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# A Laboratory Assessment of the Influence of Crumb Rubber in Hot Mix Asphalt with Recycled Steel Slag

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**Abstract:** To reduce thermal susceptibility and improve rutting and fatigue cracking resistance, increasingly more non-conventional additives and materials have been used in road pavement asphalt mixes in recent years. Non-conventional materials mainly include recycled materials, which reduce production costs and lead to environmental benefits related to their reuse. The aim of this research was to evaluate the influence of recycled tyre rubber in the production of asphalt concrete for road pavements built with recycled aggregates consisting of steel slag in relation to possible improvements in structural performance during operation (i.e., fatigue and rutting). Steel slag has a higher bulk specific gravity than natural aggregates, and it has a very porous surface that allows for a different interaction with the bitumen and the crumb rubber compared to traditional aggregates. To this end, two mixtures of asphalt concrete are compared. One was mixed with a modest percentage by weight of crumb rubber using the "dry" technique, and the other mixture did not contain crumb rubber. Indirect tensile and compression tests with cyclic loads were performed to determine the mechanical behaviour of the two mixtures at different temperatures and under different load frequencies. The results of this research indicate the better performance of the modified mixture with crumb rubber, which agrees with other experiments in the literature that have been made using natural aggregates. Furthermore, a significant increase in stiffness was found at high temperatures (up to 30%), a slight reduction (up to 8%) was found at low temperatures, and a reduction in permanent deformation was found under cyclic loads.

**Keywords:** crumb rubber; dry method; asphalt pavements; steel slag; mechanical properties; hot mix asphalt

## 1. Introduction

The constant increase in light and heavy vehicle traffic throughout the years causes increasing asphalt pavement deterioration, with a consequent reduction in asphalt's useful life.

Asphalt mixtures in road pavements are materials with viscoelastic behaviour, so their mechanical properties depend to a large extent on the operating temperature and frequency of load applications. At high temperatures and/or low load frequencies, asphalt mixes behave more viscously and less rigidly (ductile behaviour), resulting in plastic deformation. Conversely, under low operating temperatures or high frequencies loads, these materials have almost completely elastic behaviour with less deformation, albeit with a greater possibility of fatigue cracking.

To reduce the effects of temperature and load frequency on asphalt pavement and thereby increase the useful life of the pavement, non-traditional additives and materials have been used for several years, both in the bitumen and in the aggregate mix, to modify and improve the asphalt's mechanical behaviour.

The use of recycled additives and non-traditional materials is emerging as an interesting alternative to reduce the production costs of bituminous mixtures while at the same time improving their mechanical performance, thereby increasing the useful life of the pavement. The use of such non-traditional materials makes it possible to recycle materials that would otherwise have to be landfilled, with all the related economic costs and environmental impacts.

There are two primary techniques used for adding additives to a mixture: the "wet" method and the "dry" method. In the "wet" method, additives are added directly into the bitumen at high temperatures before the bitumen is mixed with the aggregates; thus, chemical and physical changes are made to the binder, which will then lead to a change in the mechanical performance of the mixture. In the dry method, additives are added to the aggregates together with the bitumen or mixed with the aggregates before the bitumen is added. The choice of method depends on the type of additive to be used. In principle, however, the dry method is simpler and less expensive since it does not require any special equipment.

This research evaluated the influence of using recycled tyre rubber in the production of asphalt concrete for road pavements that were also produced with aggregates made from steel slag to evaluate the possible improvements in paving life.

Every year, more than one billion tyres are disposed of worldwide (a total of almost 17 million tons). Most of these tyres are recycled, while the remaining portion is disposed of in landfills (when not illegally dumped) [1]. The most common applications of recycled tyres include the production of new tyres (retreaded tyres), fuel, civil engineering applications and products, agricultural uses, recreational and sports applications, and road pavement construction [2]. In the field of road pavements, the advantages of using crumb rubber have been acknowledged in several studies, and it is likely that the use of crumb rubber will increase in the coming years.

Tyre rubber (and consequently crumb rubber) is an elastomer, a polymer capable of deforming significantly when subjected to stress and recovering its initial shape as soon as stress ceases. Crumb rubber can be added to bituminous conglomerates through both wet and dry processes. The choice of process depends on the amount of crumb rubber used, its size, the required function, and the type of plants available.

Although the dry process has some advantages over the wet process, especially in terms of cost and the greater amount of rubber that can be used, research worldwide has focused mainly on the wet process. This is because the performance obtained with the dry process is not always uniform. Such uncertainties stem from the poor adhesion between crumb rubber and bitumen since the short mixing phase does not allow for their complete interaction [3]. This is not the case in the wet process, which yields more satisfactory results, as the wet process has the advantage of being able to better control the modified properties of the binder through sophisticated mixing plants [4]. The wet method, however, also has disadvantages. In general, it is necessary to use specialized plants where high temperatures and stirring processes are required; this method is, therefore, less viable economically and, in some cases, may present hardships in separating the crumb rubber and bitumen phases during storage [5]. Optimizing the dry method to achieve consistent performance for different types of aggregates could prove more promising in both economic and environmental terms.

In the "wet" method, the added amounts of crumb rubber can vary between 1.75% and 25.0% with respect to the weight of bitumen, although the top-performing amounts lie between 10% and 15% by weight, while the particle size of the crumb rubber varies between 0.15 mm and 0.60 mm. The mixing temperatures can vary between 140 °C and 195 °C (or in some cases, even higher).

Using a percentage by weight of crumb rubber of around 10% (in relation to the bitumen), the following results were obtained [6]:

- a reduction in penetration, varying between 12% and 37%;
- an increase in softening temperature of between 9% and 23%, depending on the type of basic bitumen used;
- a significant reduction in ductility at 15° and an increase in viscosity, which remained below 3 (Pa·s) at 135 °C, as required by SHRP specifications [7–9].

Comparing the original bitumen with the bitumen resulting from the addition of about 10% by weight of crumb rubber bitumen, the complex modulus of the bituminous mix was increased in the latter mixture, and the phase angle was reduced at high and medium temperatures. At the same time, however, the complex modulus—the "creep stiffness" and the "m" parameter (slope of the straight-line tangent to the stiffness logarithm versus the logarithm of time)—was reduced at lower temperatures; the first two decreases are beneficial, while the decrease in the "m" parameter represents an inconvenience. The mixtures produced with the crumb-rubber-modified bitumen presented a higher value of recoverable deformations and a minimum area within the stress–strain curve, which indicates a better ability to dissipate energy [7,8,10–15]. The addition of this additive to bitumen, therefore, seems to increase the stiffness of asphalt concretes at high temperatures, as is the case with other additives, such as plastomers (which would reduce the excessive fragility of the bitumen and, therefore, the risk of cracking at low temperatures, thereby increasing the useful life of the pavement).

Unlike the wet method, for which there are many studies using crumb rubber, the dry method has rarely been investigated, especially for non-traditional aggregates. In the dry method, the crumb rubber can perform a double function based on the size of the particles. If coarse particle sizes of crumb rubber are used, their function is to replace a part of the coarse aggregate with an elastic aggregate to increase the mixture's flexibility under load. If fine particle sizes are used instead, the crumb rubber will react more intensively with the bitumen by changing its viscosity. In the latter case, the rubber particles will absorb the lighter fractions (oils) of the maltenes present in the bitumen, which are the fractions that influence the viscous properties of the material, leaving a higher percentage of the asphaltenes in bitumen. The adsorption of the maltene fractions causes the swelling of the rubber particles, which is influenced by the temperature and bitumen-to-rubber contact time, the chemical composition of the bitumen, and the size and quantity of the crumb rubber [16].

In the "dry" method, the crumb rubber normally varies between 0.5% and 10% of the total weight of the mixture, with the most common percentage being 1%. The sizes of the granules generally range from 0.6 mm to 3.0 mm [17–20], although higher particle sizes have been used in some experiments. The mixing temperatures range between 180 °C and 190 °C [21]. Compared to mixtures without crumb rubber, the optimal bitumen content in this mixture is 0.5% higher. Indeed, the introduction of crumb rubber leads to a lower workability of the mixture [22].

At medium–high temperatures (20–70 °C), with the addition of modest quantities of crumb rubber, both the creep modulus and the stiffness modulus increase, thus reducing permanent deformations and increasing the load-bearing capacity, while higher percentages do not always improve the mixtures' mechanical characteristics. The percentage of crumb rubber needed to obtain the best results in terms of stiffness depends on the types of aggregates and bitumen and can vary from 1.5% to 12% of the weight of the mixture [17,18,20,21].

Da Silva et al. [23] indicated that the addition of crumb rubber to the asphalt concrete produced an increase in service life of 10 times for fatigue strength and of 2.5 times for resistance to permanent deformation.

Farouk et al. [24] highlighted the poor interactions between bitumen and crumb rubber as one of the major drawbacks of the dry method. This drawback causes the same crumb rubber to swell in bituminous mixes after compaction to three to five times its original size. This, in turn, hampers the achievement of the optimum mixture density if compaction takes place before the crumb rubber swelling is complete. Farouk et al. also suggest that it is preferable to use a smaller size of crumb rubber, which facilitates swelling in a short period of time before the mixture is put into operation. The authors also suggest that the percentage of bitumen should be increased to compensate for the amount absorbed by the crumb rubber. Finally, large particle sizes of crumb rubber produce a reduction in the resilient and creep modulus, even at medium–high temperatures.

Arabani et al. [25] noted that percentages of crumb rubber up to 3% (by weight of the mixture) increase the stiffness modulus and reduce permanent deformations, while percentages higher than 3% negatively affect the mechanical parameters of the mixture, thus reducing its useful life due to the poor adhesion between the bitumen and aggregates in the presence of high percentages of crumb rubber.

Sangiorgi et al. [26] analysed the behaviour of a porous bituminous mixture with the addition of crumb rubber at 1% by weight of the mixture and a fine grain size less than 1 mm, finding that, at low temperatures, the stiffness modulus, evaluated by an indirect tensile test, was lower than that of the control mixture, while at high and medium temperatures, there was no significant difference between the two.

Lastra-Gonzales et al. [5] indicated that, by using 1% by weight crumb rubber in the mixture with a grain size of less than 1 mm, there was a 30% increase in plastic deformation resistance at 60 °C and a 50% increase in stiffness (dynamic modulus) at 20 °C compared to the unmodified mixture with the crumb rubber.

Hassan et al. [16], drawing on an extensive literature review, outlined guidelines for the use of the dry method, including recommendations for the grain-size composition of the aggregates, the physical properties of the binder, and the mixing and packaging conditions of the mixtures. In summary:

- The grain size curves of the aggregates must be such that some of the aggregates are replaced by crumb rubber of the same size for mixtures of a closed type to fill the voids in the gap-graded mixtures.
- It is preferable to use bitumen with a higher degree of penetration than traditional mixtures with increased bitumen content from 1 to 2%.
- A combination of coarse and fine particle sizes is desirable to achieve better performance.
- Mixtures with lower residual vacuum values (<3%) are preferable to traditional mixtures.
- To use higher mixing temperatures, the aggregates and crumb rubber should be mixed first and the bitumen should be added afterward.
- Mixture paving and compacting should occur 1–2 h after mixing. Stirring should occur before paving, and compacting should occur as soon as possible after paving.

Moreno-Navarro et al. [18] evaluated the effects of adding crumb rubber to mixes made with high modulus bitumen, finding that this addition slightly reduced the mechanical resistance to indirect traction but increased the resistance to plastic deformation and stiffness.

Moreno et al. [17] compared mixtures of bituminous conglomerates, made via both the wet and dry methods using crumb rubber, with other mixtures made with polymer modified bitumens. The results of the experiment show that the dry method offers the best performance in both permanent deformations and stiffness.

Moreno et al. [27] analysed the behaviour of asphalt mixtures modified with the addition of crumb rubber via the dry method in terms of their sensitivity to humidity and resistance to plastic deformation, finding that the most influential factor on the mixtures' behaviours was the percentage of crumb rubber, while the digestion time (i.e., the time between mixing and compaction) was barely significant. In particular, the best results were obtained with percentages varying between 0.5% and 1.0% of crumb rubber (compared to the weight of the mixture) and a digestion time of 45 min.

Cao [4] evaluated the behaviour of gap-grade asphalt mixtures achieved via the addition of different percentages of crumb rubber, from 1 to 3% of the weight of the mixture, using the dry method. Cao found that mixtures with higher percentages of crumb rubber offered the best behaviour, both for permanent deformation at high temperatures and for cracking at low temperatures.

The analysis of previous studies shows that the addition of crumb rubber can certainly yield benefits. Uncertainties remain, however, for the dry method, as the different variables involved, such as the size, percentage, and type of crumb rubber, the bitumen quality, the mixing time, and the temperature, affect the performance of the mixture, especially when using artificial aggregates, such as steel slag, that have a higher bulk specific gravity (>3.40 g/cm$^3$) than natural aggregates and have a very porous surface that allows for a different interaction with the bitumen and the crumb rubber compared to traditional aggregates.

Although a considerable amount of research has been conducted in the past on the use of crumb rubber in asphalt concrete made with natural aggregates, little to no research has been done on crumb rubber used with artificial aggregates, in particular with the dry method.

The goal of this research, different from what is present in the literature, is to evaluate the influence of using recycled tyre rubber in the construction of asphalt concretes for road pavements produced using artificial aggregates (steel slag) that have very different characteristics compared to traditional aggregates.

Two mixtures of bituminous conglomerates made from steelwork slag were compared, and one of these mixtures was modified using the "dry" technique by adding a percentage by weight of crumb rubber. Cyclical indirect tensile tests were carried out at different temperatures and load frequencies to evaluate the viscoelastic responses of the mixtures (resilient and dynamic modulus), and cyclic compression tests (dynamic creep) were carried out to evaluate the accumulation of permanent deformations (rutting).

## 2. Materials and Methods

### 2.1. Material and Volumetric Design

In general, bituminous mixtures for road pavements are prepared using two types of materials: aggregates and a binder, usually bitumen. The mixtures covered by this study, however, are composed with an additional element, recycled tyre rubber.

For aggregates (usually crushed rock), different lithotypes can be used during the preparation of the mixtures, including limestone, basalt, and porphyry. For each of these lithotypes, mixtures with different mechanical characteristics can be obtained. In this work, we evaluate a particular mixture of aggregates in which the coarse fraction (4–8 mm) is represented by steel slag (Figure 1), the fine fraction (0.063–4 mm) by limestone sand, and the filler (less than 0.063 mm) by cement dust. The average chemical composition of steel slag, represented in oxides, falls within the range shown in Table 1.


**Figure 1.** Steel slag used as coarse fraction, 4–8 mm.

**Table 1.** Range of variation in the chemical composition of steel slag.

| Range | CaO (%) | MgO (%) | FeO (%) | $Al_2O_3$ (%) | $SiO_2$ (%) |
|---|---|---|---|---|---|
| min | 21.8 | 4.1 | 24.1 | 4.6 | 13.3 |
| max | 29.3 | 8.6 | 43.3 | 11.0 | 19.8 |

Bitumen is the binder for the solid skeletons of aggregates and may have different characteristics, mainly linked to its behaviour under different temperatures and to the stiffness it can provide to bituminous mixtures. Many types of bituminous binders are available on the market and offer the possibility of preparing mixtures with different mechanical characteristics. Traditional bitumen modified with an SBS polymer was used for preparing our mixture to facilitate workability at low temperatures and avoid the volatilization of odorous substances. The main mechanical characteristics are shown in Table 2.

**Table 2.** Binder characteristics.

| Parameter | Value | Unit | Standard |
|---|---|---|---|
| Penetration to 25 °C | 32 | dmm | UNI EN 1426 |
| Softening point | 79 | °C | UNI EN 1427 |
| Dynamic viscosity 80 °C | 352 | Pa s | UNI EN 13072-2 |
| Dynamic viscosity 160 °C | 0.35 | Pa s | UNI EN 13072-2 |

The second unconventional material used for preparing the mixture was crumb rubber (Figure 2). This material is the result of a reduction in the small grain size of exhausted tyres. Given the nature of the material, the material's characteristics can vary according to the kind of tyre (i.e., for light or heavy vehicles). The crumb rubber is, therefore, composed of a mixture of different rubbers, both natural and synthetic. The proportion of these rubbers also depends on the type of tyre. For example, the natural rubber content of heavy-duty tyres is higher than that of light-duty tyres. In addition to the presence of rubber, the crumb also consists of a variable (albeit always negligible) percentage of steel and textile materials.


**Figure 2.** Recycled crumb rubber.

The aim of the study was to verify the mechanical improvements that can be achieved by adding a certain amount of crumb rubber to a bituminous mixture that contains steel slag. In this way, we analyse a bituminous mixture almost entirely produced with recycled materials. The steel slag has a particularly porous surfaces, so there is usually a need to increase the percentage of bitumen in the formulation of the mixture. For this purpose, two identical mixtures were prepared, with one of them altered via the addition of crumb rubber. Dynamic laboratory tests were carried out to assess their mechanical behaviour. The basic recipe of the mixture is represented by an asphalt concrete commonly used in the geographical area where the experiment was carried out (North-East Italy) called "traditional surface course" (AC). In this recipe, a quantity of recycled tyre crumb rubber, equal to 0.75% by weight, was added to the mixture (a value close to the amount considered best [17–20]) using the dry technique, thereby obtaining Asphalt Rubber (AR). The grain size of the crumb rubber corresponds to that of fine sand (see Figure 3, which also shows the grain size distributions of carbonate sand and steel slag).

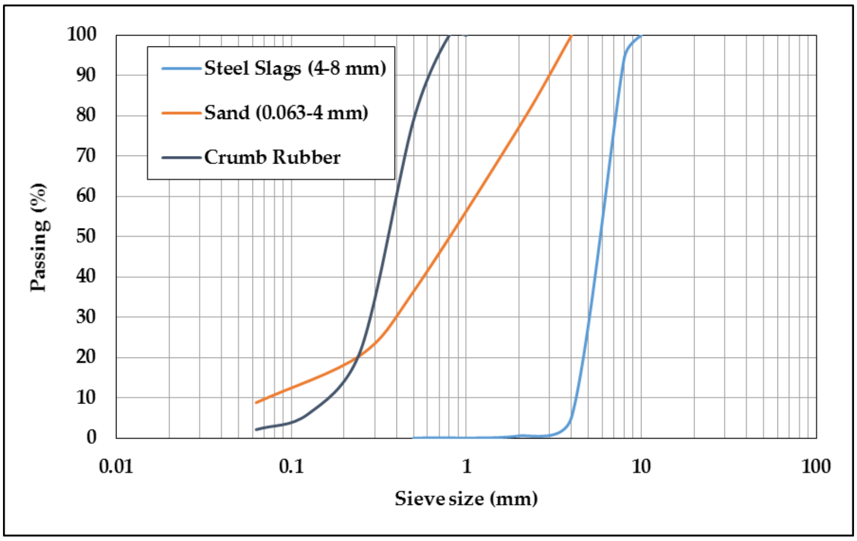


**Figure 3.** Gradation curve of the aggregates.

Table 3 and Figure 4 show the particle size distributions of the two mixtures (traditional surface course and asphalt rubber) prepared in the laboratory. The limit lines represent the maximum and minimum value for every particle size used in the mix design for this type of asphalt concrete in the geographical area where the tests were conducted.

**Table 3.** Particle size distribution of the mixtures.

| Aggregate Size (mm) | Upper Limit (%) | Lower Limit (%) | AC (%) | AR (%) |
|---|---|---|---|---|
| 10 | 100 | 100 | 100.00 | 100.00 |
| 8 | 90 | 100 | 96.50 | 94.92 |
| 4 | 35 | 50 | 36.50 | 35.37 |
| 2 | 22 | 32 | 27.40 | 26.27 |
| 0.5 | 10 | 18 | 15.69 | 14.65 |
| 0.25 | 8 | 15 | 12.35 | 11.72 |
| 0.063 | 6 | 12 | 7.53 | 7.55 |

The percentage of bitumen in the mixture, expressed as the total weight of the aggregates, was 5.5% for both the AC and AR mixtures and was chosen using the Marshall methodology while optimizing the Marshall stability, the percentage of residual air voids, the compacted mixture density, and the indirect tensile strength.

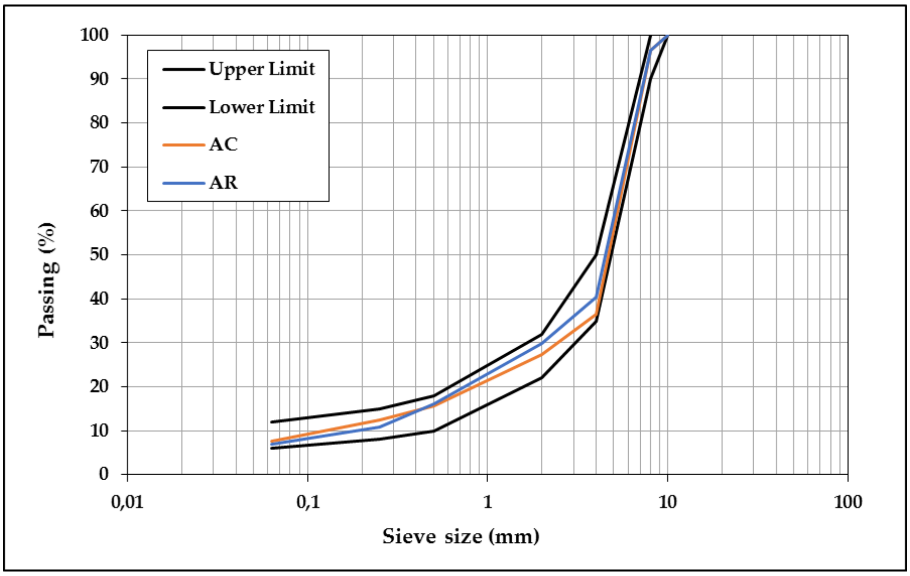


**Figure 4.** Gradation curve of the aggregate mix.

For the volumetric characteristics of the two mixtures, the specimens used to determine the mechanical characteristics were assessed after a compaction study with a gyratory press (Figure 5). The densification curves of the two mixtures, made for the test specimens with a diameter of 150 mm, were practically identical. To obtain samples with the same volumetric properties and a percentage of residual voids corresponding to that of the Marshall specimens, a number of revolutions equal to 100 (4% residual voids) was identified.

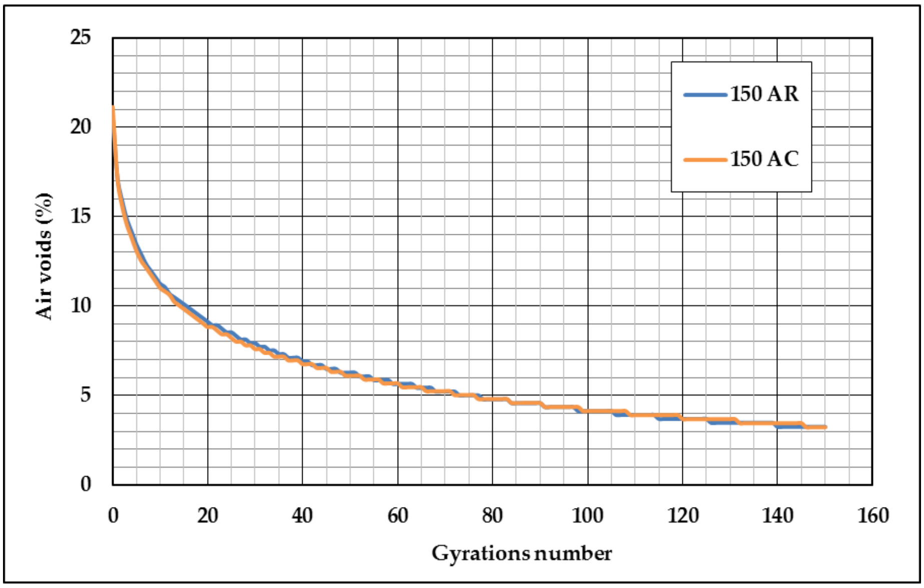


**Figure 5.** Gyratory compactor curve.

Relying on these methods, for each mixture, three samples 150 mm in diameter were prepared. The average height of the samples was 42 mm. In addition, two specimens with a height of 82 mm and a diameter of 100 mm were tested to evaluate the permanent deformations with mono-axial compression stress (dynamic creep). The temperatures of the mixtures in the compaction phase should not fall below 150 °C. The average volumetric properties are shown in Table 4.

**Table 4.** Volumetric properties of the mixtures.

| Sample | $\gamma_i$ | $\gamma_b$ | $\gamma_c$ | $b_c$ (%w/w) | av (%) | VMA (%) | VFA (%) |
|---|---|---|---|---|---|---|---|
| AC | 3.42 | 1.03 | 2.91 | 5.36 | 4.19 | 19.36 | 72.30 |
| AR | 3.40 | 1.03 | 2.89 | 5.64 | 3.94 | 19.79 | 71.50 |

Note: $\gamma_i$, aggregate bulk specific gravity; $\gamma_b$, asphalt binder specific; $\gamma_c$, bulk specific gravity of the compacted paving mix; $b_c$, asphalt content by weight of the asphalt binder in the total mixture; av, air voids; VMA, voids in the mineral aggregate; VFA, voids filled with asphalt.

### *2.2. Mechanical Properties Tests*

The mechanical properties of the two types of compacted asphalt mixtures were evaluated through dynamic tests (cyclic loads), thereby obtaining the stiffness of the samples at different temperatures, shapes, and times of load applications.

The resilient modulus is defined as the ratio between stress and reversible unit deformation when the applied load is of the pulsating type. The resilient modulus can be used to evaluate the structural response of the pavement to road loads, as well as in the design of the pavement to calculate the useful life and determine the different forms of degradation: fatigue cracking, rutting, and transverse or thermal cracking.

By evaluating the responses of the samples to repeated sinusoidal loads, the viscoelastic characterization of the material becomes more accurate with the determination of the complex modulus vector.

The sinusoidal function representing the stress can be modelled as a vector in the complex plane (Euler's equation) or written in its polar form according to Equation (1):

$$\sigma(\mathrm{t}) = \sigma_0 \cos(\omega \mathrm{t}) + i\sigma_0 \sin(\omega \mathrm{t}) = \sigma_0 e^{i\omega \mathrm{t}}. \tag{1}$$

The linear response is a vector in the complex plane delayed by the phase angle $\varphi$ according to Equation (2):

$$\varepsilon(\mathrm{t}) = \varepsilon_0 \cos(\omega \mathrm{t} - \varphi) + i\varepsilon_0 \sin(\omega \mathrm{t} - \varphi) = \varepsilon_0 e^{i(\omega \mathrm{t} - \varphi)}. \tag{2}$$

The complex modulus, defined as the ratio between two sinusoidal quantities (vectors), remains a vector quantity defined by Equation (3):

$$E^* = \frac{\sigma(t)}{\varepsilon(t)} = \frac{\sigma_0}{\varepsilon_0} \cos\varphi + i\frac{\sigma_0}{\varepsilon_0} \sin\varphi = \frac{\sigma_0}{\varepsilon_0} e^{i\varphi}. \tag{3}$$

The modulus of the complex modulus, i.e., the ratio of the sinusoidal stress amplitude to the reversible strain amplitude, is defined as the dynamic modulus and calculated according to Equation (4):

$$|\mathrm{E}^*| = E_{din} = \frac{\sigma_0}{\varepsilon_0}. \tag{4}$$

With this test, it is also possible to determine the phase shift angle (delay) of the reversible deformation and, therefore, the rates of the elastic and dissipated energy in each cycle.

The resilient modulus is determined via a Repeated Load Indirect Tension test (RLIT, Figure 6) with cyclic loads according to UNI EN 12697-26, "Test Method for Hot Mix Asphalt Stiffness" [28]. The complex modulus is also evaluated with the same type of test (indirect tension) by applying a sinusoidal load that acts along the vertical diametrical plane to a cylindrical specimen. The diametrically induced tensile stress values and the measurement of the resilient strain define the complex modulus.

In the characterization of asphalt mixtures for the sizing of road pavements, it is also necessary to quantify the irreversible deformations accumulating due to the transit of vehicles. Such deformations contribute to the formation of permanent subsidence on the road surface (wheel paths). The test used to evaluate the accumulation of permanent deformations as a function of the number of load cycles is the dynamic creep test, UNI EN 12697-25 (Repeated Load Axial test, RLA, Figure 6) [29], which was carried out on the cylindrical specimens stressed with pulsating uniaxial compression loads. The measured deformations were in the direction of the main compressive stress.

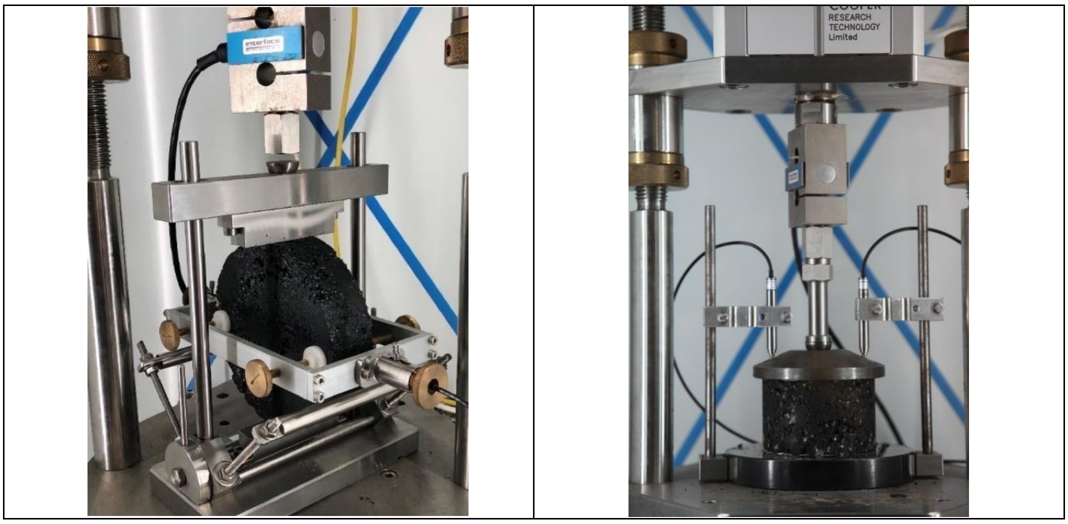


**Figure 6.** Repeated Load Indirect Tension test (RLIT) (**left**) and Repeated Load Axial test (RLA) (**right**) test setup.

Table 5 shows the operating conditions under which the tests were performed, while Figure 7 shows the main characteristics of the cycle (the peak load, pulse repetition period, and rise time), with adequate amplitude to guarantee a horizontal deformation compatible with viscoelastic behaviour. For the 150 mm diameter samples, the maximum deformation of the diameter was 7 µm.

**Table 5.** Operating conditions of the tests.

| Test | Standard | Waveform | Pulse (s) | Rest (s) | Frequency (Hz) | Load |
|---|---|---|---|---|---|---|
| Resilient modulus | EN 12697-26 | Haversine | 0.248 | 2.742 | | $\varepsilon \leq 7$ µm |
| Complex modulus | | Sinusoidal | | | 10, 5, 1, 0.1 | 100 kPa |
| Plastic deformation | | Haversine | 0.1 | 0.9 | | 100 kPa |

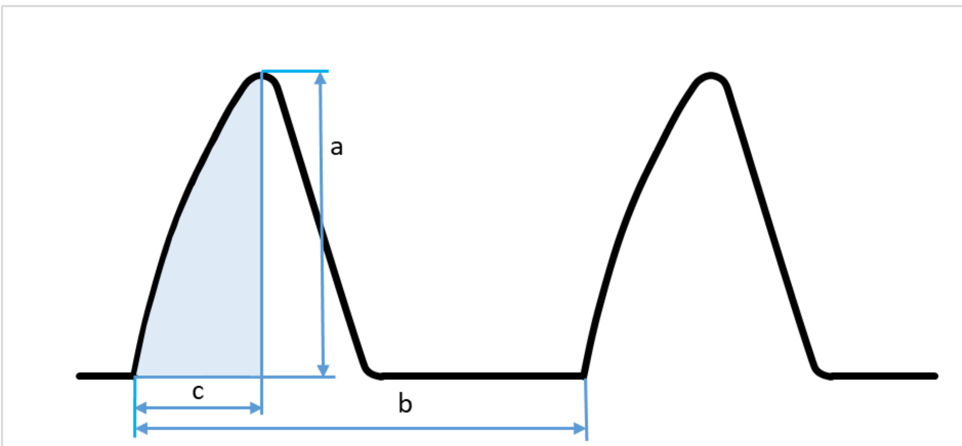


**Figure 7.** Form of the load pulse, UNI EN 12697-26: (**a**) peak load, (**b**) pulse repetition period, and (**c**) rise time.

As the rheological behaviour of materials like asphalt is influenced by the temperature and frequency of the applied loads, to compare the results, we combined the modulus values at different test frequencies into a single continuous master curve [30]. This single curve was obtained by choosing a reference temperature and translating the modulus values obtained at the other test temperatures horizontally. The correlation between frequency and temperature is expressed by the "shift factor" parameter. This parameter provides a measure of the amount of data translation, in terms of frequency, that must be performed to obtain a single master curve. For the construction of the master curve, the function of sigmoidal correlation was used (see Equation (5)) [31,32] alongside optimization of the minimum squares at the reference temperature of 20 °C:

$$\xi = \mathrm{f}\cdot\mathrm{a}(\mathrm{T}). \tag{5}$$

This can also be expressed in logarithmic terms by Equation (6), while Equation (7) represents the cyclic dynamic modulus:

$$\log(\xi) = \log(\mathrm{f}) + \log[\mathrm{a}(\mathrm{T})] \tag{6}$$

$$\log\left(\left|E^{*}_{IDT}\right|\right) = \delta + \frac{\alpha}{1 + e^{\beta - \gamma \log(\xi)}} \tag{7}$$

where

- $\left|E^{*}_{IDT}\right|$ is the cyclic dynamic modulus;
- f is the frequency;
- T is the temperature;
- a(T) is the shift factor;
- $\xi$ is the reduced frequency;
- $\delta$ is the minimum modulus value;
- $\alpha$ is the variation of the modulus;
- $\beta$, $\gamma$ are the form factors.

To evaluate the behaviour of a bituminous conglomerate in terms of its permanent deformations, the sample must be stressed with several thousand load cycles, and the accumulated permanent deformations must be recorded based on the same number of load cycles. A load cycle consists of a Haversine type load pulse with a 0.1 s duration followed by a rest time of 0.9 s.

The typical results of a repeated load test are shown in Figure 8 for the accumulated permanent deformations as a function of the number of load cycles.

As shown in Figure 8, the trend of permanent deformations in the secondary stage can be represented by a straight line in a bilogarithmic diagram according to Equation (8):

$$\log \varepsilon_p = \log a + b \log N \tag{8}$$

In normal coordinates, the prediction model for the accumulation of permanent deformations is represented with a power function according to Equation (9):

$$\varepsilon_p = aN^b \tag{9}$$

Parameter "a" represents the intercept at N (number of cycles), while "b" represents the slope of this line. Parameter "a" does not truly represent the deformation found in the first load cycle but derives exclusively from the characteristics of the second stage load with a constant slope.

To determine the equation for the secondary section of the curve, the number of cycles (Nss) at which the primary section of the curve ends and the secondary section begins must be defined. There is no universally recognized procedure to define these elements. Various approaches have been reported in the literature, some of which lead to very inconsistent results. The NCHRP recommends examining the derivatives of the power function by calculating the regression coefficients from time to time until they become constant (in this study, they are equal to 0.5). Once this point and the corresponding numbers of Nss cycles have been defined, the data from the cycles preceding this value can be excluded, and the model can only be defined with the remaining data corresponding to the second stretch at a constant slope.

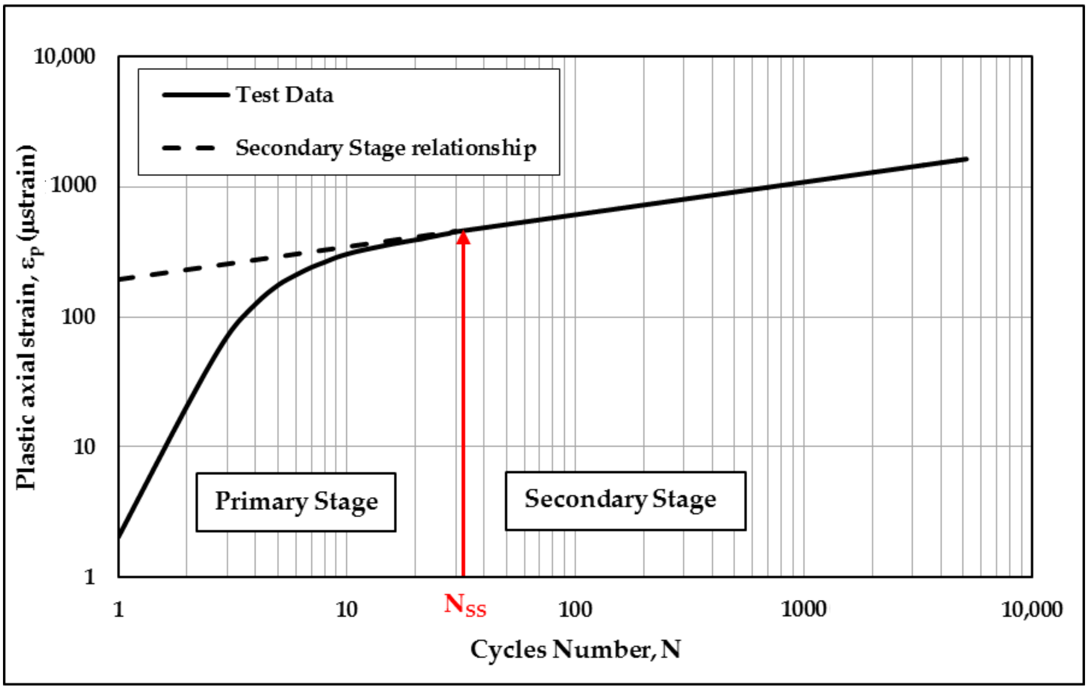


**Figure 8.** Typical dynamic creep curve.

## 3. Results and Discussion

### *3.1. Resilient Modulus*

Following the indications of the European standard EN 12697-26 (Annex C) and applying a pulsating load with the rest period shown in Figure 7, the rigidity of the thickened mixtures shown in Table 6 was obtained according to the four temperatures tested experimentally: 0, 10, 20, and 30 °C.

**Table 6.** Resilient Modulus Indirect Tension Test EN 12697-26, Annex C.

| | AR | | AC | | |
|---|---|---|---|---|---|
| **T (°C)** | **MR (MPa)** | **St. dv.** | **MR (MPa)** | **St. dv.** | **Var (%)** |
| 0 | 18,800 | 687 | 18,600 | 977 | +1.0 |
| 10 | 11,200 | 1180 | 11,100 | 1003 | +0.9 |
| 20 | 5630 | 673 | 5000 | 590 | +12.5 |
| 30 | 3400 | 382 | 2650 | 484 | +27.8 |

The comparison between the resilient modules (MR) of the two mixtures, depending on the temperature, shows the advantage of adding crumb rubber to the mixture, even in small quantities. Under high temperatures, the increase in stiffness is about 28%.

### *3.2. Dynamic Modulus*

For a more effective comparative analysis of the linear viscoelastic behaviour of the two asphalt mixtures, the specimens were subjected to repeated loads with amplitudes varying between a minimum and a maximum value using the sinusoidal law with variable frequencies of 10, 5, 1, and 0.1 Hz.

In this case, the maximum amplitudes of the loads guaranteed linear viscoelastic behaviour. The values of the complex modulus (dynamic modulus) obtained at the four temperatures and four frequencies are shown in Table 7, Figures 9 and 10. In the mixtures with the addition of crumb rubber, an increase in stiffness can be observed under medium and high temperatures at any frequency (and at low temperatures, only at the two lowest frequencies), while a slight decrease in stiffness can be observed at low temperatures and at the two highest frequencies.

**Table 7.** Dynamic Modulus (MR) Indirect Tension Test.

| | | AR | | AC | |
|---|---|---|---|---|---|
| **T (°C)** | **f (Hz)** | **MR (MPa)** | **St. dv.** | **MR (MPa)** | **St. dv.** |
| 0 | 10 | 20,719 | 1731.8 | 22,382 * | 1464.8 |
| | 5 | 20,190 | 1214.5 | 21,157 * | 2161.0 |
| | 1 | 18,756 | 1402.0 | 18,458 * | 2477.5 |
| | 0.1 | 15,031 | 1774.6 | 14,206 * | 2883.9 |
| 10 | 10 | 12,476 | 1005.1 | 9297 | 964.8 |
| | 5 | 11,152 | 1741.8 | 8311 | 1027.8 |
| | 1 | 8862 | 1553.3 | 6349 | 1105.4 |
| | 0.1 | 5954 | 1095.1 | 3791 | 777.0 |
| 20 | 10 | 7727 | 1136.5 | 7533 | 808.8 |
| | 5 | 6964 | 946.6 | 6662 | 534.2 |
| | 1 | 5272 | 661.5 | 5015 | 590.4 |
| | 0.1 | 3379 | 535.0 | 2988 | 363.5 |
| 30 | 10 | 5010 | 615.5 | 3713 | 858.9 |
| | 5 | 4451 | 587.1 | 3519 | 505.1 |
| | 1 | 3214 | 462.0 | 2428 | 415.1 |
| | 0.1 | 1984 | 309.8 | 1523 | 302.7 |

Note: * the average of 10 measurements for the two samples.

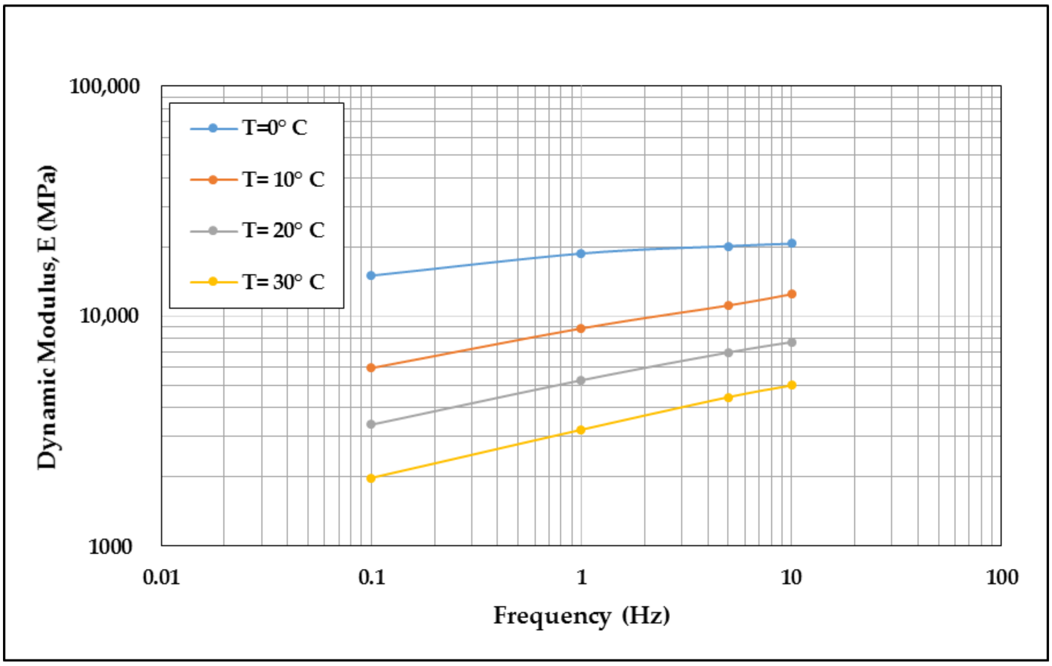


**Figure 9.** Dynamic modulus of 150 mm diameter Asphalt Rubber (AR) samples, at different temperatures and frequencies.

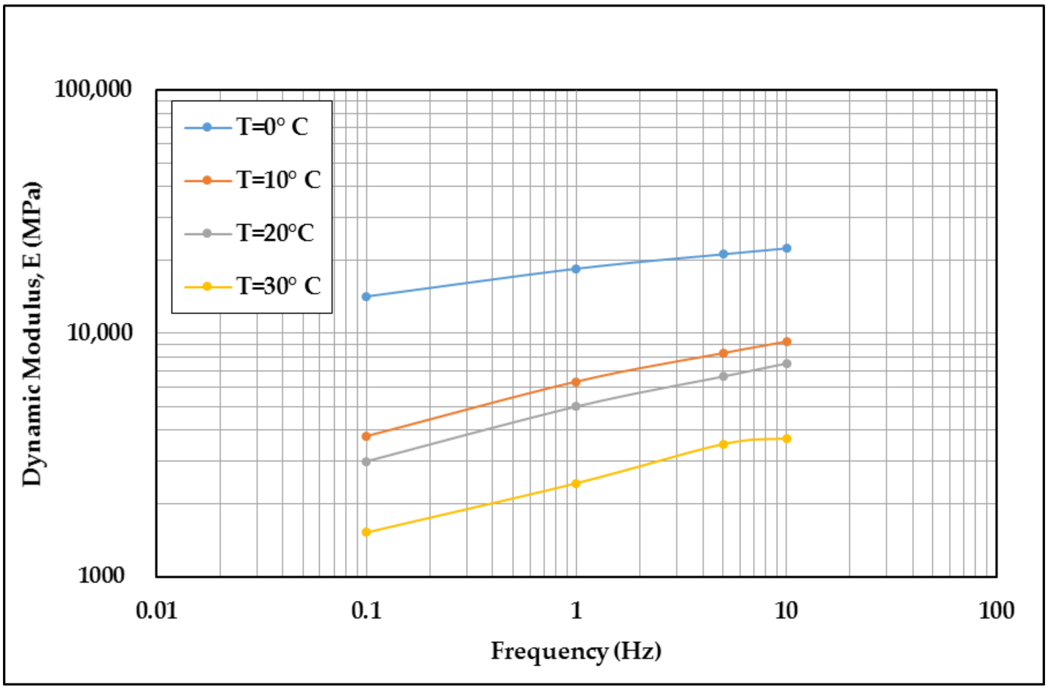


**Figure 10.** Dynamic modulus of 150 mm diameter "traditional surface course" (AC) samples, at different temperatures and frequencies.

At the lowest temperature and the highest frequency, the mixture with crumb rubber has a lower dynamic modulus of about 8%, while at the highest temperature and at the lowest frequency, the dynamic modulus is 30% higher.

Figure 11 shows some representative vectors of the complex modulus, particularly the values for the two mixtures AR and AC at temperatures of 0, 10, and 30 °C at a frequency of 10 Hz.

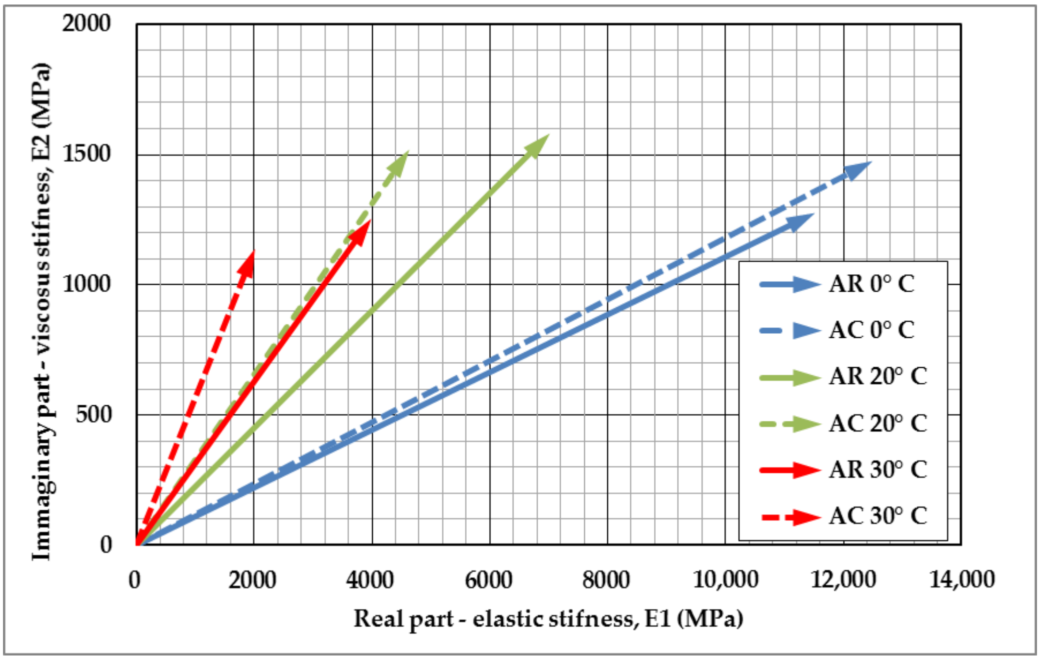


**Figure 11.** Complex modulus for AC and AR, at 0, 20, and 30 °C.

As shown in Figure 11, the phase angle is always lower in the AR sample. In particular, it is possible to observe that the range of phase angle variation between AR and AC is very high. At high temperature (30 °C), the reduction of phase angle of the AR mixture compared to the AC mixture is 41%. This reduction is less evident at low temperature (0 °C), where the reduction of phase angle in AR is 6% less of AC. In any case, this reveals more elastic behaviour compared to the traditional mixture. At high temperatures, the influence of the crumb rubber on the phase angle value becomes more evident. The values of the modulus, however, behave variably according to the temperatures (as highlighted in Table 7 and Figures 8 and 9). Since the crumb rubber is less sensitive to temperature, at low temperatures, the traditional mixture shows a higher modulus, as the whole binder has a higher stiffness. At higher temperatures, where the bitumen tends to soften, thereby making the mixture less rigid, the crumb rubber contributes to reducing this phenomenon due to its low thermal susceptibility. Indeed, the mixture including this component at higher temperatures has a higher modulus than that without crumb rubber.

### *3.3. Master Curve*

With the values of the dynamic modulus as a function of the test frequencies and temperatures (see Table 7), the master curves of the two mixtures were obtained (see Figure 12) along with the correlation parameters and the scale factors as a function of the temperatures (see Table 8). At low frequencies, the master curve of the mixture with crumb rubber has stiffness values higher than those of the mixture without the crumb rubber. At high frequencies, the mechanical behaviour of the mixtures changes. The curves cross each other, and the curve relative to the asphalt mix with the crumb rubber assumes lower stiffness values compared to the curve relative to the sample without the crumb rubber. The equations of the two curves can be used to determine the frequency at which this intersection occurs.

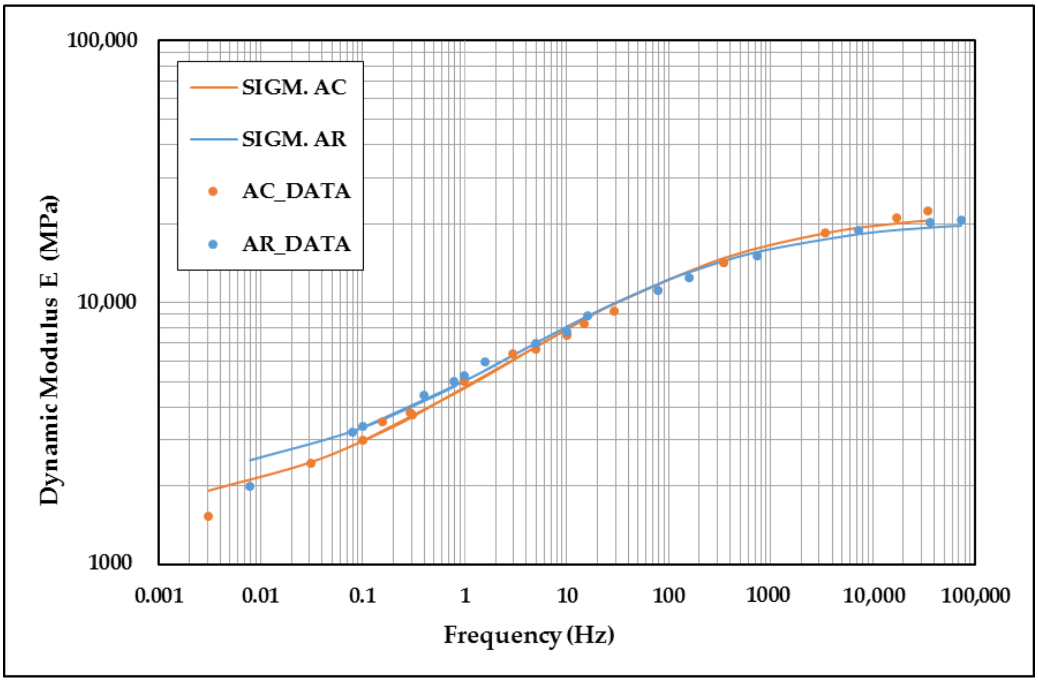


**Figure 12.** Master curve of AC and AR samples.

**Table 8.** Master curve parameters.

| Samples | $E_{min}$ (MPa) | $E_{max}$ (MPa) | δ (MPa) | α (MPa) | β | γ |
|---|---|---|---|---|---|---|
| AR | 1984 | 20,719 | 1984 | 18,735 | 1.638 | 0.910 |
| AC | 1523 | 22,382 | 1523 | 20,859 | 1.709 | 0.892 |

A linear interpolation can be used to calculate the stiffness values at each temperature. The parameters of the two linear function are reported in Figure 13.

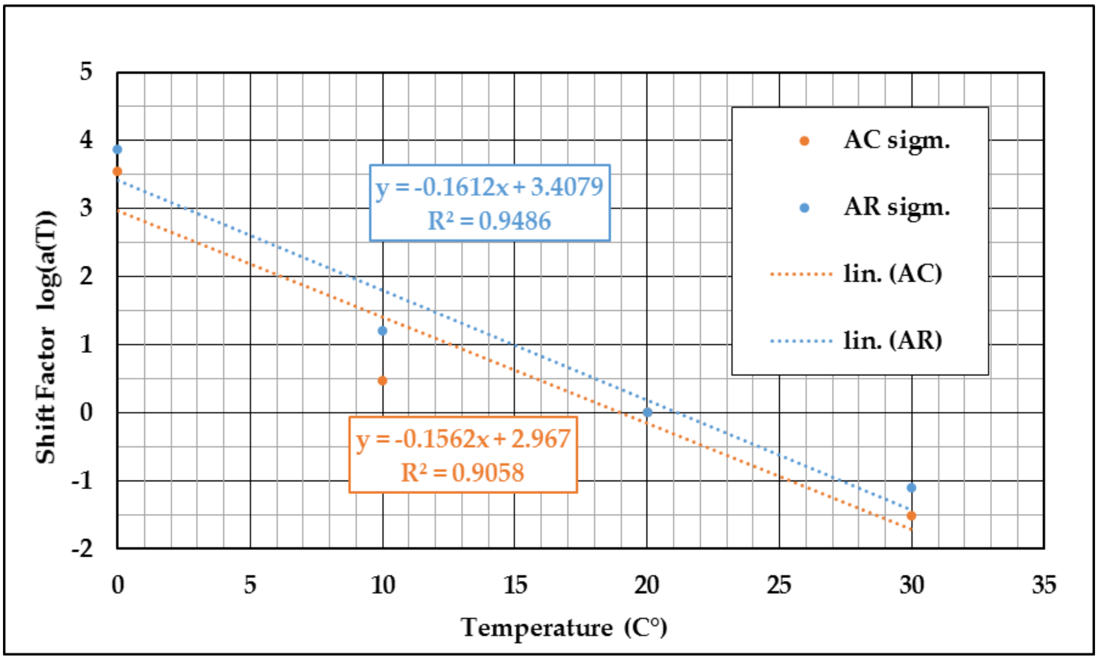


**Figure 13.** Shift factor.

## 3.4. Permanent Deformation (Rutting)

Figures 14 and 15 show the experimental results obtained from the tests carried out at 30 °C, indicating the maximum or total deformation (tot), the minimum or plastic deformation (min), and the Peak to Peak or reversible deformation (rev).

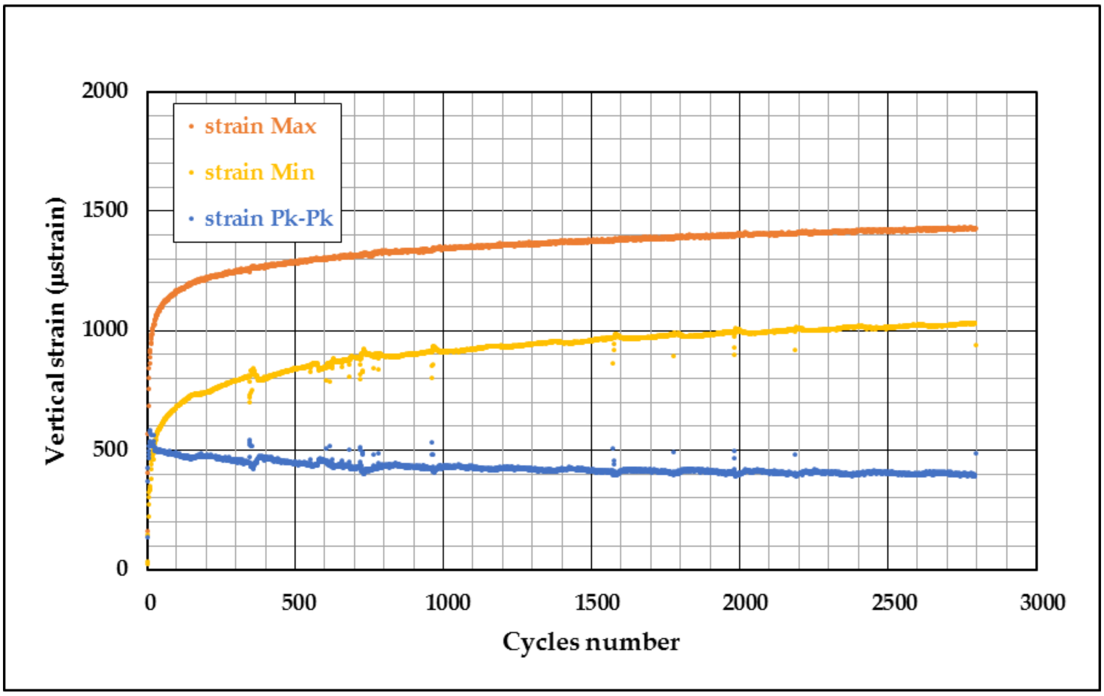


**Figure 14.** Dynamic creep curve for AR at 30 °C; strain Max $\varepsilon_{tot}$, strain Min $\varepsilon_{p}$, and strain Pk-Pk $\varepsilon_{rev}$.

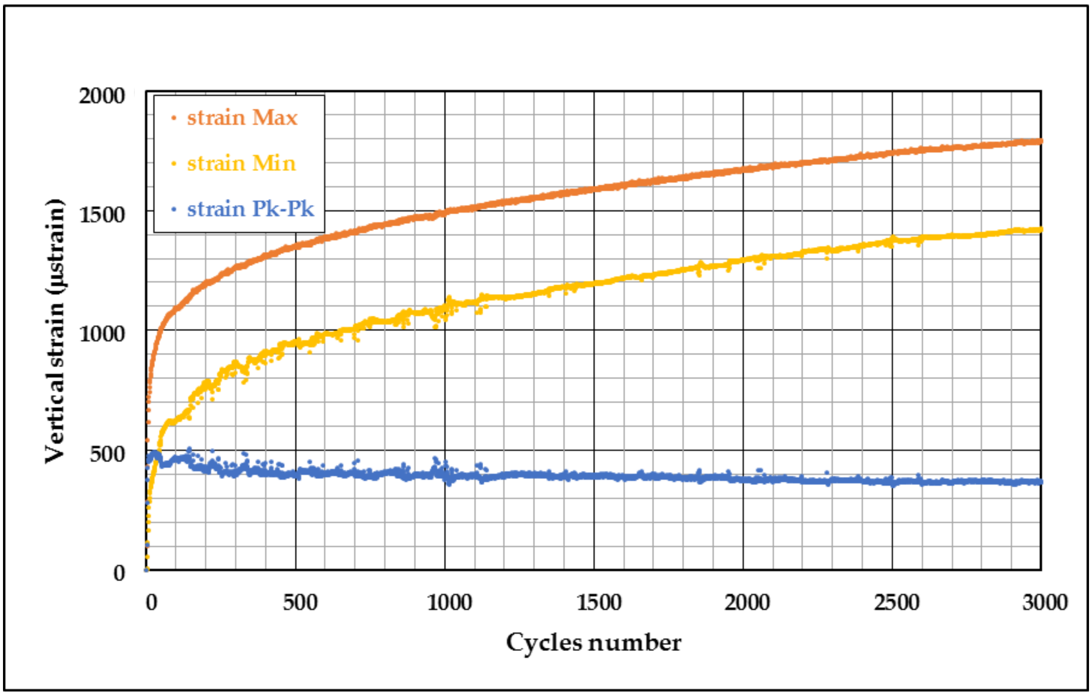


**Figure 15.** Dynamic creep curve for AC at 30 °C; strain Max $\varepsilon_{tot}$, strain Min $\varepsilon_p$, and strain Pk-Pk $\varepsilon_{rev}$.

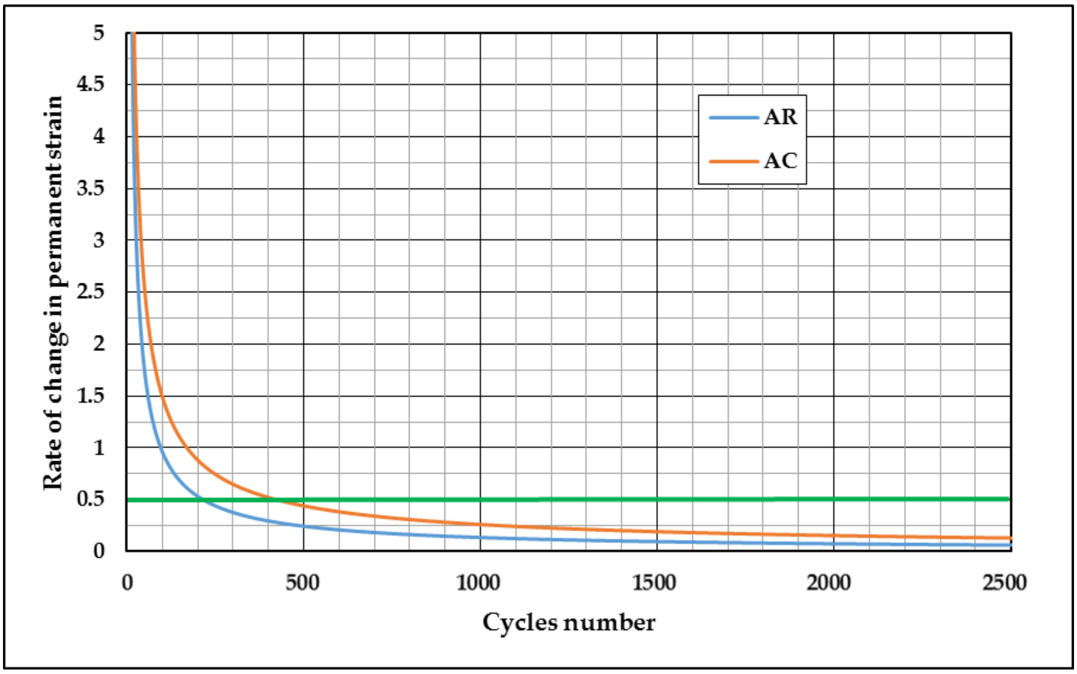


**Figure 16.** Rate of change in the permanent vertical strain vs. cycle number.

In this study, Nss, the number of cycles at which the primary section of the curve ends and the secondary section begins, was equal to 500 cycles with a gradient of variation less than 0.5 for the cumulative permanent deformations (see Figure 16). Figure 17 and Table 9 show the functions interpolating the secondary tract and the parameters of the equations.

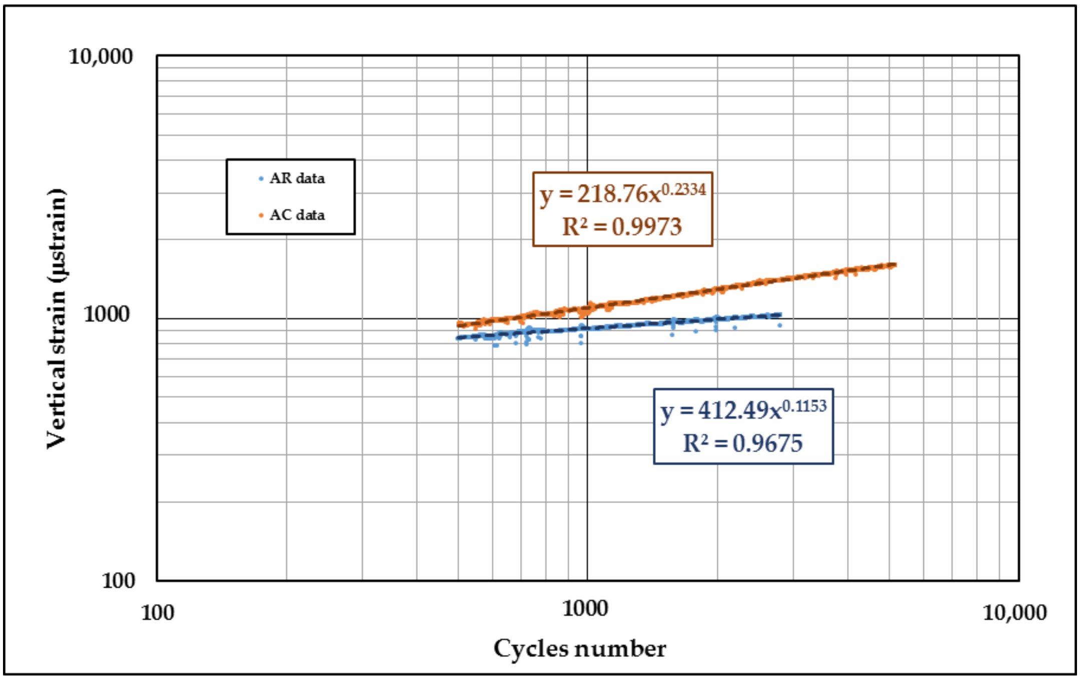


**Figure 17.** Secondary section of vertical strain vs. cycle number relationship, for AR and AC samples.

**Table 9.** Parameters for the secondary section correlation.

| Parameters | AC | AR |
|---|---|---|
| a | 218.7 | 412.49 |
| b | 0.2334 | 0.1153 |
| $\varepsilon_p$ (n =1000) | 1097 μstrain | 915 μstrain |
| $\varepsilon_p$ (n = 10,000) | 1877 μstrain | 1193 μstrain |
| $\varepsilon_{rev}$ (n = 1000) | 389 μstrain | 432 μstrain |

Analysing the results of dynamic creep tests with cyclic uniaxial compression loads, better behaviour was observed for the mixture with the addition of crumb rubber with regard to the accumulation of permanent deformations, which contribute to the degradation of asphalt pavements (after 10,000 cycles the permanent deformations of the AR mixture are 63% of those of the AC mixture). The line describing the secondary creep behaviour associated with variation in the volume of the thickened mixture, in a bilogarithmic diagram of εp vs. n° cycles, has a lower slope than the mixture without the crumb rubber.

## 4. Conclusions

This study highlighted how mixtures of aggregates (natural and artificial) and bitumen with the addition of crumb rubber obtained from end-of-life tyres provide an appealing alternative to traditional formulations. This study addressed the use of artificial aggregates to produce an asphalt concrete using steel slag together with recycled tyre rubber. These two materials correlate to several environmental concerns related to the disposal of waste resulting from their processing. The use of these materials in road pavements as secondary raw materials is an example of applying the concept of a circular economy. The use of these materials in pavements has not only provided environmental benefits but has also led to improvements in the mechanical properties of the mixtures. The benefits of using steel slag have already been highlighted elsewhere [33], such as their lower abrasion over time, which increases the life of the pavement surface course. The addition of crumb rubber leads to further improvements in structural performance considering the stresses of road loads.

Notably, our comparative analysis of the experimental results showed different mechanical behaviours under different test temperatures and frequencies. In particular, we observed variations in the dynamic modulus with temperature under a given frequency value in the two mixtures. It is also possible to identify the temperature value that will invert this behaviour. At high temperatures, the dynamic modulus of AR is always greater (up to 30%) than that of AC, while at low temperature, the dynamic modulus of AR is lower (up to 8%) than that of AC. This result is clear when comparing the master curves of the two thickened mixtures with the same volumetric properties.

Moreover, the phase angle is always lower in the AR mixture (up to 41%). This reveals more elastic behaviour compared to the traditional mixture.

The increase in stiffness at high temperatures found in the mixture containing the recycled tyre rubber led to a significant reduction in maximum tensile stress, thereby improving performance due to fatigue degradation. To accurately quantify this advantage, further laboratory tests will be needed to fully compare the fatigue degradation laws of the two mixtures.

Similar results emerge when comparing the accumulation of permanent deformation between the two mixtures. Laboratory tests have shown a better response from mixtures with crumb rubber. For such mixtures, the slope of the straight line for the accumulation of permanent deformation in the secondary creep is lower. The higher stiffness of the AR mixture at high temperatures reduces the unit compression deformations in the bitumen bonded layers and, consequently, reduces the accumulation of permanent deformations.

On the basis of the results of this research, a field experiment on road pavement that aims to verify the optimal behaviour of mixtures with crumb rubber and steel slag is underway.

In the future, it will be important to evaluate the performance of this mixture, which can be largely prepared with recovered products, scrap from metallurgical processes, and end-of-life tyres, relative to the amount of crumb rubber added. The future objective will be to identify the optimal quantities of crumb rubber and binder, with the latter being different from traditional blends, for use in very porous steel slag.

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