

Evaluation of rheological behavior and resistance to permanent deformation of asphalt mixtures modified with nanoclay and SBS polymer

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ABSTRACT

Rutting is one of the main distresses identified in flexible pavements, especially in regions with tropical climate and on highways submitted to slow heavy traffic. In order to reduce this problem, beyond other distresses, modified asphalt mixtures have been designed and studied. The modifiers that can be added include polymers (a consolidated technology for highways) and nanomaterials (a new but promising technology). In this context, this paper presents the results of a study on rheological behavior and resistance to permanent deformation of four different asphalt mixtures: with conventional asphalt binder (reference mixture, CAP 50/70); with binder modified with nanoclay (3% NC); with binder modified with styrene-butadiene-styrene polymer (SBS 60/85), and with binder modified with both nanoclay and SBS (3% NC + 2% SBS). For this analysis, the mixtures were evaluated based on complex modulus (4PB) and permanent deformation (French traffic simulator) tests. The results showed a better rheological behavior with greater resistance to permanent deformation for the mixture 3% NC + 2% SBS. This mixture could represent a competitive alternative for roads where a high resistance of the asphalt mixture is required to avoid the rutting, especially in regions of tropical climate and on highways submitted to slow heavy traffic.

Keywords: Modified asphalt mixtures, nanomaterials, polymers, rheological behavior, permanent deformation.

1. INTRODUCTION

In the search for better performance of asphalt surfaces in relation to rutting, besides a higher strength to reduce other distresses, 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 [2-9]. 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 [10-15]. 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.

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

1 of this study was to carry out a comparative analysis of the rheological behavior and
 2 resistance to permanent deformation of four different asphalt mixtures: (1) a reference
 3 mixture, produced with a conventional asphalt binder (CAP 50/70 – Petroleum Asphalt
 4 Cement with a penetration range between 5.0 and 7.0 millimeters) [16]; (2) a mixture with
 5 binder modified with nanoclay (3% NC), produced in laboratory [16]; (3) a mixture with
 6 binder modified with the polymer styrene-butadiene-styrene (SBS 60/85, with a minimum
 7 softening point of 60 °C and a minimum elastic recovery of 85%), produced industrially [17];
 8 and (4) a mixture with binder modified with nanoclay and SBS (3% NC + 2% SBS), also
 9 produced in the laboratory [18]. This analysis was carried out through complex modulus tests
 10 (4 PB, in the four point apparatus) and permanent deformation tests (in the LCPC –
 11 *Laboratoire Central des Ponts et Chaussées* traffic simulator). Besides evaluating the
 12 behavior of the complex modulus and the phase angle of the mixtures with variations in the
 13 test load frequency and temperature, the rheological study allowed the prediction of the
 14 resistance of asphalt mixtures with regard to rutting.

15 2. MATERIALS AND METHODS

16 2.1 Materials

17

18 2.1.1 Aggregates and granulometric composition

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20 The aggregates used in the production of the asphalt mixtures are of basaltic origin.
 21 The characterization of these materials was provided by Melo [16]. The author [16] also
 22 presents the characterization of the hydrated lime used in the study, which corresponds to
 23 type CH-1 dolomitic.

24 The formulation of the asphalt mixtures was comprised of 43% gravel, 15.5% crushed
 25 gravel, 40% of grit and 1.5% of lime [16]. This composition was established by the Leopoldo
 26 Américo Miguez de Mello Research and Development Center (CENPES / Petrobras), aimed
 27 at obtaining mixtures with a high resistance to permanent deformation.

28

29 2.1.2 Conventional asphalt binder

30

31 The conventional asphalt binder used in this study was a CAP 50/70, with PG 58-22.
 32 This binder was used in the production of the reference asphalt mixture and as a matrix for
 33 the modification of the binders 3% NC and 3% NC + 2% SBS. The conventional asphalt
 34 binder characterization is shown in Table 1.

35

36 **TABLE 1 Characterization of the conventional asphalt binder (CAP 50/70)**

Property	Unit	Standard	Result
Penetration	0.1 mm	ASTM D 5 [19]	57
Softening point	°C	ASTM D 36 [20]	47.9
Thermal susceptibility index	-	-	-1.44
Apparent viscosity			
at 135 °C (<i>spindle 21, 20 rpm</i>)	cP	ASTM D 4402 [21]	290
at 150 °C (<i>spindle 21, 50 rpm</i>)			150
at 175 °C (<i>spindle 21, 100 rpm</i>)			60

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38 2.1.3 Asphalt binder modified with SBS polymer

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40 The binder modified with SBS polymer was industrially produced and supplied by
 41 Greca Asfaltos S.A. The characterization of this material is provided in Table 2.

TABLE 2 Characterization of the binder modified with SBS polymer (SBS 60/85)

Property	Unit	Standard	Result
Penetration	0.1 mm	ABNT NBR 6576 [22]	50
Softening point	°C	ABNT NBR 6560 [23]	73.0
Elastic recovery	%	ABNT NBR 15086 [24]	90
Apparent viscosity			
at 135 °C (<i>spindle 21, 20 rpm</i>)	cP	ABNT NBR 15184 [25]	1910
at 150 °C (<i>spindle 21, 50 rpm</i>)			640
at 175 °C (<i>spindle 21, 100 rpm</i>)			290

2.1.4 Modifiers

The organophilic 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 , particle size after dispersion of 1 x 500 nm and density of 1.7 g/cm^3 . This nanomaterial is thermally stable at temperatures below 262.4 °C [16].

The SBS polymer 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 Study method

This study was carried out in four 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 with 3% nanoclay in relation to the weight of CAP 50/70) and 3% NC + 2% SBS (modified with 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, in which the resistance of asphalt mixtures, produced with modified binders with 1%, 2% and 3% of nanoclay, was evaluated to permanent deformation [16]. The addition of 2% SBS together with 3% NC was aimed at obtaining an asphalt material with elastic recovery. A low polymer content was adopted for economic reasons and based on results reported by Pamplona et al. [10], who studied the modification with 2.5% of nanoclay and 2.5% of SBS. 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). This modification was carried out according to procedures presented by Melo [16] and Carlesso [18]. The modified binders were then characterized according to the following properties: penetration (ABNT NBR 6576 [22]), softening point (ABNT NBR 6560 [23]), elastic recovery (ABNT NBR 15086 [24]), phase separation (ABTN NBR 15166 [26]) and apparent viscosity (ABNT NBR 15184 [25]).

Stage 2 consisted of establishing the binder contents in the design of the asphalt mixtures studied and in the molding of plates. For this, the Superpave design methodology was applied, according to the standards AASHTO M 323 [27] and AASHTO R 35 [28], with the use of a gyratory compactor. After that, for each mixture studied, a plate with dimensions of 60 x 40 x 9 cm (for sawing and obtaining prismatic specimens for the complex modulus tests) and two plates of 50 x 18 x 5 cm (for the permanent deformation tests) were molded. This molding was carried out at the LCPC compacting table, following the recommendations of the French standard AFNOR NF P 98-250-2 [29] for a heavy traffic highway. Plates with dimensions of 60 x 40 x 9 cm were then sawn and five specimens with dimensions close to 6.3 x 5.0 x 40.0 cm were obtained from each plate.

In Stage 3, the characterization of rheological behavior of different mixtures was carried out. This characterization was conducted, based on complex modulus tests, in a four-point bending test machine, following the recommendations of the European standard EN

1 12697-26 [30]. For each asphalt mixture, two of the five specimens produced in the previous
 2 stage were tested. Temperatures of 0 °C, 5 °C, 10 °C, 15 °C, 20 °C, 25 °C and 30 °C, with
 3 load frequencies of 0.1 Hz, 0.2 Hz, 0.5 Hz, 1 Hz, 2 Hz, 5 Hz, 10 Hz and 20 Hz, were
 4 evaluated, keeping the deformation amplitude at 50 $\mu\epsilon$. For the interpretation of the results,
 5 master curves and Black spaces were analyzed.

6 In Stage 4, the resistance to permanent deformation of the four different mixtures was
 7 carried out. In this stage, tests were performed in the French traffic simulator, following the
 8 standard AFNOR NF P 98-253-1 [31]. The results of these tests, after 30,000 load cycles,
 9 were compared to the limits established by French [32] and European [33] guidelines.

10 3. PRESENTATION AND DISCUSSION OF RESULTS

11 3.1 Characterization of modified binders

12
 13 The results for the characterization of the asphalt binders modified in the laboratory
 14 can be observed in Table 3.

15
 16 **TABLE 3 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	ABNT NBR 6576 [22]	55	36
Softening point	°C	ABNT NBR 6560 [23]	50.2	56.9
Elastic recovery	%	ABNT NBR 15086 [24]	6	49
Phase separation (24h / 48h)	°C	ABNT NBR 15166 [26]	1.0 / -	0.5 / 0.8
Apparent viscosity				
at 135 °C (<i>spindle</i> 21, 20 rpm)	cP	ABNT NBR 15184 [25]	410	760
at 150 °C (<i>spindle</i> 21, 50 rpm)			210	370
at 175 °C (<i>spindle</i> 21, 100 rpm)			90	160

17
 18 On comparing the results in Table 3 with those provided for the empirical
 19 characterization of CAP 50/70 (Table 1) and SBS 60/85 (Table 2), it can be observed that, as
 20 expected, the modification of the binders caused a decrease in the penetration and an increase
 21 in the softening point. Notable among these results are the relatively low penetration obtained
 22 for the binder 3% NC + 2% SBS and the relatively high softening point for the binder SBS
 23 60/85. This reflects gains in the stiffness in the first case in contrast with gains related to the
 24 sensitivity to high temperatures in the second case. These characteristics indicate that the
 25 asphalt mixtures formulated with these binders can be very resistant to permanent
 26 deformation. Thus, better performance is expected in relation to the permanent deformation
 27 for the mixtures SBS 60/85 and 3% NC + 2% SBS. However, it is important to highlight that
 28 the prediction of the behavior of asphalt mixtures based on the empirical characterization of
 29 modified binders shows high limitations.

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

36 3.2 Definition of binder contents and plate molding

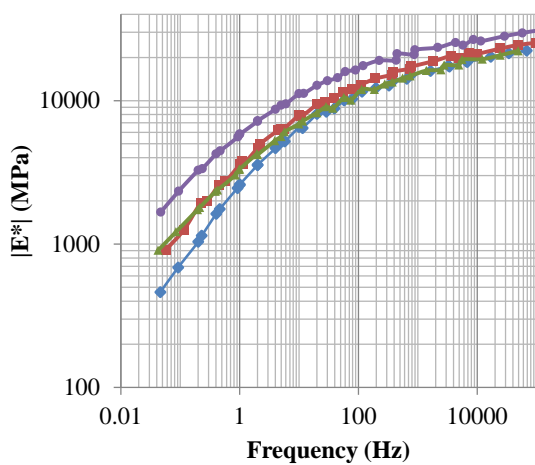
37
 38 In the dosage study of the asphalt mixtures, the following binder contents were
 39 obtained: 4.4% for the mixture CAP 50/70, 4.1% for 3% NC, 4.5% for SBS 60/85 and 4.3%

1 for 3% NC + 2% SBS. As can be observed, the addition of nanoclay to the conventional
 2 binder can lead to a reduction in the binder content of the asphalt mixture design. In contrast,
 3 with the inclusion of the polymer SBS, a higher binder content is required. Thus, the
 4 intermediate result obtained for the 3% NC + 2% SBS mixture in the dosage study
 5 demonstrates the combination of the positive effect of the nanoclay, in terms of mixture
 6 workability, with the negative effect of the presence of the polymer. However, the variation
 7 in the binder contents of the design can be kept within the interval allowed ($\pm 0.3\%$) in the
 8 field, based on Brazilian current construction specifications.

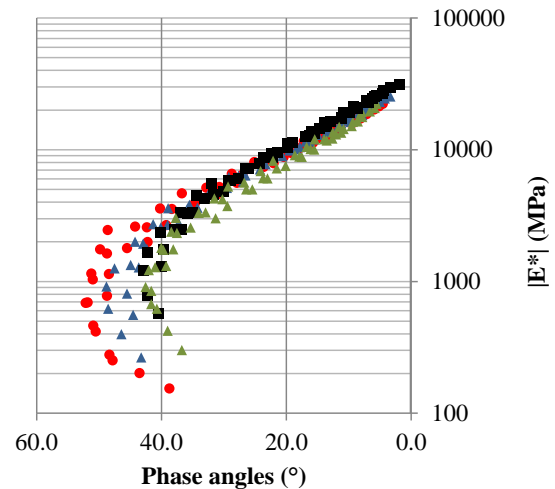
9 In the molding stage, specimens with void volumes between 3.52% and 4.54% and
 10 plates with void volumes between 4.50% and 6.68% were obtained. It should be noted that
 11 both plates molded to obtain the specimens used in the modulus tests and those molded for
 12 the permanent deformation tests were produced aiming at a void volume of 4%, as defined in
 13 the dosage study. However, during the compaction procedure, it was found to be difficult to
 14 control the final thickness of the plates, hindering, as a result, the precise obtainment of the
 15 void volume of the design. Nevertheless, considering that the specimens and the plates
 16 showed compaction degrees within the interval of 97% to 101% admitted in field, in Brazil,
 17 they were considered suitable for use in the tests.

19 3.3 Rheological characterization

21 Figure 1 shows the master curves and Figure 2 shows the Black spaces for different
 22 asphalt mixtures, built based on the results of complex modulus tests.



24
 25 **FIGURE 1 Master curves (reference**
 26 **temperature = 20 °C)**



27
 28 **FIGURE 2 Black spaces**

29 In general, the positioning of the master curves indicates higher stiffness for the
 30 modified asphalt mixtures. An exception, however, is the mixture SBS 60/85 which, at high
 31 load frequencies (equivalent to low temperatures), shows a rheological behavior similar to
 32 that of the conventional mixture. In comparison with the other mixtures studied, the mixture
 33 3% NC + 2% SBS showed notably high values for the complex modulus. This characteristic
 34 can be considered as an indication that the mixture will have greater resistance to permanent
 35 deformation. An analysis of the master curves also reveals the lower frequency susceptibility
 36 of the modified mixtures, based on the slopes of the curves. In the field, this would mean that
 37 the stiffness of these mixtures should be less sensitive to variations in the traffic speed,
 38 suggesting a high potential for their use along segments with a steep slope and slow traffic. In
 39

1 this regard, it is possible to establish a behavior hierarchy, where the mixture modified with
2 both modifiers is at the top, followed by SBS 60/85, 3% NC and the conventional mixture.

3 In Figure 2, it can be noted that the modified mixtures present an aspect of graphic
4 shortening (in relation to the phase angle) when compared with the conventional mixture.
5 This shortening illustrates the obtainment of smaller phase angles and reflects the more
6 elastic behavior of these mixtures. Of the modified mixtures, it can be observed that the 3%
7 NC + 2% SBS mixture has a higher concentration of low phase angles, followed by the SBS
8 60/85. The elastic behavior of these mixtures, both modified with SBS, reflects the influence
9 of the presence of an elastomeric polymer in the binders. Considered in isolation, this would
10 suggest a better performance of them in relation to their resistance to permanent deformation.

11 **3.4 Resistance to permanent deformation**

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13
14 In the permanent deformation tests, the following rutting depths (%) were obtained
15 after 30,000 load cycles: 9.5% for the mixture CAP 50/70, 6.7% for 3% NC, 6.2% for SBS
16 60/85 and 4.0% for 3% NC + 2% SBS.

17 According to the results, of the asphalt mixtures studied, the resistance to permanent
18 deformation was highest for the 3% NC + 2% SBS mixture, followed by the SBS 60/85, 3%
19 NC and conventional mixtures. In proportional terms, in practice, it is estimated that the
20 replacement of the conventional mixture with that containing the two modifiers would reduce
21 the rutting depth in the field by up to 58%. Considering the replacement of the reference
22 mixture with the SBS 60/85 and 3% NC mixtures, the corresponding reductions could reach
23 up to 35% and 29%, respectively. The best behavior in terms of permanent deformation
24 observed for mixture 3% NC + 2% SBS is in agreement with the performance prediction
25 carried out by way of the empirical characterization of the binders (lower penetration) and the
26 rheological study of the mixtures (higher stiffness and lower phase angles).

27 Regarding the maximum limits for the rutting depth established by the French [32]
28 and the European [33] specifications, all of the asphalt mixtures under study met the 10%
29 criterion. However, only the 3% NC + 2% SBS mixture satisfied the more restrictive criterion,
30 corresponding to 5%.

31 **4. CONCLUSIONS**

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

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

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

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