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Evaluation of Rheological Behavior, Resistance to Permanent Deformation, and Resistance to Fatigue of Asphalt Mixtures Modified with Nanoclay and SBS Polymer

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Abstract: Fatigue cracking and rutting are among the main distresses identified in flexible pavements. To reduce these problems and other distresses, modified asphalt mixtures have been designed and studied. In this regard, this paper presents the results of a study on rheological behavior and resistance to permanent deformation and to fatigue of four different asphalt mixtures: (1) with conventional asphalt binder (CAP 50/70); (2) with binder modified by nanoclay (3% NC); (3) with binder modified by styrene–butadiene–styrene polymer (SBS 60/85); and (4) with binder modified by nanoclay and SBS (3% NC + 2% SBS). For this analysis, the mixtures were evaluated based on complex modulus, permanent deformation tests, and fatigue tests (4PB, in the four-point bending apparatus), with the subsequent application of numerical simulations. The results obtained show a better rheological behavior related to greater resistance to permanent deformation for the mixture 3% NC + 2% SBS, which could represent an alternative for roads where a high resistance to rutting is required. Otherwise, on fatigue tests, higher resistance was observed for the SBS 60/85 mixture, followed by the 3% NC + 2% SBS mixture. Nevertheless, based on the results of the numerical simulations and considering the possibility of cost reduction for the use of the 3% NC + 2% SBS mixture, it is concluded that this modified material has potential to provide improvements to the road sector around the world, especially in Brazil.

Keywords: modified asphalt mixtures; nanomaterials; polymers; rheological behavior; fatigue cracking; permanent deformation

1. Introduction

Rutting and fatigue cracking can be considered the main distresses of flexible pavements. Besides reducing the safety and comfort of roadway users, it results in a need for increased vehicle maintenance.

In the search for better performance of asphalt coatings in relation to these problems, besides a higher strength to reduce other defects, the use of modified asphalt mixtures is one option available. In this context, asphalt mixtures modified with polymers have been applied successfully to road engineering since the 1970s, when they were first used in Europe [1]. In recent years, with the advent of nanotechnology, modification with the use of nanomaterials has also gained the attention of the scientific community. Studies carried out with asphalt nanocomposites have demonstrated the good performance and potential of these materials in the paving sector [2–12]. More recently, the behavior of asphalt binders and mixtures modified with polymers and nanomaterials have been investigated. The results obtained in this line of research have also been positive [13–21]. However, most studies have been limited to the binders, and there is a need for more investigations that consider the mixtures, taking into account the interaction between the binder, the granulometry, and the aggregate characteristics. This is important and should be considered because the total composition of the asphalt mixture will be in contact with traffic and weather during the pavement's lifespan, although the binder characteristics have a prominent role.

Thus, considering the high performance of mixtures modified with the addition of polymers and the potential for the application of nanomaterials to road engineering, the aim of this study was to carry out a comparative analysis of the rheological behavior and resistance to permanent deformation and to fatigue of four different asphalt mixtures: (1) a reference mixture, produced with a conventional asphalt binder (CAP 50/70: Petroleum Asphalt Cement with a penetration range between 5.0 and 7.0 millimeters) [22]; (2) a mixture with binder modified by nanoclay (3% NC), produced in laboratory [22]; (3) a mixture with binder modified by the polymer styrene–butadiene–styrene (SBS 60/85, with a minimum softening point of 60 °C and a minimum elastic recovery of 85%), produced industrially [23]; and (4) a mixture with binder modified by nanoclay and SBS (3% NC + 2% SBS), also produced in the laboratory [24]. This analysis was carried out through complex modulus and fatigue tests (4 PB, in the four-point bending apparatus) and permanent deformation tests (in the LCPC (Central Laboratory of Bridges and Roads/Laboratoire Central des Ponts et Chaussées) traffic simulator). Besides evaluating the behavior of the complex modulus and the phase angle of the mixtures with variations in the test load frequency and temperature, the rheological study allowed the prediction of the resistance of asphalt mixtures, especially with regard to rutting.

2. Materials and Methods

2.1. Materials

2.1.1. Aggregates and Granulometric Composition

The aggregates used in the production of the asphalt mixtures are of basaltic origin. The characterization of these materials is provided in Table 1. It can be observed that the properties listed are in conformity with the criteria established by the Superpave methodology (whenever applicable), verifying the suitability of the aggregates for the formulation of the mixtures.

Table 1. Characterization of aggregates [22].

Property	Standard	Result	Superpave Criterion
Bulk density of coarse aggregate	ASTM C 127 [25]	2.953 g/cm^3	n/a
Apparent density of coarse aggregate	ASTM C 127 [25]	2.880 g/cm^3	n/a
Absorption of coarse aggregate	ASTM C 127 [25]	0.8%	n/a
Bulk density of fine aggregate	DNER-ME 084 [26]	2.974 g/cm^3	n/a
Bulk density of powdery material	DNER-ME 085 [27]	2.804 g/cm^3	n/a
Angularity of coarse aggregate	ASTM D 5821 [28]	100%/100%	100%/100% min. [1]
Angularity of fine aggregate	ASTM C 1252 [29]	49.2%	45% min.
Flat and elongated particles	ABNT NBR 6954 [30]	9.6%	10% max.
Clay content (Sand equivalent)	AASHTO T 176 [31]	61.2%	50% min.
Hardness (Los Angeles abrasion)	ASTM C 131 [32]	11.6%	35–45% max.
Soundness	ASTM C 88 [33]	2.1%	10–20% max.
Deleterious materials	AASHTO T 112 [34]	0%	0.2–10% max.

Note: [1] e.g.,: 85%/80% means that 85% of coarse aggregate has one or more fractured faces and 80% has two or more fractured faces.

Table 2 presents the characterization of the hydrated lime used in the study, which corresponds to type CH-1 dolomitic.

Table 2. Characterization of the hydrated lime [22].

Property	Result
Loss on ignition	18.6%
Insoluble residue	1.9%
Carbon dioxide (CO_2)	2.5%
Calcium oxide (CaO)	45.1%
Magnesium oxide (MgO)	33.5%
Total non-volatile oxides (CaO + MgO)	96.5%
Total non-hydrated oxides	27.6%
Non-hydrated CaO	0.0%
Calcium (Ca)	32.2%
Magnesium (Mg)	20.2%
Bulk Density	3.00 g/cm^3

The formulation of the asphalt mixtures, based on the granulometric curve shown in Figure 1, was comprised of 43% gravel, 15.5% of crushed gravel, 40% of grit, and 1.5% of lime. This composition was established by the Leopoldo Américo Miguez de Mello Research and Development Center (CENPES/Petrobras), which aimed at obtaining mixtures with a high resistance to permanent deformation. The granulometric curve, aggregates, and hydrated lime were chosen because they were also applied in an experimental monitored road stretch still under evaluation by the authors.

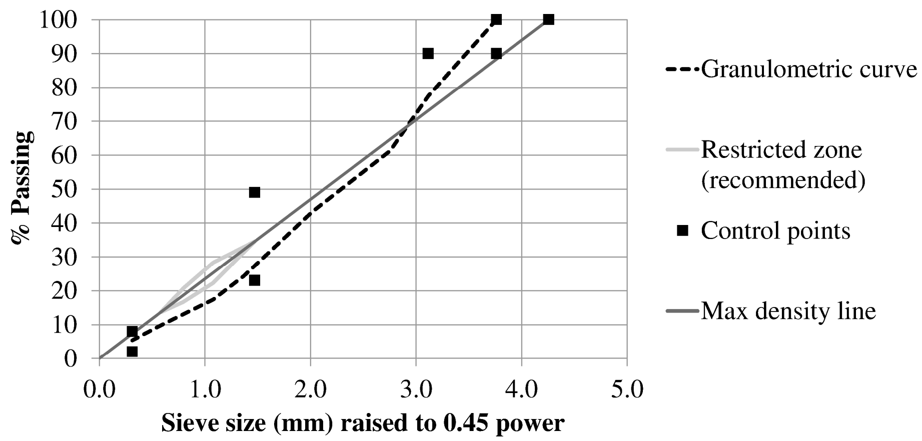

Figure 1. Granulometric curve for aggregate composition.

2.1.2. Conventional Asphalt Binder

The conventional asphalt binder used in this study was a CAP 50/70 (Petroleum Asphalt Cement with a penetration range between 5.0 and 7.0 millimeters), with PG 58–22 (Performance Grade 58-22). This binder was used in the production of the reference asphalt mixture and as a matrix for the modification of 3% NC and 3% NC + 2% SBS binders. The asphalt binder characterization is shown in Table 3.

2.1.3. Asphalt Binder Modified with SBS Polymer

The binder modified with the SBS polymer was industrially produced and supplied by Greca Asfaltos S.A. The characterization of this material is provided in Table 4.

Table 3. Characterization of the conventional asphalt binder (CAP 50/70).

Property	Unit	Standard	Result
Penetration	0.1 mm	ASTM D 5 [35]	57
Softening point	°C	ASTM D 36 [36]	47.9
Thermal susceptibility index	-	-	−1.44
Brookfield viscosity			
at 135 °C (*spindle* 21, 20 rpm)	cP	ASTM D 4402 [37]	290
at 150 °C (*spindle* 21, 50 rpm)			150
at 175 °C (*spindle* 21, 100 rpm)			60

Table 4. Characterization of the asphalt binder modified by styrene–butadiene–styrene (SBS) polymer (SBS 60/85).

Property	Unit	Standard	Result
Penetration	0.1 mm	ASTM D 5 [35]	50
Softening point	°C	ASTM D 36 [36]	73.0
Elastic recovery	%	ABNT NBR 15086 [38]	90
Apparent viscosity			
at 135 °C (*spindle* 21, 20 rpm)	cP	ASTM D 4402 [37]	1910
at 150 °C (*spindle* 21, 50 rpm)			640
at 175 °C (*spindle* 21, 100 rpm)			290

2.1.4. Modifiers

The organophillic nanoclay used in the modification of CAP 50/70 is known commercially as Dellite 67G. It has a particle size (dry) of 7–9 µm, a particle size after dispersion of 1 × 500 nm, and density of 1.7 g/cm^3. It has the following chemical composition: carbon (45.50%), silica (33.42%), aluminum (16.08%), iron (3.60%), chloride (0.80%), titanium (0.31%), potassium (0.27%), and strontium (0.02%). According to the results of thermogravimetry tests, this nanomaterial is thermally stable at temperatures below 262.4 °C [22].

The polymer SBS used as a modifier was Kraton D1101. It has a linear structure, with a polystyrene content of between 30% and 32%, and it was supplied in granules.

2.2. Methods

This study was carried out in seven stages.

Firstly, in Stage 1, the modification of the conventional binder and the characterization of the modified binders were carried out. In this stage, the binders 3% NC (modified by 3% nanoclay in relation to the weight of CAP 50/70) and 3% NC + 2% SBS (modified by 3% of nanoclay and 2% of SBS polymer) were obtained. The content of nanoclay was established as 3% based on the results of a previous optimization study developed by Melo [22], who evaluated the permanent deformation resistance of asphalt mixtures produced with binders modified by 1%, 2%, and 3% of nanoclay and verified a better performance for the 3% NC mixture. This nanoclay content was also identified in a recent study [39] as the most frequent value mentioned in the related literature. The addition of 2% SBS together with 3% NC was aimed at obtaining an asphalt material with elastic recovery. Low polymer content was adopted based on results reported by Pamplona et al. [20], who studied the modification with 2.5% of nanoclay and 2.5% of SBS, and also for economic reasons. It means the authors would like to produce a binder with reduced polymer content due to the cost of this material. However, another optimization of the modifier amount, including the SBS content, is recommended for future studies.

The inclusion of nanoclay and the polymer SBS in the base binder CAP 50/70 was carried out in a laboratory high shear mixer (Silverson, model L5M-A), and the modification procedures were defined based on studies reported in the literature [5,11,12,15–20]. The modification to produce the 3% NC binder was carried out at 150 °C, with a shearing speed of 5000 rpm and compatibilization period of 100 min. In this regard, the previously mentioned literature review study [39] also identified the following values as more frequent in similar studies: temperature of 160 °C, mixing speed of 4000 rpm, and mixing process duration of 120 min. This indicates this work is consistent with the literature, since the adopted and mentioned values are close. In the case of the 3% NA + 2% SBS binder, a modification temperature of 180 °C and a mixing period of 180 min were adopted, maintaining the shearing speed of 5000 rpm. These differences in time and temperature of modification were established in order to allow the complete dispersion of the modifiers. However, it should be noted that this can also do some influence on the binders and mixtures behavior. It is also interesting to mention that, as the binders were produced using a laboratory high shear mixer, for the reproduction of these materials in large quantities, with the same characteristics and aiming at the adequate dispersion of the modifiers, it would be necessary to use of an industrial high-shear mixer or another solution that provides the same results in terms of modifiers dispersion.

After production, the modified binders were then characterized according to the following properties: penetration (ASTM D 5 [35]), softening point ASTM D 36 [36]), elastic recovery (ABNT NBR 15086 [38]), phase separation (ABTN NBR 15166 [40]), and apparent viscosity (ASTM D 4402 [37]).

Stage 2 consisted of establishing the design binder contents of the asphalt mixtures, beginning the part of this study that aims to evaluate the influence of the modifiers when interacting in the asphalt mixtures. For this, the Superpave mix design method was applied, according to the standards AASHTO M 323 [41] and AASHTO R 35 [42], with the use of a gyratory compactor. Considering a maximum nominal size of the aggregate of 19 mm and a heavy volume of traffic, the binder contents of the design correspond to the following dosage criteria that were met simultaneously: (1) percentage of void volume in Ninitial (9 spins, Vv@Ninitial) > 11.0%; (2) percentage of void volume in Ndesign (125 spins, Vv@Ndesign) = 4.0%; (3) voids in mineral aggregate (VMA) ≥ 13.0%; (4) voids filled with asphalt (VFA) between 65% and 75%; and (5) dust to effective binder ratio between 0.8 and 1.6. It is important to note that, during the design mix and the subsequent steps, the asphalt mixtures were produced and compacted at temperatures of which the binder viscosities corresponded respectively to 0.17 Pa.s and 0.28 Pa.s.

In Stage 3, after the design binder content definition, for each mixture, three plates were molded with one having dimensions of 60 × 40 × 9 cm (for sawing and obtaining prismatic specimens for the complex modulus and fatigue tests) and two plates having dimensions of 50 × 18 × 5 cm (for the permanent deformation tests). This molding was carried out at the LCPC compacting table, following the recommendations of the French standard AFNOR NF P 98–250–2 [43] for a heavy traffic highway. Plates with dimensions 60 × 40 × 9 cm were then sawn, and five specimens with dimensions close to 6.3 × 5.0 × 40.0 cm were obtained from each plate.

In Stage 4, the rheological behavior characterization of the different mixtures was evaluated. This characterization was based on complex module tests, carried out in a four-point bending test machine, following the recommendations of the European standard EN 12697–26 [44]. For each asphalt mixture, two specimens produced in the previous stage were tested. These tests were carried out under alternating bending with the application of sinusoidal loading keeping the deformation amplitude at 50 $\mu\varepsilon$. Temperatures of 0 °C, 5 °C, 10 °C, 15 °C, 20 °C, 25 °C, and 30 °C with load frequencies of 0.1 Hz, 0.2 Hz, 0.5 Hz, 1 Hz, 2 Hz, 5 Hz, 10 Hz, and 20 Hz, were evaluated. For the interpretation of the results, master curves and black spaces were analyzed. The master curves were built in the reference temperature of 20 °C, based on the TTS principle (time–temperature superposition) and by applying the Williams–Landel–Ferry equation, of which the constants were calculated using the Viscoanalyse software (developed by the LCPC).

In Stage 5, the resistance to permanent deformation of the four different mixtures was evaluated. In this stage, tests were performed in the French traffic simulator following the standard EN 12697–22+A1 [45]. These tests were carried out at 60 °C, with the application of a single axle with single wheel load, intensity of 5 kN, tire inflation pressure of 0.6 MPa, and frequency of 1 Hz.

The development of rutting recesses was measured after the following numbers of cycles: 100, 300, 1000, 3000, 10,000, and 30,000. The results of these tests after 30,000 cycles were compared to the limits established by the French [46] and European [47] guidelines.

Stage 6 consisted in evaluating the fatigue resistance of the asphalt mixtures. As mentioned for the rheological characterization, fatigue tests were also carried out in four-point bending test machine but followed the recommendations of the European standard EN 12697–24 [48]. In this stage of the study, about fifteen specimens were tested for each mixture under controlled deformation at deformation levels between 80 and 375 με (μm/m). These tests were performed considering a frequency of 10 Hz (simulating the vehicles speed, in practice, of 72 km/h [49]) and at the temperatures between 15 °C and 20 °C (defined according to the average temperature of the region of this study). As a rupture criterion, it adopted a reduction of 50% of the initial complex modulus. At the end of the tests, it was possible to obtain the fatigue models of the mixtures, as presented in Equation (1):

$$N = k(\mu\varepsilon)^{-n}, \tag{1}$$

where N = number of cycles (loading applications) until asphalt concrete reaches 50% of initial stiffness; $\mu\varepsilon$ = maximum tensile strain applied on the material; and k, n = constants mainly dependent on stiffness and asphalt content of the mixture.

After that, in Stage 7, numerical simulations were carried out, considering the structures presented in Figure 2 (S1, S2, S3, and S4 with the same subgrades, subbases, and bases but different asphalt layers). These structures were chosen for analysis and comparisons because they represent variations of the structure S1, which was constructed in a roadway in the region where the authors developed this work and where the asphalt surface presents early distresses. This road stretch with the structure S1 is being monitored by the authors in other projects. The simulations aimed at estimating the tensile strain suffered by the lower fibers of the asphalt layers during the passage of vehicles (at 170 mm depth, according Figure 2). Thus, in the simulations, it was considered the load configuration shown in Figure 3, at the speed of 20 m/s and the fatigue tests' temperature. Applying this stage results to the fatigue models obtained in Stage 6, it was possible to estimate the lifespan in terms of asphalt concrete fatigue fracture of each structure analyzed. This procedure represents an initial estimation for comparisons, and lab to site shift factors can be applied in future researches. It also should be noted that the viscoelastic behavior of the asphalt mixtures was considered in the simulations by using the Huet-Sayegh rheological parameters, obtained from the results of Step 5.

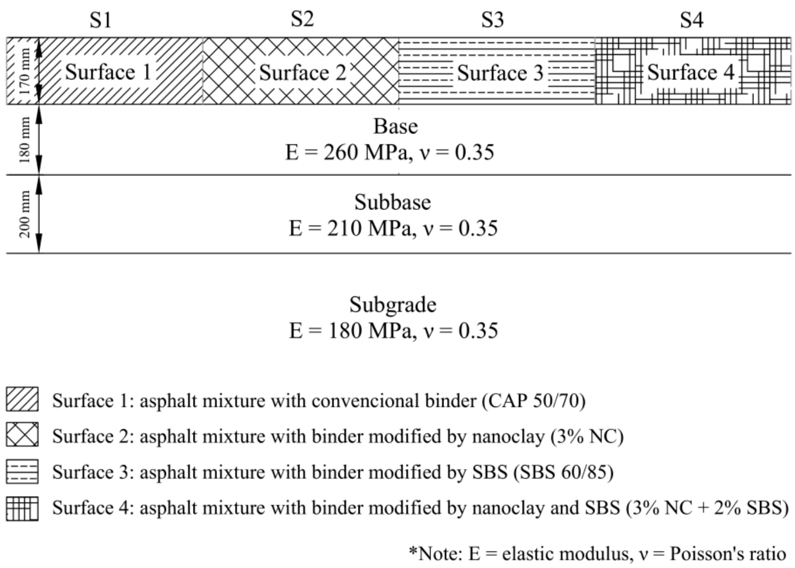

Figure 2. Pavement structures evaluated in the numerical simulations.

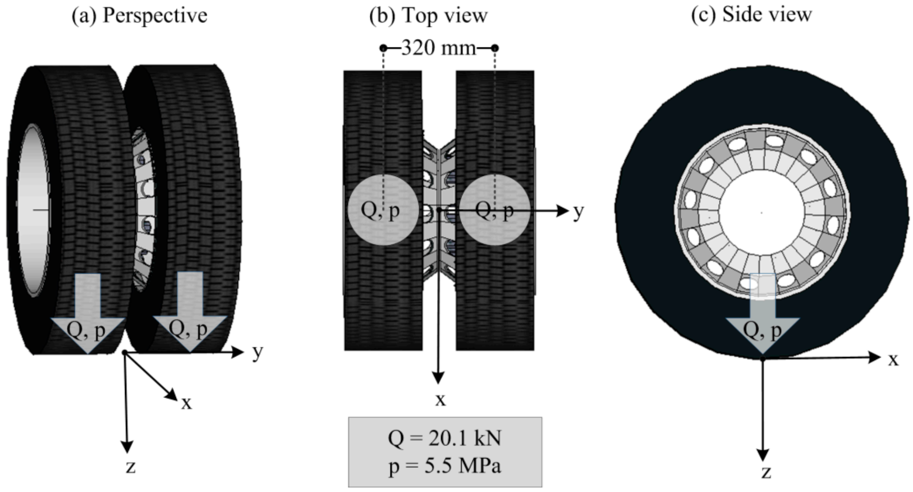

Figure 3. Load configuration considered for the numerical simulations.

3. Results and Discussion

3.1. Characterization of Modified Binders

Results of the laboratory modified asphalt binders' characterization can be observed in Table 5.

Table 5. Characterization of asphalt binders 3% NC and 3% NC + 2% SBS.

Propriety	Unit	Standard	Asphalt Binder	
			3% NC	3% NC + 2% SBS
Penetration	0.1 mm	ASTM D 5 [35]	55	36
Softening point	°C	ASTM D 36 [36]	50.2	56.9
Elastic recovery	%	ABNT NBR 15086 [38]	6	49
Phase separation (24 h/48 h)	°C	ABNT NBR 15166 [40]	1.0/-	0.5/0.8
Apparent viscosity	cP	ASTM D 4402 [37]		
at 135 °C (*spindle* 21, 20 rpm)			410	760
at 150 °C (*spindle* 21, 50 rpm)			210	370
at 175 °C (*spindle* 21, 100 rpm)			90	160

In comparing the results in Table 5 with those provided for the empirical characterization of CAP 50/70 (Table 3) and SBS 60/85 (Table 4), it can be observed that, as expected and also demonstrated by other authors in similar studies [13–21], the binders modification caused a penetration decrease and a softening point increase. Notable among these results are the relatively low penetration obtained for the binder 3% NC + 2% SBS and the relatively high softening point for the binder SBS 60/85. This reflects gains in the stiffness in the first case in contrast with gains related to the sensitivity to high temperatures in the second case. These characteristics indicate that the asphalt mixtures formulated will be very resistant to permanent deformation. Thus, according to the results obtained in the empirical characterization of the binders, better performance is expected in relation to the permanent deformation for the mixtures SBS 60/85 and 3% NC + 2% SBS. However, it is important to highlight that the behavior prediction of asphalt mixtures based on the modified binders' empiric characterization shows high limitations.

In relation to the elastic recovery property, the obtainment of a higher value is observed for the binder SBS 60/85. This result is due to the relatively high content of the elastomeric polymer added to the material (approximately 4%). On the other hand, the low elastic recovery of the binder 3% NC is related to the fact that, in the modification, the nanoclay does not function as an elastomeric product.

With regard to storage stability, the tests carried out with binders modified by nanoclay (3% NC and 3% NC + 2% SBS) revealed that they do not show significant phase separation. By way of comparison, it can be noted that the results are below the maximum limit (5 °C) established by the Brazilian Specification for Asphalt–Polymer [50].

In the same way, the results obtained for the modified binders' viscosity also met the limits established by the Brazilian Specification for Asphalt–Polymer [50] despite the considerable increases in comparison to CAP 50/70 (conventional).

3.2. Definition of Binder Contents

Table 6 shows the design binder contents obtained for the mixtures.

Table 6. Design binder contents of different asphalt mixtures.

Asphalt Binder	Design Binder Content (%)
CAP 50/70	4.4
3% NC	4.1
SBS 60/85	4.5
3% NC + 2% SBS	4.3

As can be observed in Table 6, the nanoclay addition to the conventional binder can lead to a reduction in the design binder content. In contrast, with the polymer SBS addition, higher binder content is required. Thus, the intermediate result obtained for the 3% NC + 2% SBS mixture in the design mix study demonstrates the combination of the nanoclay positive effect, in terms of mixture workability, with the negative effect of the presence of the polymer.

Also concerning the different binder contents obtained for the mixtures it should be noted that their rheological behavior, resistance to permanent deformation and resistance to fatigue (whose results will be presented later) can be influenced because the mixtures were not produced with exactly the same binder content (an exception to this comment are the mixtures CAP 50/70 and 3% NC, which were both produced with the same binder content = 4.4%). However, it's important to highlight the previously mentioned variation in the design binder contents is meeting the limit values allowed in the field (± 0.3%), based on Brazilian current construction specifications.

3.3. Plate Molding

Table 7 reports the results of void volume percentages checked for the specimens used in the complex modulus and fatigue tests. Table 8 also reports the results of void volume percentages but, in this case, obtained for the plates used in the permanent deformation tests.

Table 7. Volumetric characterization of specimens used in complex modulus and fatigue tests.

Asphalt Mixture	Void Volume (%)	
	Mean	Standard Deviation
CAP 50/70	4.53	0.87
3% NC	3.85	0.38
SBS 60/85	4.62	0.39
3% NC + 2% SBS	3.83	0.54

It should be noted that the plates molded to obtain specimens (modulus and fatigue tests) and those molded for permanent deformation tests were produced by aiming at 4% void volumes, as defined in the design mix study. However, during the compaction procedure, carried out at the LCPC compacting table (compaction procedure different from that used in the mixtures design step), it was found to be difficult to control the final thickness of the plates, hindering the precise obtainment of the void volumes equal to 4%. Even so, considering that the specimens and plates met the compaction degrees admitted in the field in Brazil (between 97% and 101%), they were considered suitable for use in the tests.

Table 8. Volumetric characterization of plates used in permanent deformation tests.

Asphalt Mixture	Plate	Void Volume (%)
CAP 50/70	P1	6.13
	P2	4.50
3% NC	P1	6.55
	P2	6.68
SBS 60/85	P1	5.87
	P2	5.57
3% NC + 2% SBS	P1	5.37
	P2	5.69

3.4. Rheological Characterization

Figure 4 shows the master curves obtained for the asphalt mixtures evaluation. In general, the positioning of the curves indicates higher stiffness for the modified asphalt mixtures. An exception, however, is the mixture SBS 60/85 which, at high load frequencies (equivalent to low temperatures), shows a very similar rheological behavior to the conventional mixture. In comparison with the other studied mixtures, the mixture 3% NC + 2% SBS showed notably high values for the complex modulus. This characteristic can be considered as an indication that the mixture will have greater resistance to permanent deformation but can be hampered in terms of fatigue resistance.

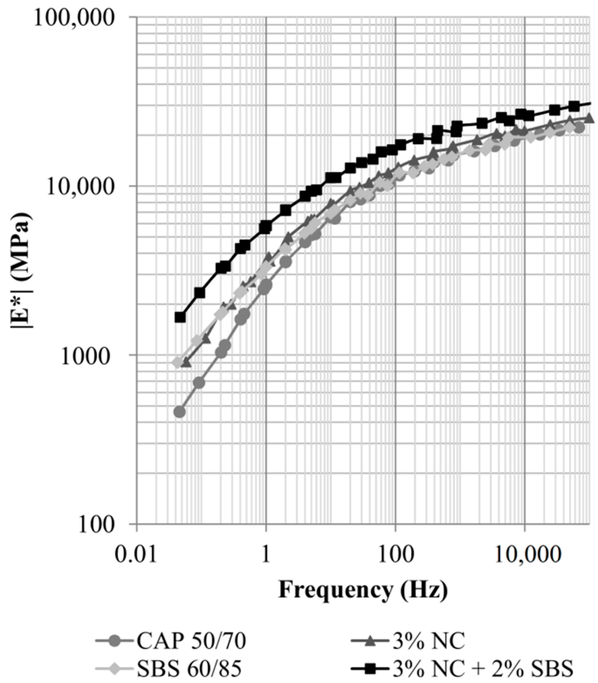

Figure 4. Master curves (reference temperature = 20 °C).

In addition, an analysis of the master curves also reveals the lower frequency susceptibility of the modified mixtures based on the slopes of the curves. In the field, this would mean that the stiffness of these mixtures should be less sensitive to variations in the traffic speed, suggesting a high potential for their use along segments with a steep slope and slow traffic. In this regard, it is possible to establish a behavior hierarchy, where the mixture modified with both modifiers is at the top, followed by SBS 60/85, 3% NC, and the conventional mixture.

It also can be noted in Figure 4 that the gains related to the frequency susceptibility of the modified mixtures are more significant at lower load frequencies (equivalent to higher temperatures). This may be considered a positive aspect in relation to the permanent deformation phenomenon in regions of tropical climate and on highways submitted to slow loads or in mountainous regions.

Figure 5 shows the black spaces for the different asphalt mixtures.

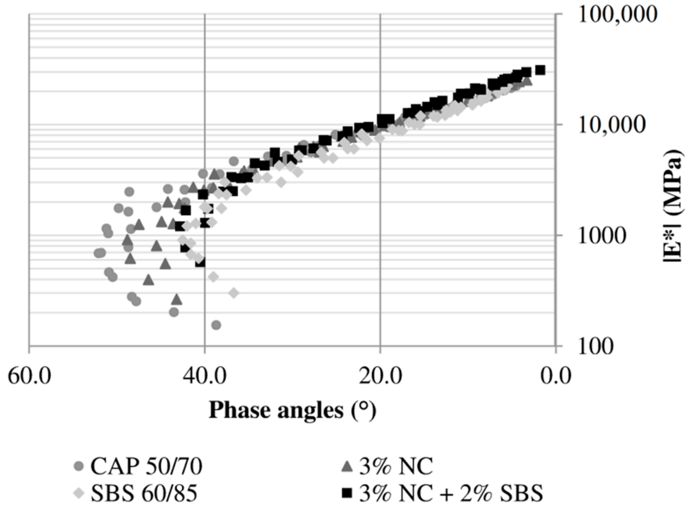

Figure 5. Black spaces.

It can be noted in Figure 5 that the modified mixtures present an aspect of graphic shortening (in relation to the phase angle) when compared with the conventional mixture. This shortening illustrates the obtainment of smaller phase angles and reflects the more elastic behavior of these mixtures. Of these, it can be observed that the 3% NC + 2% SBS mixture has a higher concentration of low phase angles, followed by the SBS 60/85 mixture. The elastic behavior of them, both modified with SBS, reflects the influence of the presence of an elastomeric polymer in the binders. Considered in isolation, this would suggest a better performance of these mixtures in relation to their resistance to permanent deformation and to fatigue cracking.

3.5. Resistance to Permanent Deformation

In Figures 6 and 7, the curves obtained in the permanent deformation tests and the results for the rutting depth (%) of mixtures after 30,000 cycles are provided, respectively.

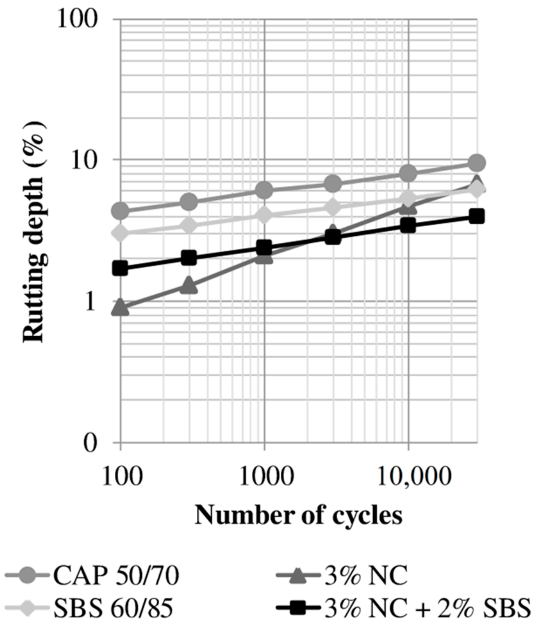

Figure 6. Curves obtained in permanent deformation tests.

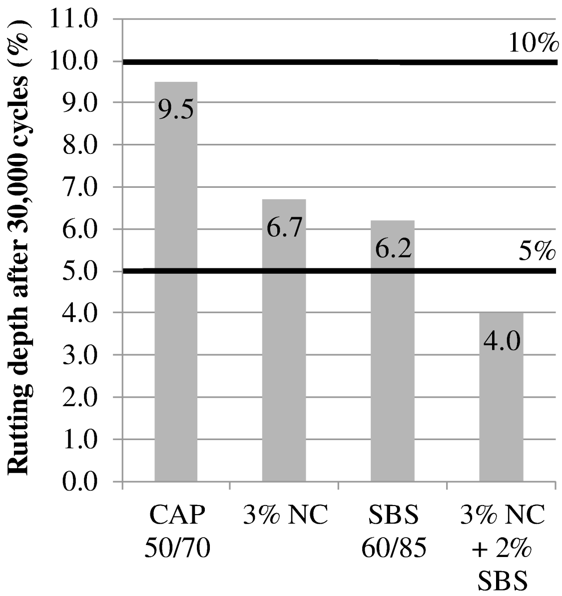

Figure 7. Results for rutting depth (%) after 30,000 cycles.

According to the results shown in Figures 6 and 7, the resistance to permanent deformation, considering 30,000 load cycles was highest for the 3% NC + 2% SBS mixture, followed by the SBS 60/85, 3% NC, and conventional mixtures. In proportional terms, in practice, it is estimated that the replacement of the conventional mixture containing the two modifiers would reduce the rutting depth in the field by up to 58%. Considering the replacement of the reference mixture with the SBS 60/85 and 3% NC mixtures, the corresponding reductions could reach up to 35% and 29%, respectively.

The best behavior in terms of permanent deformation observed for 3% NC + 2% SBS mixture is in agreement with the performance prediction carried out in the empirical characterization of the binders (lower penetration) and the rheological study of the mixtures (higher stiffness and lower phase angles). In the same way, the results for the phase angles were also effective in the prediction of the performance hierarchy of the mixtures in relation to resistance to permanent deformation.

About the rate of deformation increase, which can be measured by the WTS (wheel-tracking slope) parameter, Figure 6 shows similar behaviors between the mixtures CAP 50/70, SBS 60/85, and 3% NC + 2% SBS. On the other hand, it is observed that the 3% NC mix exhibits a lower initial deformation with a higher deformation increase rate. In this sense, considering about 3000 load cycles, for example, the deformation level was the same for the mixtures 3% NC and 3% NC + 2% SBS; however, due to a higher deformation increase of the mixture modified only by nanoclay, the scenario becomes different at the end of the test. High rates of deformation increase are considered a negative characteristic for asphalt mixtures performance, and according to the results obtained in this work, simultaneous action of SBS can improve this behavior of the 3% NC mixture.

Regarding the maximum limits for the rutting depth established by the French [46] and the European [47] specifications, all of the asphalt mixtures under study met the 10% criterion for 30,000 load cycles. However, only the 3% NC + 2% SBS mixture satisfied the more restrictive criterion, corresponding to 5%.

3.6. Resistance to Fatigue Cracking

The results of the fatigue tests are shown in Figure 8. Below the graph, the representative fatigue models of the mixtures are presented alongside the respective coefficients of determination (R^2) and the specific deformations for 10^6 cycles (ε_6). The models relate the tensile strain ($\mu\varepsilon$) applied on the specimens to the number of cycles (N, loading applications) until asphalt concrete reaches 50% of initial stiffness.

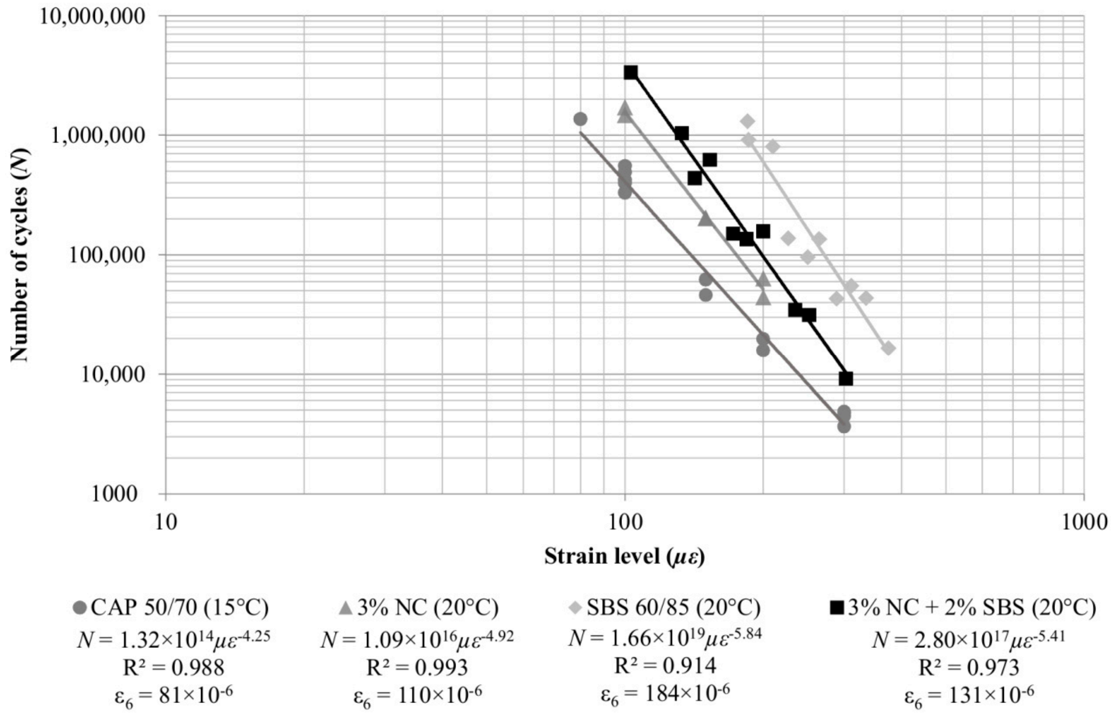

Figure 8. Curves obtained in fatigue tests.

For the conditions adopted in the tests carried out in this research, from the curves shown in Figure 8, it can be observed the higher fatigue strength of the SBS 60/85 mixture, followed by the 3% NC + 2% SBS, 3% NC and conventional mixtures.

Concerning the fatigue behavior prediction based on the rheological study, it can be noted that it was partially accomplished. In this sense, the superior behavior of the SBS 60/85 and 3% NC + 2% SBS mixtures was expected, due to the greater predominance of the elastic behavior (lower phase angles). However, according to the respective results, it was expected that the best performance would be of the 3% NC + 2% SBS mixture, in comparison to the SBS 60/85, which did not occur. Probably, in terms of fatigue, the mixture modified by nanoclay and SBS was impaired due to the considerable increase in the stiffness of the material, which was verified in the empirical characterization of the binder (low penetration) and also in the rheological study of the mixtures.

3.7. Results of Numerical Simulations

Table 9 presents the numerical simulation results applied to the fatigue models.

Table 9. Numerical simulations results applied to the fatigue models.

Structure/Asphalt Surface	Strain (με)	Fatigue Model	Number of Cycles (N)
S1/Surface with CAP 50/70	65	$N = 1.32 \times 10^{14}\ \mu\varepsilon^{-4.25}$	2.60×10^6
S2/Surface with 3% NC	75	$N = 1.09 \times 10^{16}\ \mu\varepsilon^{-4.92}$	6.49×10^6
S3/Surface with SBS 60/85	80	$N = 1.66 \times 10^{19}\ \mu\varepsilon^{-5.84}$	1.28×10^8
S4/Surface with 3% NC + 2% SBS	61	$N = 2.80 \times 10^{17}\ \mu\varepsilon^{-5.41}$	6.15×10^7

From the results presented in Table 9, the following hierarchy can be established for the asphalt mixtures, in terms of fatigue lifespan, starting by the longest one: structure S3 (surface with SBS 60/85 mixture), structure S4 (surface with 3% NC + 2% SBS mixture), structure S2 (surface with 3% NC mixture), and structure S1 (surface with conventional mixture).

First, comparing the structures S1 and S2, it could be observed that the use of the 3% NC mixture, in substitution to the conventional one, would represent an increase of 1.5 times in the fatigue lifespan of the surface. When the same comparison is carried out between the S1 and S4 structures, it is noted that the simultaneous addition of the polymer to the nanoclay modified mixture would enhance these gains to about 23 times. However, despite the high performance of the 3% NC + 2% SBS mixture and keeping the thicknesses of the layers, it is noted that the use of the SBS 60/85, in substitution to the mixture modified simultaneously by nanoclay and polymer, would double the fatigue lifespan of the surface. In this sense, looking for the equivalent performance of these two mixtures, it requires an increase in the thickness of the 3% NC + 2% SBS modified surface. Even so, considering a possibility of cost reduction from the substitution of the SBS 60/85 mixture by the 3% NC + 2% SBS (due to the lower polymer content), it is expected that this substitution, besides being technically feasible, could also be economically viable. This finding highlights the application potential of the 3% NC + 2% SBS mixture, which may present as a competitive alternative to the other mixtures studied.

4. Conclusions

According to the objective previously defined, this study enabled four different asphalt mixtures to be compared in terms of their rheological behavior, resistance to permanent deformation, and resistance to fatigue cracking. The mixtures were prepared with different binders as follows: conventional (CAP 50/70), modified with nanoclay (3% NC), modified with SBS polymer (SBS 60/85), and modified with both nanoclay and SBS (3% NC + 2% SBS).

Based on the results obtained in this study, the mixture modified with the binder 3% NC + 2% SBS showed a better rheological behavior and a higher resistance to permanent deformation. This mixture presented the highest values for the complex modulus and the lowest phase angles, which is considered an indication of higher resistance to rutting. In the permanent deformation tests, it showed the lowest percentage of rutting depth, thus confirming the predictions based on the rheological study.

On the other hand, considering the fatigue tests results, a better resistance was observed in the SBS 60/85 mixture. In this respect, the predictions made according to the rheological behavior were partially verified. Even so, on the next stage of this study, numerical simulations of pavement structures demonstrated the use of the 3% NC + 2% SBS surface as a replacement for the SBS 60/85 being technically viable (with thickness adjustments), and considering the possibility of reducing costs from this substitution, it could also be economically viable. In this sense, considering the costs of the materials used in the research, it is believed that the mixture modified by nanoargila and SBS (with lower content) would be cheaper than that modified by a higher SBS content. However, economic viability with specific comparisons should be verified in future studies.

Thus, it is concluded that the use of polymers science together with the application of nanotechnology can lead to great advances in the area of modified asphalt mixtures for use on roadways where a higher performance of the paving materials is required. Especially in relation to rutting, it is highlighted that this alternative has great potential to provide improved asphalt surfaces, mainly in regions of tropical climate and on highways submitted to slow heavy traffic.

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