As shown in Figure 3, the most significant impacts are observed in the aging of unmodified asphalt. For samples with 18% RTR to 25% RTR, the effect of UV exposure is almost negligible. Samples from PAV show a considerable increase in aging, making it the most aggressive test for asphalt. It can be inferred that a higher percentage of RTR in the dispersions offers better protection against this type of aging. A quantity of three replicates were made for the test.

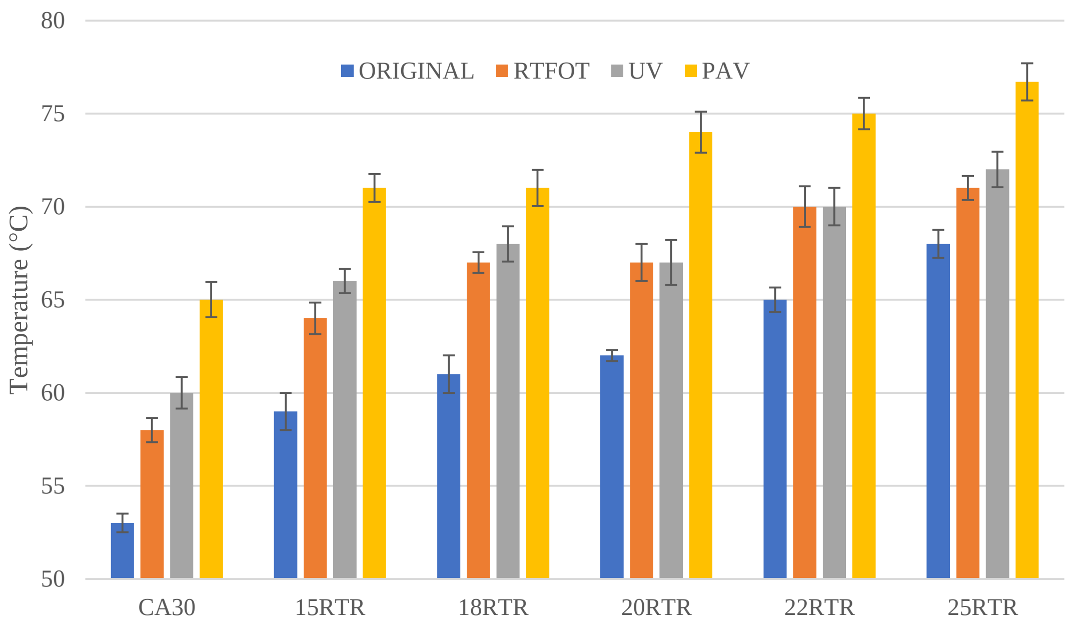

Fig. 4. Softening point of the evaluated asphalts

Table 3 shows the percentage variations of the aging groups compared to Original group. The improvement in asphalts with increasing RTR content in response to aging is evident. The greatest benefit is observed in PAV group, where the variation decreases from 22.6% to 20.3% with the initial percentage of RTR incorporation, ultimately reaching 12.8% for the 25RTR sample. This analysis indicates a lower susceptibility to changes in this parameter in response to the aging conditions tested.

Table 3. Percentage increase in Softening Point compared to Original group.

	Percentage variation (%)		
	RTFOT	UV	PAV
CA30	9.4	13.2	22.6

15RTR	8.5	11.9	20.3
18RTR	9.8	11.5	16.4
20RTR	8.1	8.1	19.4
22RTR	7.7	7.7	15.4
25RTR	4.4	5.9	12.8

4.2 Fourier Transform Infrared Spectroscopy (FTIR)

The spectra obtained from the analyzed samples can be observed in Figures 5 to 10, in which the wavelengths of the asphalts of all groups are displayed.

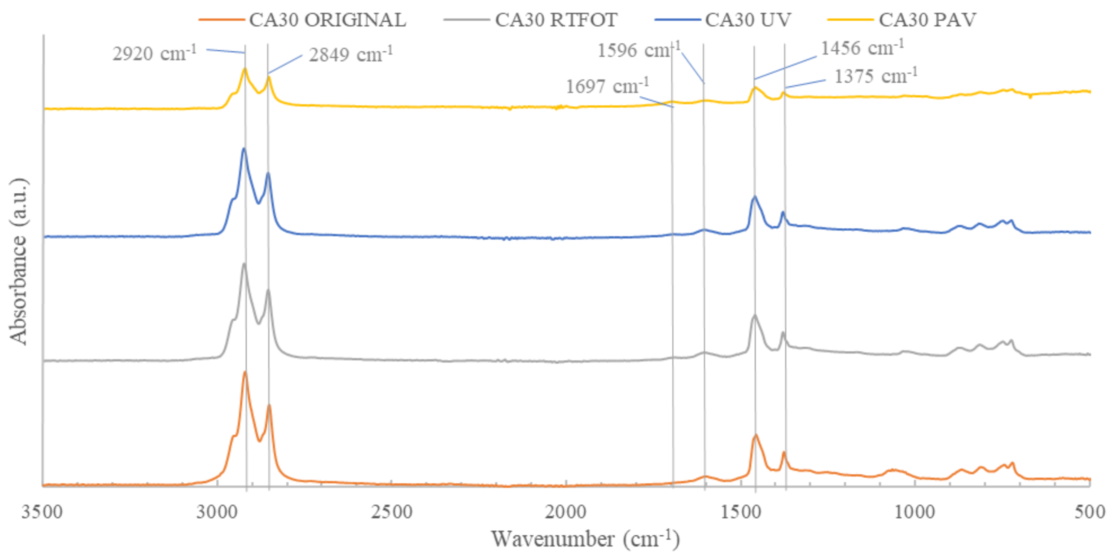

Fig. 5. FTIR spectrum of the CA30 sample

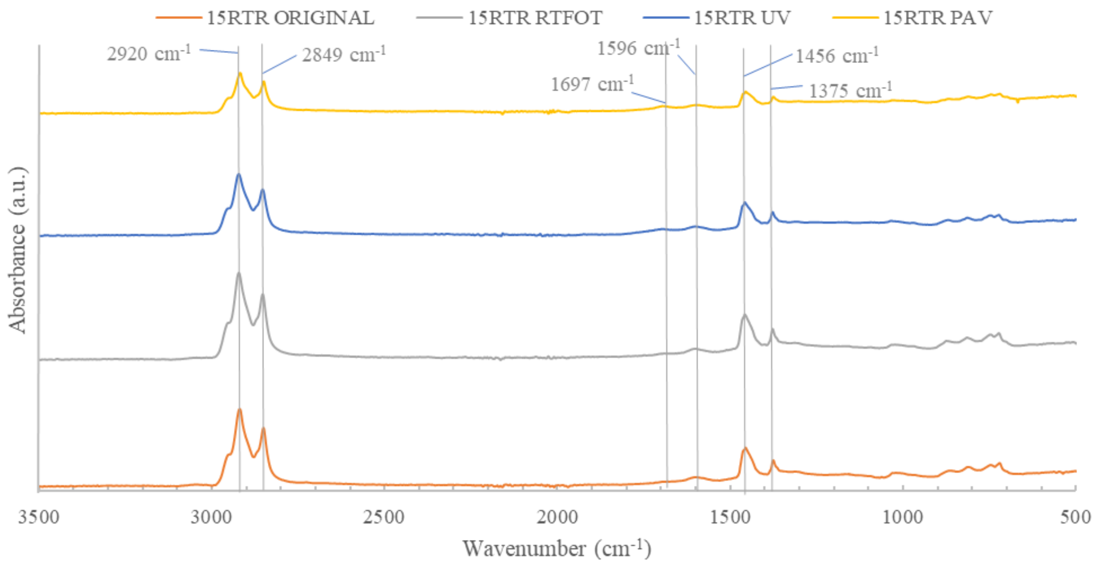

Fig. 6. FTIR spectrum of the 15RTR sample

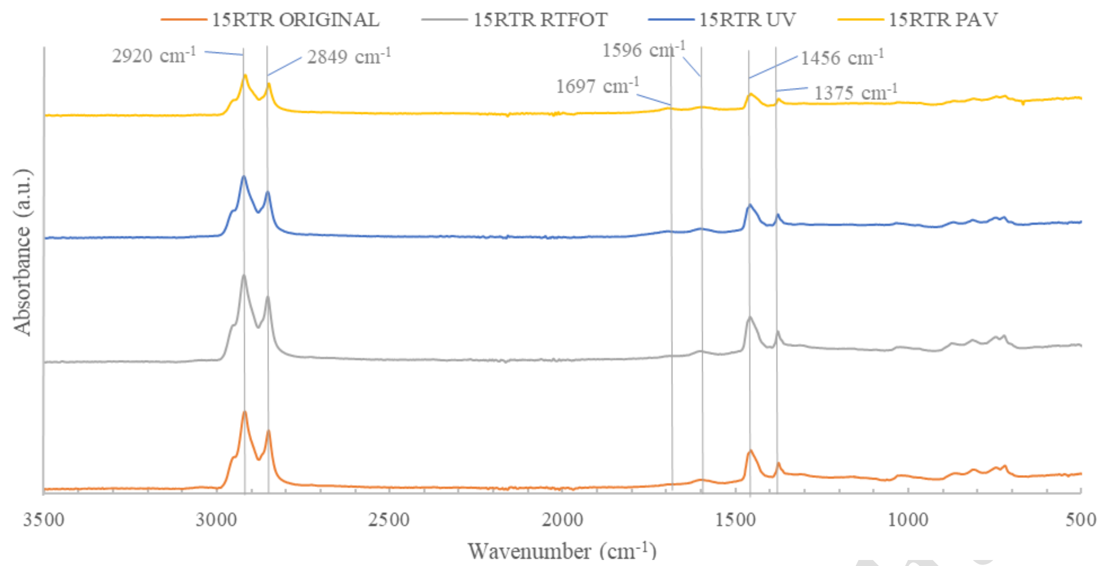

Fig. 7. FTIR spectrum of the 18RTR sample

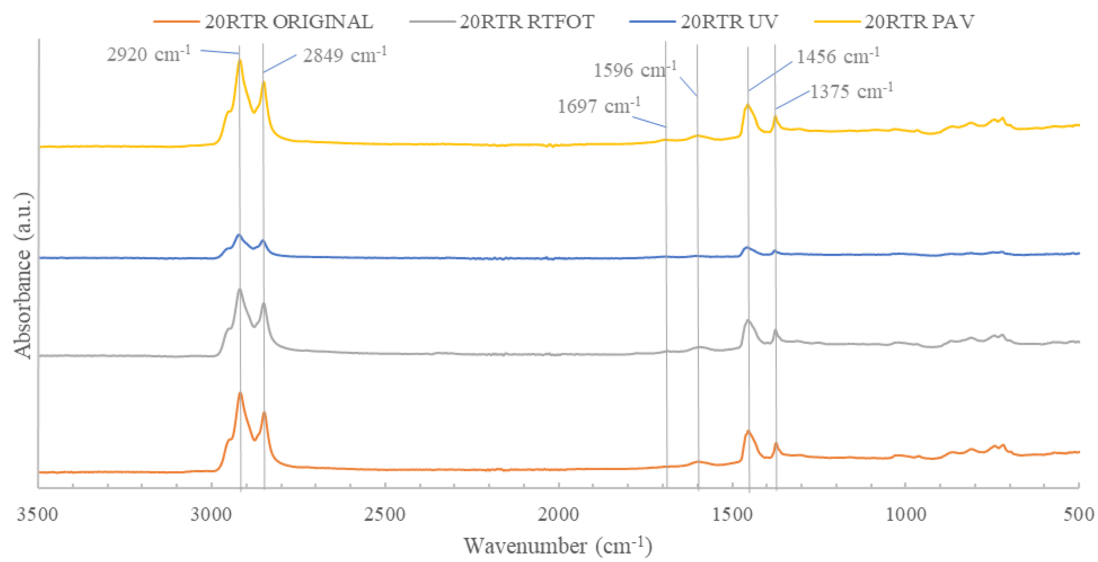

Fig. 8. FTIR spectrum of the 20RTR sample

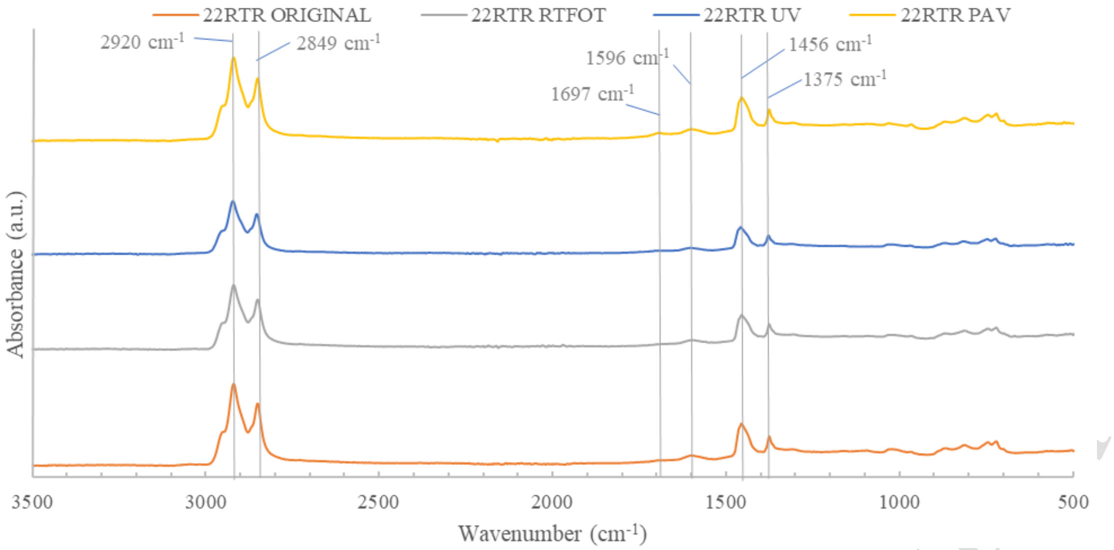

Fig. 9. FTIR spectrum of the 22RTR sample

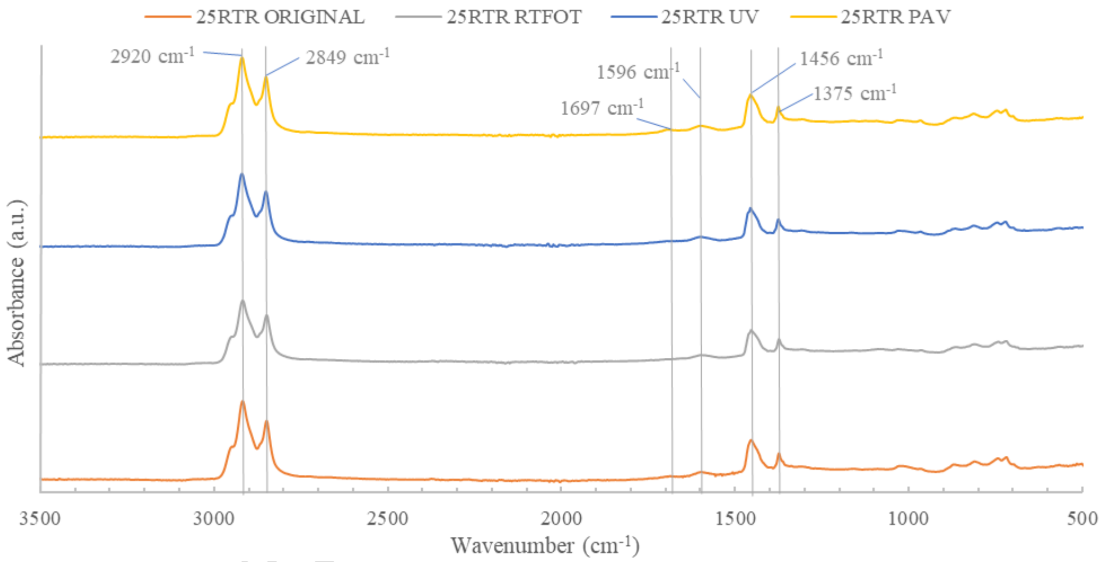

Fig. 10. FTIR spectrum of the 25RTR sample

The analysis is performed for the 1697 cm^{-1} band, which identifies carbonyl groups and indicates the aging process caused by asphalt oxidation. As shown in Table 4, the base sample exhibits the lowest values for Original, RTFOT, and UV groups when compared to the asphalts modified with RTR. This is due to the fact that the modified asphalts undergo a heating process during dispersion. However, a progressive reduction in the variation between PAV and Original groups is observed as RTR is incorporated, with the value halving for the first asphalt percentage. These results suggest a notable improvement in the aging resistance of the modified asphalts. Similar results are stated by Chang et al. (2020) and Cortés et al. (2010).

Table 4. Analysis of the variation of the 1697 cm^{-1} band.

	CA30	15RTR	18RTR	20RTR	22RTR	25RTR
ORIGINAL (cm^{-1})	0.0126	0.0269	0.0458	0.0697	0.0550	0.0504

RTFOT (cm^{-1})	0.0148	0.0398	0.0710	0.0837	0.0782	0.0548
Variation (cm^{-1})	0.0022	0.0129	0.0252	0.0140	0.0231	0.0044
UV (cm^{-1})	0.0153	0.0421	0.0341	0.0354	0.0287	0.0627
Variation (cm^{-1})	0.0028	0.0153	-0.0117	-0.0344	-0.0263	0.0123
PAV (cm^{-1})	0.0401	0.0401	0.0480	0.0554	0.0565	0.0499
Variation (cm^{-1})	0.0275	0.0132	0.0022	-0.0144	0.0015	-0.0005

4.3 Frequency Sweep – Master Curve

Figures 11 to 16 present the master curves obtained from the samples, showing the complex shear modulus across a frequency range of 0.001 Hz to 100 Hz. The master curves were obtained at a reference temperature of 55°C.

As seen in Figures 11 to 16, there is a significant increase in the complex shear modulus values in the master curves as the percentage of RTR powder incorporated increases. The greatest increase is observed between the base asphalt and the modified asphalts with lower percentages of RTR. This trend is most noticeable at lower frequencies (around 0.001 Hz to 0.1 Hz), where higher resistance is provided against the passage of slower vehicles. Conversely, at application frequencies above 1 Hz, the unmodified binder shows the most rigid response. It is worth noting that the greatest benefit in modulus increase at low frequencies comes from the initial level of RTR incorporation (15%).

In all samples, no particular deterioration is observed with the progression of the proposed aging conditions, which demonstrates good performance under simulated mixing and service conditions.

The CA30 sample exhibits similar behavior under Original, RTFOT, and UV conditions, but not under PAV condition, where higher moduli and a more rigid asphalt are observed due to the loss of volatiles caused by aging. The 15RTR sample follows a similar trend, but for PAV condition, the results are closer to those of Original, RTFOT, and UV group. For the 18RTR, 20RTR, 22RTR, and 25RTR samples, the master curves are closer among the proposed conditioning conditions, indicating a more stable material in the face of various aging conditions. This analysis can be seen in Figure 17, which shows the variation in aging between the CA30 and 25RTR samples. The growth of the modulus of the 25RTR sample for CA30 can be observed. On the other hand, a smaller distance is observed between the most aggressive aging (PAV) and the rest of the conditionings for the 25NFU sample, while the PAV group is four times larger than the Original sample of CA30.

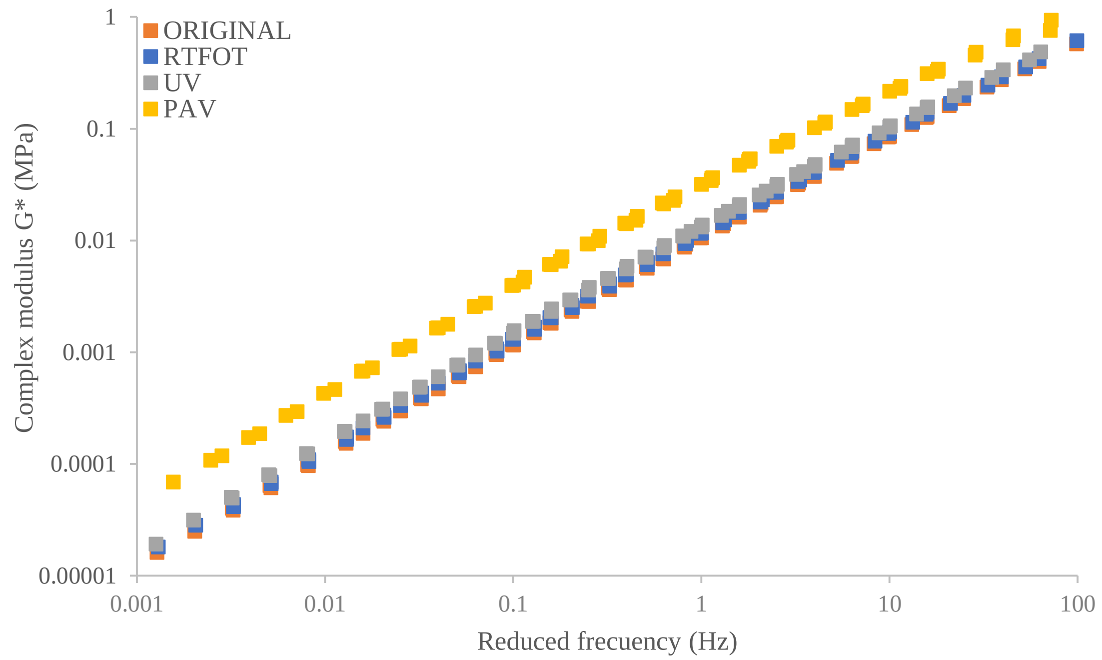

Fig. 11. Master curves of Complex Modulus for CA30 ($T°_{ref}$ = 55 °C)

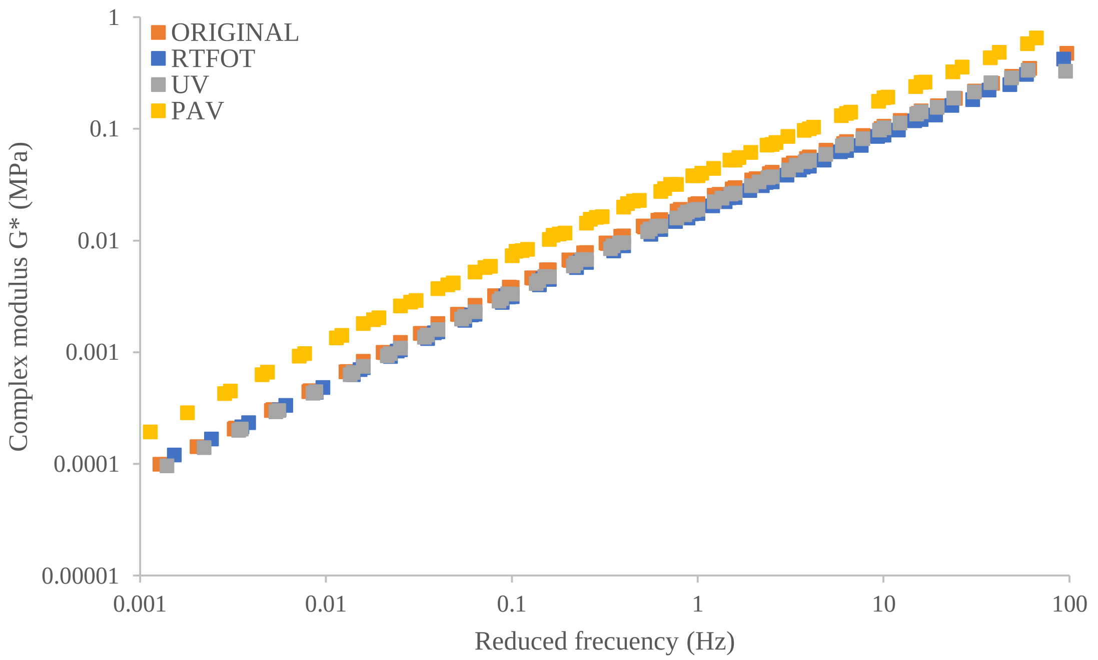

Fig. 12. Master curves of Complex Modulus for 15RTR ($T°_{ref}$ = 55 °C)

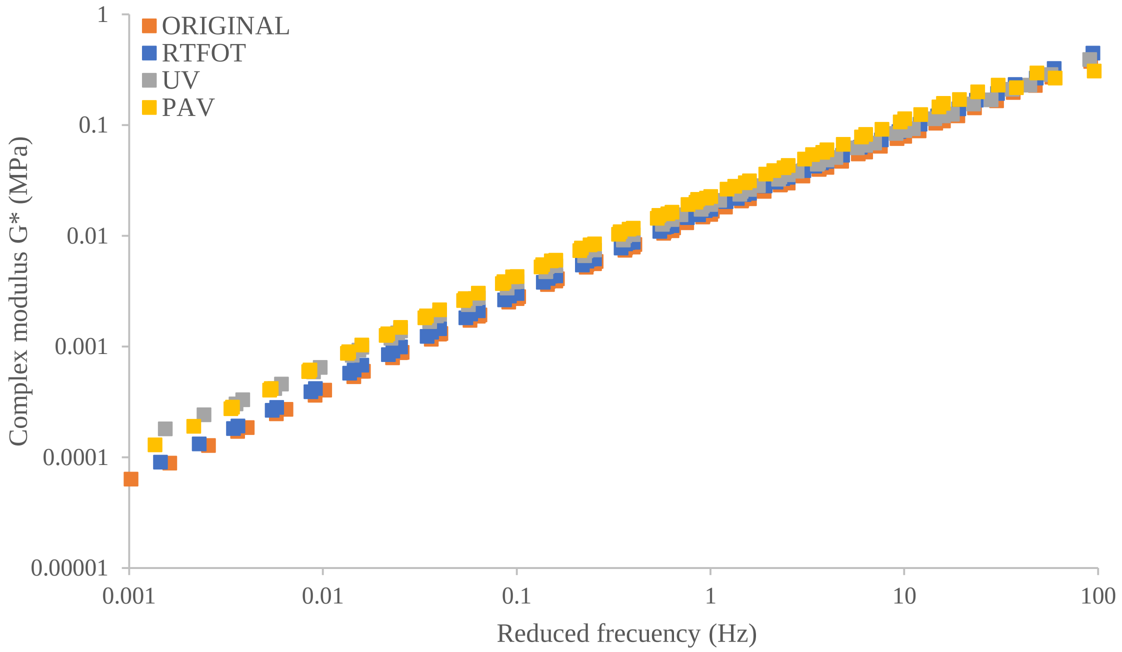

Fig. 13. Master curves of Complex Modulus for 18RTR (T°$_{ref}$ = 55 °C)

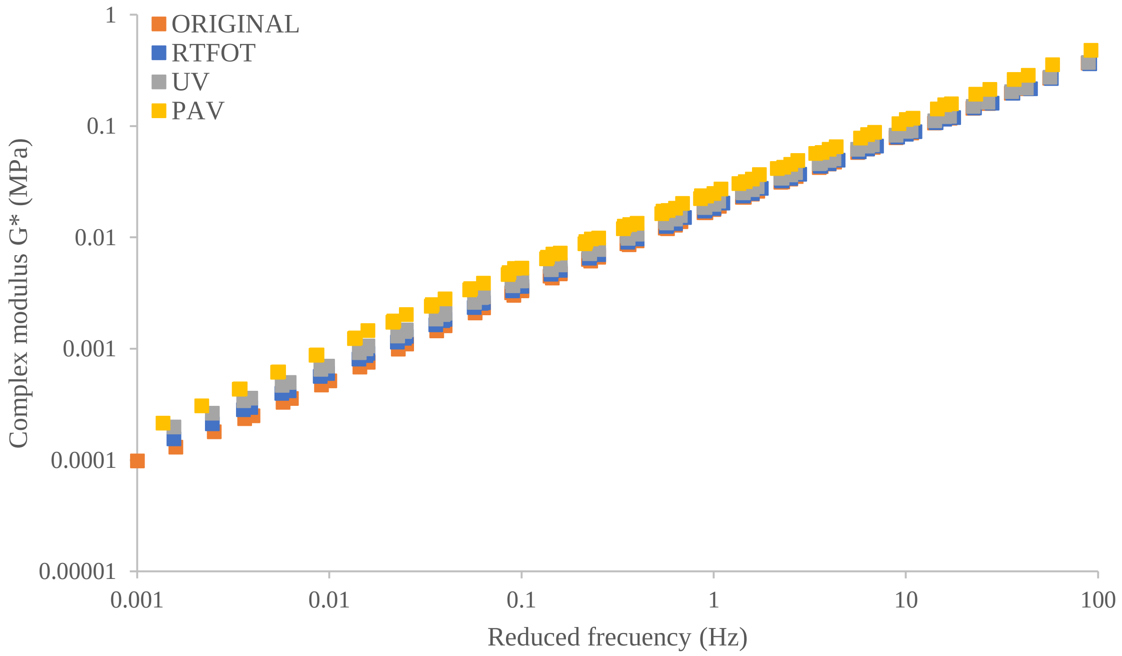

Fig. 14. Master curves of Complex Modulus for para 20RTR (T°$_{ref}$ = 55 °C)

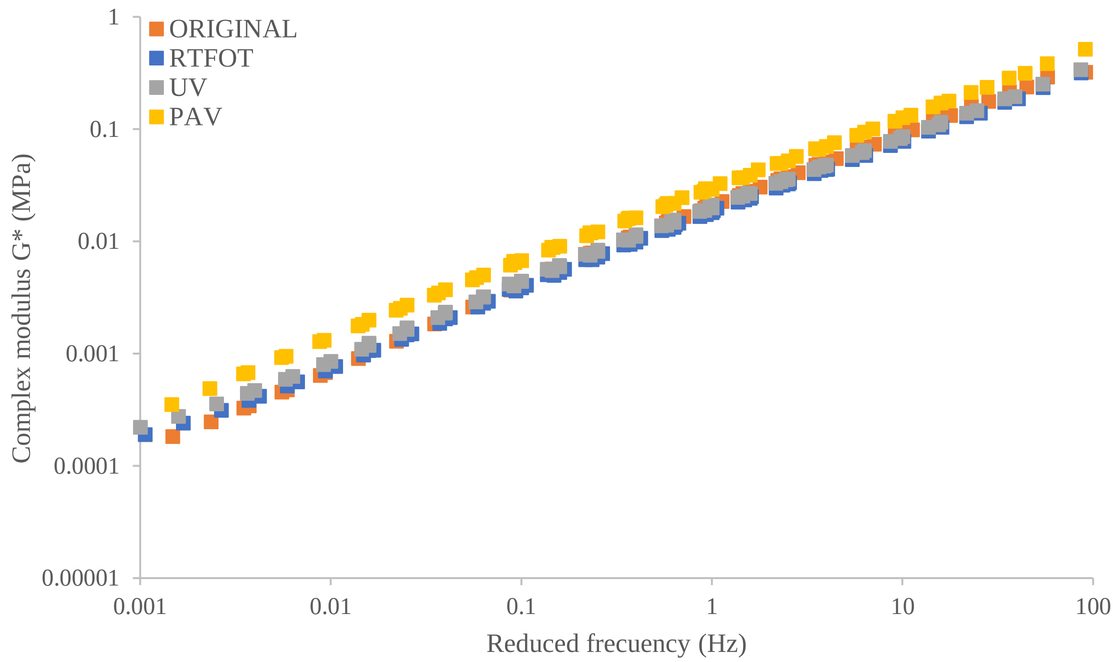

Fig. 15. Master curves of Complex Modulus for 22RTR (T°$_{ref}$ = 55 °C)

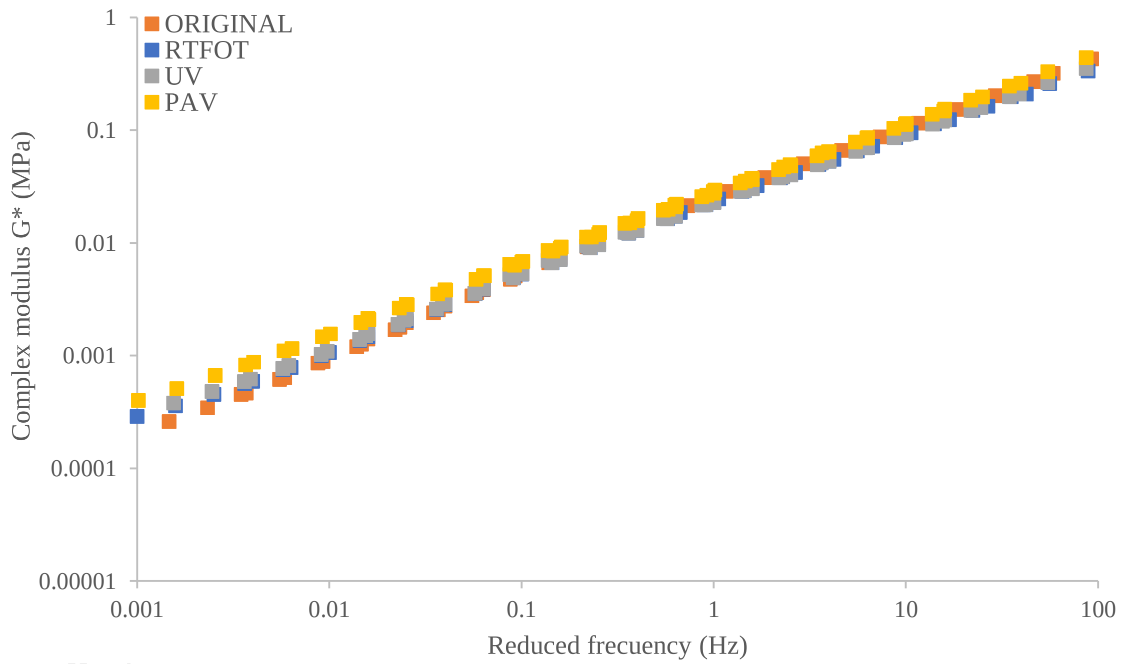

Fig. 16. Master curves of Complex Modulus for 25RTR (T°$_{ref}$ = 55 °C)

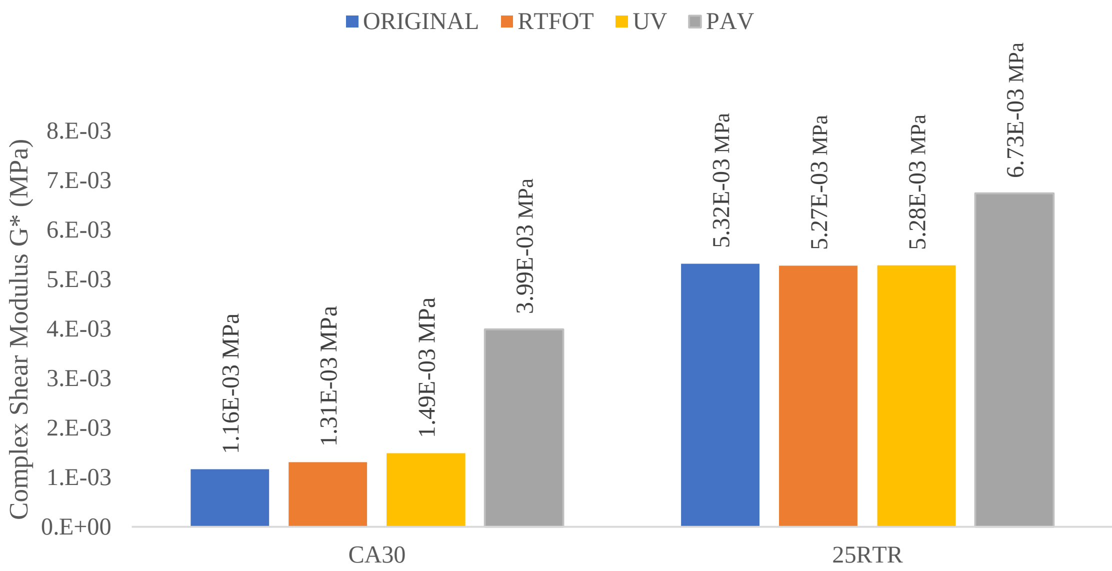

Fig. 17. Complex shear modulus for samples CA30 and 25RTR (fr=0.1Hz)

4.4 SHRP – SUPERPAVE Parameter

Figure 18 shows the results of the G*sinδ parameter for the evaluated asphalts under different aging conditions. The blue line represents the threshold established by SUPERPAVE for fatigue performance, set at 5 MPa. As observed, the difference between the various conditioning treatments decreases as the proportion of RTR incorporated increases. Specifically, the CA30 sample remains below the threshold only for Original group, while for the other conditions, it no longer meets the required performance. For the 15RTR sample, only PAV group exceeds the limit.

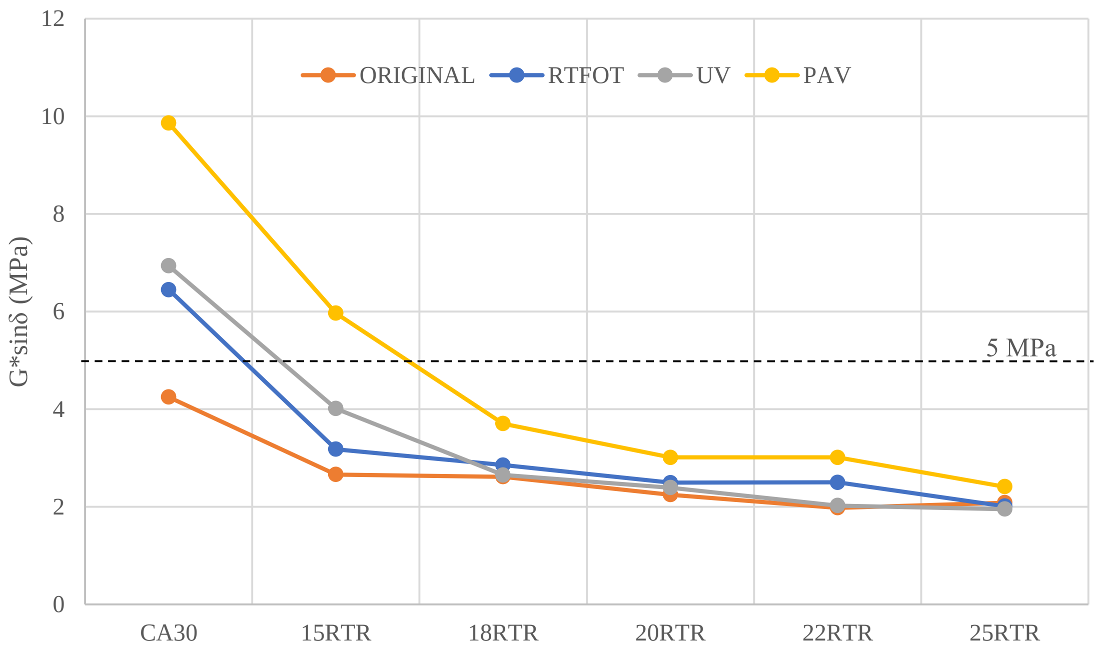

Fig. 18. SHRP SUPERPAVE G*sinδ parameter at 20 °C

For the 15RTR sample, Original group drops to 40% of the value obtained by the CA30 sample. In this case, PAV group is the only one that exceeds the threshold, by just 1 MPa. From the 18RTR sample onward, all groups fall below the limit, with the 25RTR sample showing very little variation between Original and PAV gropus. Furthermore, starting from the 18RTR sample, the results for Original, RTFOT and UV groups show significant similarity.

5. Conclusions

Successful dispersions of RTR powder with asphalt were carried out at proportions of 15%, 18%, 20%, 22%, and 25% by weight relative to 100% of asphalt weight (designated as 15RTR, 18RTR, 20RTR, 22RTR, and 25RTR, respectively), along with the base asphalt, referred to as CA30. It is important to note that RTR generation in Argentina is tailored to the specific needs of the asphalt industry, with high production rates, as previously mentioned.

Three aging conditions were applied to simulate the different stages of aging that asphalt cement undergoes. As a baseline for comparison, unaged samples, referred to as Original group, were analyzed. Short-term aging was simulated using the RTFOT oven, medium-term aging with UV chamber exposure, and long-term aging was simulated in a Pressure Aging Vessel (PAV).

The softening point of the samples showed a consistent increase across all samples after short-term aging, indicating a uniform effect of RTR incorporation on this property. A notable benefit was observed in samples aged in the UV chamber, particularly at higher proportions of RTR, as the variation in softening point compared to the unaged samples was smaller. In contrast, long-term aging led to a significant increase in the softening point, highlighting the aggressiveness of this conditioning method.

The master curves of the complex shear modulus demonstrate that the incorporation of increasing amounts of RTR enhances the material's susceptibility to aging. Furthermore, a corresponding increase in the modulus values is observed with the addition of higher RTR content

Fourier Transform Infrared (FTIR) spectra of the asphalt samples were analyzed across a wavelength range of 500 to 3500 cm^{-1}. The results showed a decrease in the variation of the functional group peaks, particularly in the band around 2,924 cm^{-1}, where a reduction in variation was observed for the PAV group samples compared to the Original samples. This suggests that increasing amounts of RTR in the asphalt binder lead to a more stable chemical structure.

The SUPERPAVE SHRP parameter $G^*\sin\delta$, evaluated at 20°C, showed clear improvement with higher RTR incorporation. For the CA30 sample, only the Original group remained below the 5 MPa threshold, while all other groups exceeded it, with values nearly double for PAV group. In the case of the 15RTR sample, Original group dropped to nearly 40% of the value observed for the CA30 sample. For the 15RTR sample, the only group exceeding the 5 MPa limit was PAV, by just 1 MPa. From the 18RTR sample onward, all groups fell below the threshold, with the 25RTR sample showing very little variation between Original and PAV groups.

Through the different evaluations, the benefit of RTR in asphalt can be seen. The best results against aging were obtained with the highest percentages, showing benefits in the evaluations of softening point, complex shear modulus and the SHRP SUPERPAVE parameter.

References

AASHTO M320. (2010). Standard Specification for Performance-Graded Asphalt Binder. American Association of State Highway and Transportation Officials.

AASHTO R28. (2022). Accelerated Aging Of Asphalt Binder Using a Pressurized Aging Vessel. American Association of State Highway and Transportation Officials.

AASHTO T240. (2022). Effect of Heat and Air on a Moving Film of Asphalt Binder (Rolling Thin-Film Oven Test). American Association of State Highway and Transportation Officials.

AASHTO T315. (2012). Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR). American Association of State Highway and Transportation Officials.

Alsheyab, M. A., Khedaywi, T., & Ogiliat, O. (2023). Effect of waste tire rubber on properties of asphalt cement and asphalt concrete mixtures: State of the art. International Journal of Pavement Research and Technology, 1-12. https://doi.org/10.1007/s42947-023-00361-4

Apaza, F. R., Fernández Vázquez, V., Paje, S. E., Gulisano, F., Gagliardi, V., Saiz Rodríguez, L., & Gallego Medina, J. (2024a). Towards Sustainable Road Pavements: Sound Absorption in Rubber-Modified Asphalt Mixtures. Infrastructures, 9(4), 65. https://doi.org/103390/infraestructures9040065

Apaza, F. R. A., Jurado-Piña, R., Díaz-Torrealba, R., Gulisano, F., Boada-Parra, G., & Gallego, J. (2024b). Texture and skid resistance of asphalt mixtures with crumb rubber: laboratory assessment. International Journal of Pavement Engineering, 25(1), 2378335. https://doi.org/10.1080/10298436.2024.2378335

ASTM D5. (2020) Standard Test Method for Penetration of Bituminous Materials. American Society for Testing and Materials.

ASTM D36. (2020) Standard Test Method for Softening Point of Bituminous (Ring-and-Ball Apparatus). American Society for Testing and Materials.

ASTM D2872. (2022) Standard Test Method for Effect of Heat and Air on a Moving Film of Asphalt Binder (Rolling Thin-Film Oven Test). American Society for Testing and Materials.

ASTM D4402. (2023) Standard Test Method for Viscosity Determination of Asphalt at Elevated Temperatures Using a Rotational Viscometer. American Society for Testing and Materials.

ASTM G154. (2023) Standard Practice for Operating Fluorescent Ultraviolet (UV) Lamp Apparatus for Exposure of Materials. American Society for Testing and Materials.

ATM 122. (2022) Torsional Recovery of Polymer Modified Binders. Austroads Test Methods and Specifications.

Bahia, H. U., & Davies, R. (1994). Effect of crumb rubber modifiers (CRM) on performance related properties of asphalt binders. *Asphalt paving technology*, *63*, 414-414.

Botasso, G., & Segura, A. (2013). Estudio experimental de microaglomerado asfáltico antiderrapante modificado con NFU. Obras y proyectos. (14), 36-44 http://dx.doi.org/10.4067/S0718-28132013000200003

Briliak, D., & Remisova, E. (2022). EXPERIMENTAL STUDY ON THE STRUCTURAL BEHAVIOUR OF ASPHALT MIXTURES EXPOSED TO UV RADIATION. International Multidisciplinary Scientific GeoConference: SGEM, 22(6.1), 155-162. 10.5593/sgem2022/6.1/s26.19

CEDEX. (2013). Neumáticos Fuera de uso. Catálogo de residuos utilizados en la construcción.

Chavarria Salas, P. (2005). Desarrollo de modelos de agrietamiento por fatiga para el concreto asfáltico en Costa Rica.

Chang, M., Zhang, Y., Pei, J., Zhang, J., Wang, M., & Ha, F. (2020). Low-temperature rheological properties and microscopic characterization of asphalt rubbers containing heterogeneous crumb rubbers. *Materials*, *13*(18), 4120. https://doi.org/10.3390/ma13184120

Chaubey, N. K., & Mishra, A. K. (2024). Feasibility of using electric induction furnace steel slag and copper slag in the production of hot mix asphalt. *Civil Engineering Infrastructures Journal*, *57*(2), 225-246. 10.22059/ceij.2023.354048.1906

Chavez, F., Marcobal, J., & Gallego, J. (2019). Laboratory evaluation of the mechanical properties of asphalt mixtures. Construction and Building Materials, 205, 164-174. https://doi.org/10.1016/j.conbuildmat.2019.01.159

Cortés, C., Servia Canto, Pérez, A., García, J., Fermoso, J., Esquenal, J., & Potti, J. (2010). Envejecimiento Foto-oxidativo de betunes asfálticos. Comunicaciones libres de la V Jornada Nacional de ASEFMA.

Gallego, J. (2018). Guía para la fabricación y puesta en obra de mezclas bituminosas con polvo de neumático

Guo, F., Shen, Z., Jiang, L., Long, Q., & Yu, Y. (2024). Study on the Performance of Asphalt Modified with Bio-Oil, SBS and the Crumb Rubber Particle Size Ratio. *Polymers*, *16*(13), 1929. https://doi.org/10.3390/polym16131929

Gallego, J., Rodríguez-Alloza, A. M., & Saiz-Rodríguez, L. (2020). Evaluation of warm rubberized stone mastic asphalt mixtures through the Marshall and gyratory compactors. Materials, 13(2), 265. https://doi.org/10.3390/ma13020265

Lee, S. J., Akisetty, C. K., & Amirkhanian, S. N. (2008). The effect of crumb rubber modifier (CRM) on the performance properties of rubberized binders in HMA pavements. *Construction and Building Materials*, *22*(7), 1368-1376. https://doi.org/10.1016/j.conbuildmat.2007.04.010

LEMaC. (2019). Guía de Metodologías y Procedimientos para uso Vial desarrollados en el LEMaC. edUTecNe.

Liu, H., Zhang, Z., Tian, Z., & Lu, C. (2022). Exploration for uv aging characteristics of asphalt binders based on response surface methodology: insights from the uv aging influencing factors and their interactions. Construction and Building Materials, 347, 128460. https://doi.org/10.1016/j.conbuildmat.2022.128460

Khadim, H. M., & Al-Mosawe, H. M. (2024). Crumb rubber modification for enhanced rutting resistance in asphalt mixtures. *Open Engineering*, *14*(1), 20220500. https://doi.org/10.1515/eng-2022-0500

Kök, B. V., & Çolak, H. (2011). Laboratory comparison of the crumb-rubber and SBS modified bitumen and hot mix asphalt. *Construction and Building Materials*, *25*(8), 3204-3212. https://doi.org/10.1016/j.conbuildmat.2011.03.005

Morea, F., Nosetti, R., Gonzalez, L., & Sánchez, A. (2023). Performance analysis of non-conventional Stone Mastic asphalt (SMA) elaborated with crumb rubber bitumen or by mean of glass macrofibers addition. Construction and Building Materials, 400, 132654. https://doi.org/10.1016/j.conbuildmat.2023.132654

Moreno Navarro, F., Sol-Sánchez, M., Gámiz, F., & Rubio-Gámez, M. C. (2018). Mechanical and thermal properties of graphene modified asphalt binders. *Construction and Building materials*, *180*, 265-274. https://doi.org/10.1016/j.conbuildmat.2018.05.259

Nanjegowda, V. H., & Biligiri, K. P. (2023). Utilization of high contents of recycled tire crumb rubber in developing a modified-asphalt-rubber binder for road applications. *Resources, Conservation and Recycling*, *192*, 106909. https://doi.org/10.1016/j.resconrec.2023.106909

Panda, M., & Mazumdar, M. (1999). Engineering properties of EVA-modified bitumen binder for paving mixes. *Journal of materials in civil engineering*, *11*(2), 131-137. https://doi.org/10.1061/(ASCE)0899-1561(1999)11:2(131)

Pouranian, M. R., Notani, M. A., Tabesh, M. T., Nazeri, B., & Shishehbor, M. (2020). Rheological and environmental characteristics of crumb rubber asphalt binders containing non-foaming warm mix asphalt additives. *Construction and Building Materials*, *238*, 117707. https://doi.org/10.1016/j.conbuildmat.2019.117707

Read, J., & Whiteoak, D. (2003). *The shell bitumen handbook*. Thomas Telford.

Rodríguez-Alloza, A. M., Gallego, J., Pérez, I., Bonati, A., & Giuliani, F. (2014). High and low temperature properties of crumb rubber modified binders containing warm mix asphalt additives. *Construction and Building Materials*, *53*, 460-466. https://doi.org/10.1016/j.conbuildmat.2013.12.026

Segura, A., Botasso, G., Raggiotti, B., Zapata, I., & Rebollo, O. (2023). Valoración inicial de mezclas asfálticas retardante de fisuras reflejas elaboradas con asfaltos con alta incorporación de NFU. *Obras y proyectos*, (34), 18-28. http://dx.doi.org/10.21703/0718-2813.2023.34.2443

SIGNUS. (2017). Guía para la fabricación y puesta en obra de mezclas bituminosas con polvo de neumático.

Soengas, C., Gerardi, C., Botto, G., Zapata, M., & Botasso, G. (2022). Comparación de resultados del coeficiente de absorción acústica, microtextura y macrotextura entre microaglomerados en caliente realizados con asfalto AM-3 vs Asfalto Caucho. *Proceedings of XVIII Congreso Argentino de Vialidad y Tránsito.*

Sol-Sánchez, M., del Barco Carrión, A. J., Hidalgo-Arroyo, A., Moreno-Navarro, F., Saiz, L., & del Carmen Rubio-Gámez, M. (2020). Viability of producing sustainable asphalt mixtures with crumb rubber bitumen at reduced temperatures. *Construction and Building Materials*, *265*, 120154. https://doi.org/10.1016/j.conbuildmat.2020.120154

Soleymani, H. R., Zhai, H., & Bahia, H. (2004). Role of modified binders in rheology and damage resistance behavior of asphalt mixtures. *Transportation Research Record*, *1875*(1), 70-79. https://doi.org/10.3141/1875-08

Song, L., Hou, L., Tu, P., Fan, P., & Gao, J. (2023). Study on UV aging characteristics of low-grade asphalt in the desert climate. Materials Research Express, 10(9), 095308. https://doi.org/10.1088/2053-1591/acee49

Tauste, R., Moreno-Navarro, F., Sol-Sánchez, M., & Rubio-Gámez, M. C. (2018). Understanding the bitumen ageing phenomenon: A review. *Construction and Building Materials*, *192*, 593-609. https://doi.org/10.1016/j.conbuildmat.2018.10.169

Wang, H., Liu, X., van de Ven, M., Lu, G., Erkens, S., & Skarpas, A. (2020). Fatigue performance of long-term aged crumb rubber modified bitumen containing warm-mix additives. *Construction and Building Materials*, *239*, 117824. https://doi.org/10.1016/j.conbuildmat.2019.117824

Wang, H., Huang, Y., Jin, K., & Zhou, Z. (2022). Properties and mechanism of SBS/crumb rubber composite high viscosity modified asphalt. *Journal of Cleaner Production*, *378*, 134534. https://doi.org/10.1016/j.jclepro.2022.134534

Yu, X., Li, D., Leng, Z., Yao, H., & Wang, S. (2023). Weathering characteristics of asphalt modified by hybrid of micro-nano tire rubber and SBS. *Construction and Building Materials, 389, 131785*. https://doi.org/10.1016/j.conbuildmat.2023.131785