Optimization of Carbon Nanotube Content of Asphalt Nanocomposites with Regard to Resistance to Permanent Deformation

João V. Staub de Melo, Glicério Trichês, Liseane P. Thives

***Abstract*—**This paper presents the results of the development of asphalt nanocomposites containing carbon nanotubes (CNTs) with high resistance to permanent deformation, aiming to increase the performance of asphalt surfaces in relation to the rutting problem. Asphalt nanocomposites were prepared with the addition of different proportions of CNTs (1%, 2% and 3%) in relation to the weight of asphalt binder. The base binder used was a conventional binder (50-70 penetration) classified as PG 58-22. The optimum percentage of CNT addition in the asphalt binder (base) was determined through the evaluation of the rheological and empirical characteristics of the nanocomposites produced. In order to evaluate the contribution and the effects of the nanocomposite (optimized) in relation to the rutting, the conventional and nanomodified asphalt mixtures were tested in a French traffic simulator (Orniéreur). The results obtained demonstrate the efficient contribution of the asphalt nanocomposite containing CNTs to the resistance to permanent deformation of the asphalt mixture.

***Keywords*—**Asphalt nanocomposites, asphalt mixtures, carbon nanotubes, nanotechnology, permanent deformation.

I. Introduction

ONE of the structural defects most commonly encountered in asphalt pavements is permanent deformation. This can be defined as a depression in the wheel track with the possible occurrence of an elevation along the edges of this depression. Permanent deformation mainly originates from the instability of the asphalt concrete due to the excessive fluency of the mixture, aggravated by high temperatures, heavy traffic and the relief conditions [1]. In this context, this phenomenon is one of the main problems in developing countries with a tropical climate, where an increase in the volume and aggressiveness of the traffic has been recorded and also better quality asphalt pavements and coatings are required [2]. This defect leads to the formation of an uneven pavement surface, increasing the irregularity, the discomfort to road users and in some cases a loss of drivability. On rainy days the accumulation of water in the wheel tracks can cause accidents due to the phenomenon of aquaplaning, which occurs when vehicles lose the tire/pavement adherence required to maintain their stability.

The permanent deformation of an asphalt mixture can be considered as the simultaneous occurrence of viscous deformation of the asphalt binder and plastic deformation of the mineral structure of the asphalt mixture. From this perspective, both the aggregate and the asphalt binder play a role in the mechanical behavior of the asphalt mixture: the binder due to its consistency and rheology and the aggregate as a result of the internal friction forces between its particles [3].

According to Hunter [4], one way to ensure that the asphalt provides its contribution to the resistance to permanent deformation is the use of an asphalt binder which is not only more rigid but also which behaves as an elastic solid in the pavement at high temperatures. Thus, when the load is applied to the asphalt mixture the binder tends to behave more as a rubber, returning to its original position and not accumulating deformations. Based on this approach, the modification of asphalt binders can improve their performance and, consequently, that of the asphalt concrete mixtures. Several types of modifiers (elastomeric and plastomeric polymers, ground tire rubber, among others) have been employed as asphalt binders to improve the properties of asphalt concrete considering the main mechanisms of pavement degradation.

With regard to improving the properties of materials, a revolution has occurred in this field based on the use of nanomaterials as reinforcement in different matrixes. In this area, nanoscience offers great potential and new materials can be developed with properties superior to those of existing materials. Nanocomposites are a new class of materials, in which at least one dimension of the dispersed particles lies within the nanometric scale [5].

Several types of nanoloads are currently being developed, for instance, metal oxide nanoparticles, CNTs and nanoclays. In the past two decades several authors [6]-[11] have begun to study inorganic/organic nanocomposites, in particular nanocomposites of polymeric and asphalt matrices.

In this paper, the results of a study aimed at the development of asphalt nanocomposites containing CNTs with a high potential with regard to contributing to the resistance to permanent deformation of asphalt mixtures are reported.

II. Materials

In this study, the following materials were used: conventional asphalt binder (asphalt matrix) and CNTs (reinforcing load) for the preparation of asphalt

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nanocomposites, and aggregate minerals and hydrated lime for the production of the asphalt mixtures.

The base asphalt binder used in the study is classified according to the penetration as being within a range of 50-70 tenths of a millimeter and as PG 58-22 according to the Superpave classification.

The CNTs are comprised of multiple layers and have the characteristics shown in Table I, where the micrographic details of this nanomaterial can be observed.

The aggregate mineral selected for the formulation of the asphalt mixtures is of basaltic origin and its properties are shown in Table II.

The hydrated lime used in this study is of the type CH-I dolomitic. Table III shows the characteristics of this material.

TABLE I
CHARACTERISTICS OF CNTS

Properties	Results
External diameter	50-80 nm
Internal diameter	5-15 nm
Length	10-20 μm
Density	2.1 g/cm^3
Specific surface area	60-80 m^2/g
Carbon*	97.37%
Nickel*	1.86%
Iron*	0.55%
Chlorine*	0.20%
Sulfur*	0.02%

*Results obtained in the analysis by energy dispersive X-ray fluorescence (Shimadzu EDX-700).

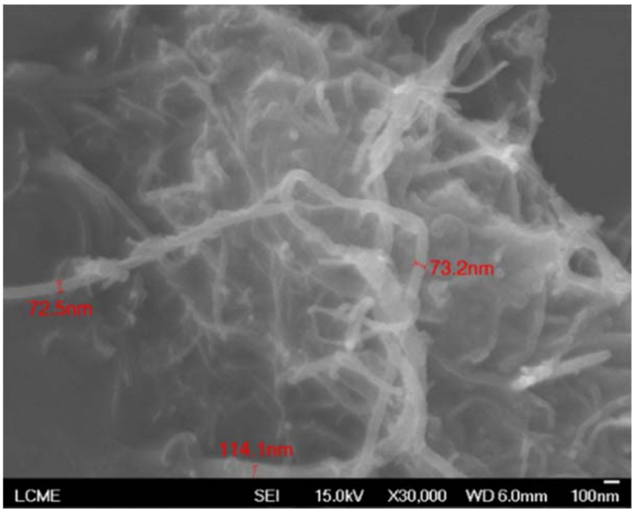

Fig. 1 Micrograph of a CNT: magnification 30,000 times

The generation of the granulometric curve used in the formulation of asphalt mixtures adhered to the Superpave specification for a nominal maximum size of 19 mm, satisfying the criteria of control points and restricted zone (recommended). Based on the granulometric study of the aggregates, the curve shown in Fig. 2 was established with a percentage passing through each sieve as shown in Table IV. The granulometric curve is comprised of 43% 3/4" gravel, 15.5% 3/8" gravel, 40% stone dust and 1.5% lime.

TABLE II
RESULTS OBTAINED FOR THE CHARACTERIZATION OF THE AGGREGATE

Properties	Results
True specific mass of the coarse aggregate [12]	2.953 g/cm^3
Apparent specific mass of the coarse aggregate [12]	2.880 g/cm^3
Absorption of coarse aggregate [12]	0.8%
True specific mass of fine aggregate [13]	2.974 g/cm^3
True specific mass of powdered material [14]	2.804 g/cm^3
Angularity of coarse aggregate [15]	100%
Angularity of fine aggregate [16]	49.2%
Flat elongated particles [17]	9.6%
Clay content (sand equivalent) [18]	61.2%
Hardness (Los Angeles abrasion) [19]	11.6%
Soundness [20]	2.1%
Deleterious material [21]	0%

*The coarse aggregate corresponds to the fraction which passes through a 3/4" sieve and is retained by a Nº 4 sieve; fine aggregate represents the fraction which passes through a Nº 4 sieve and is retained by a Nº 200 sieve; the powdered material passes through a Nº 200.

TABLE III
CHEMICAL AND PHYSICAL CHARACTERISTICS OF THE HYDRATED LIME

Properties	Results
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%
Density	3.0 g/cm^3

TABLE IV
GRANULOMETRIC COMPOSITION OF AGGREGATE MIXTURE

Sieves - ASTM Series	% Passing
3/4"	100.0
1/2"	77.5
3/8"	61.3
Nº 4	43.3
Nº 10	24.3
Nº 16	17.4
Nº 30	12.6
Nº 50	9.8
Nº 100	7.6
Nº 200	5.4

III. METHODS

In order to develop an optimized nanocomposite containing CNTs, with regard to its resistance to permanent deformation, asphalts with different contents of CNTs incorporated were produced. The definition of the optimum content of CNTs was based on an evaluation of the empirical and rheological properties of the nanocomposites produced in relation to the non-nanomodified (conventional) asphalt binder. After the optimization of the nanocomposite containing CNTs the performance in relation to the permanent deformation of the asphalt mixtures produced with the optimized nanocomposite and with the conventional asphalt binder was evaluated.

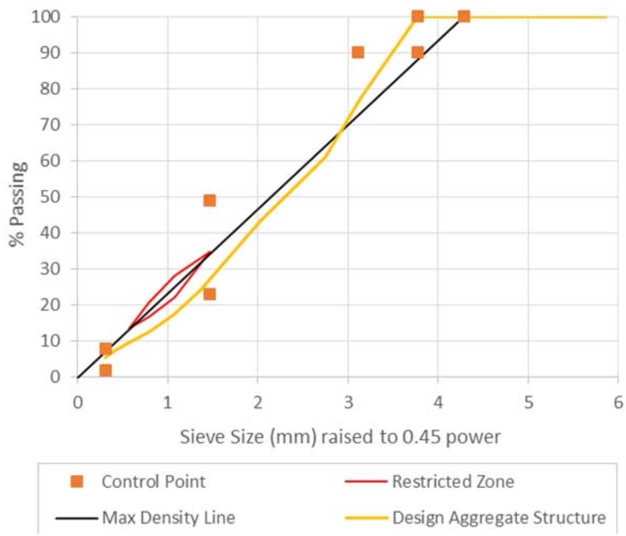

Fig. 2 Granulometric composition of aggregate mixture

A. Nanomodification of Conventional Asphalt Binder

The conventional asphalt binder was modified using a high shear mixer. The nanomodification was carried out with asphalt binder at a temperature of 150°C (rotational viscosity of 0.15 Pa.s), with a shear level of 5000 rpm and a mixing time of 100 min. Three asphalt nanocomposites were produced differentiated by the content of CNTs incorporated, according to Table V.

TABLE V
ASPHALT NANOCOMPOSITES PRODUCED

Names of Asphalt Nanocomposites	% Addition of CNTs
CNT-1%	1%
CNT-2%	2%
CNT-3%	3%

B. Optimization of Nanocomposite Containing CNTs

The definition of the optimum CNT content incorporated into the asphalt binder matrix was carried out through an evaluation of the properties of the nanocomposites produced. The traditional properties assessed were changes in the penetration [22], softening point [23] and thermal susceptibility index. The rheological evaluation of virgin samples and short-term aged residues was conducted using a dynamic shear rheometer (DSR) applying the rolling thin-film oven test (RTFOT) [24]. In these tests, the complex shear modulus (|G*|) and the phase angle (δ) [25] were determined at temperatures of 52°C, 58°C, 64°C, 70°C and 76°C. The optimization of the nanocomposites containing CNTs was carried out based on an evaluation of the gains in the properties obtained with the addition of CNTs in relation to the conventional asphalt binder. The aim was to develop asphalt nanocomposites which are less susceptible to temperature and more rigid and elastic.

C. Mix Design of Asphalt Mixture

In order to evaluate the performance of the optimized nanocomposites containing CNTs in terms of their resistance to permanent deformation, asphalt mixtures were formulated and compacted sheets were submitted to testing using the French traffic simulator (Orniéreur).

The mix design of the asphalt mixture was carried out according to the Superpave methodology and with the use of a gyratory compactor. The procedures adopted in the mix design were those recommended in the standards AASHTO M 323 [26] and AASHTO R 35 [27]. Three parameters were fixed for the modeling: compaction angle of 1.25°, compaction pressure of 0.6 MPa and gyration speed of 30 rpm. All specimens were molded into pieces with a diameter of 150 mm and a height of approximately 110 mm. The study on the mix design of the mixture was carried out for a high volume of traffic ($N_{initial}$ = 9 gyrations, N_{design} = 125 gyrations and $N_{maximum}$ = 205 gyrations). The asphalt binder content of the mix design was defined as that which fulfilled the following Superpave mix design criteria: pore percentage $N_{initial}$ > 11%, N_{design} = 4% and $N_{maximum}$ > 2%; voids in the mineral aggregate (VMA) ≥ 13%; voids filled with asphalt (VFA) between 65% and 75%; and dust proportion 0.8-1.6%.

D. Evaluation of Performance in Relation to Permanent Deformation of Asphalt Mixtures

After the mix design, two sheets (50 x 18 x 5 cm) of each asphalt mixture studied were molded on an IFSTTAR compaction table. The sheets were compacted according to the specifications of AFNOR NF P 98-250-2 [28]. The performance of the asphalt mixtures with regard to their resistance to permanent deformation was evaluated using the French equipment Orniéreur, according to the standard AFNOR NF P 98-253-1 [29]. In this test, the asphalt mixture sheets, at 60°C, were submitted to the passage of 30000 cycles of a rolling axle with a frequency of 1 Hz, loading of 5 kN and tire inflation pressure 0.6 MPa. During the test the depth of the foundation of the wheel track in relation to the sheet thickness was obtained for 100, 300, 1000, 3000, 10000 and 30000 cycles.

IV. RESULTS AND DISCUSSION

A. Optimization of Nanocomposite Containing CNTs

Figs. 3 and 4 show the effect of the CNTs in the asphalt binder with regard to the penetration (25°C, 100 g and 5 s) and the softening point, while Fig. 5 shows the thermal susceptibility index of the virgin samples.

Figs. 3 and 4 verify the reduction in the penetration and increase in the softening point with the addition of CNTs, resulting in greater sensitivity to variations in temperature, as observed from the thermal susceptibility index (TSI) (Fig. 5). A higher softening point and lower TSI indicate asphalts with lower susceptibility to permanent deformation. However, the consistency and thermal susceptibility of the asphalt binders alone does not guarantee good performance with regard to the permanent deformation of the asphalt mixture, and these need to be determined together with the rheological properties of the material.

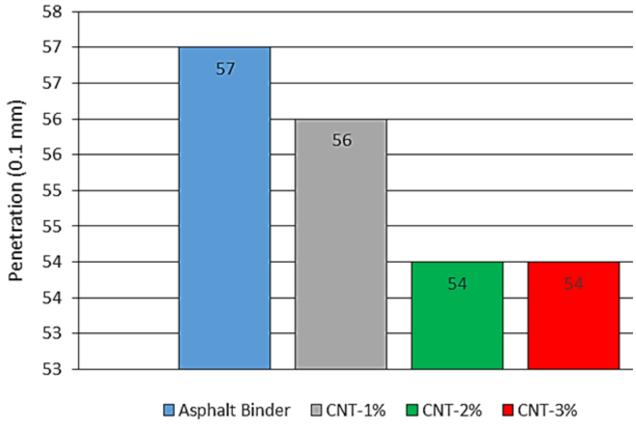

Fig. 3 Penetration of virgin samples

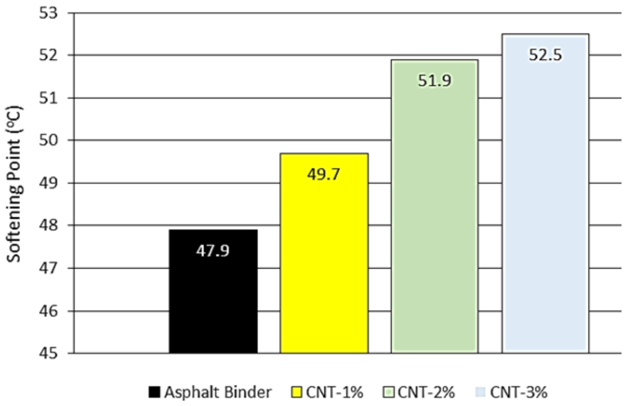

Fig. 4 Softening point of virgin samples

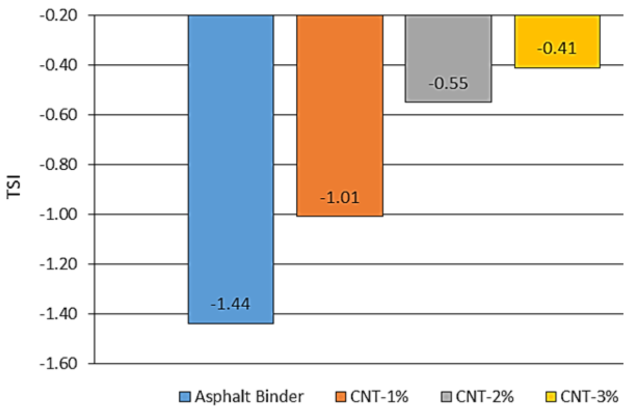

Fig. 5 Thermal susceptibility index (TSI) of virgin samples

The viscoelastic properties of the virgin and short-term aged asphalt binders (RTFOT), were studied using a dynamic shear rheometer (DSR). The rheometry was carried out at high temperatures (52^{o}C up to 76^{o}C), and the evaluation was directed toward the complex shear modulus (|G*|) and phase angle (δ), in order to obtain the parameter associated with permanent deformation (|G*|/sin δ).

The complex shear modulus (|G*|) and phase angle (δ) of asphalt binders indicate their contribution to the resistance to permanent deformation. At high temperatures, the rheological study is carried out based on the |G*|/sin δ parameter established according to the dissipated energy approach, where a rigid and elastic asphalt binder is sought. Thus, high values for the complex shear modulus (|G*|) are favorable, since these represent a high resistance to deformation, and low values for the phase angle (δ) are desirable, since this reflects a greater elastic component of the total deformation. Permanent deformation is considered a phenomenon under controlled stress and the greater the value for the parameter |G*|/sin δ the lower the amount of energy dissipated during each load cycle, according to (1) and (2) for the dissipated energy approach.

$$W_i = \pi \sigma_i \varepsilon_i \sin \delta_i \tag{1}$$

In the case of controlled stress, we have: $\sigma_i = \sigma_o$ and $\varepsilon_i = \frac{\sigma_o}{|G^*|}$. Thus:

$$W_i = \pi \sigma_i \varepsilon_i \sin \delta_i \rightarrow W_i = \pi \sigma_o^2 \left(\frac{1}{|G_i^*|/\sin \delta_i} \right) \tag{2}$$

where: W_i = energy dissipated in the load cycle i; σ_i = stress in the load cycle i; σ_o = initial stress; ε_i = strain in the load cycle i; δ_i = phase angle between the stress and strain signals in the load cycle i; and, $|G_i^*|$ = complex modulus in the load cycle i.

Figs. 6 and 7 show the results for the parameter |G*|/sin δ as a function of the variation in the temperature for the virgin samples and the residues aged in the RTFOT, respectively.

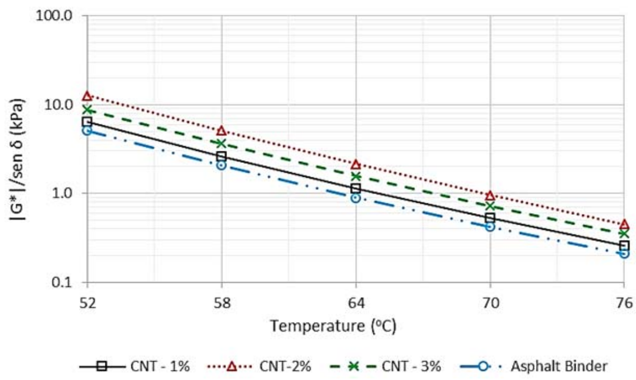

Fig. 6 Relation between |G*|/sin δ and temperature for the virgin samples

Figs. 6 and 7 clearly show the influence of the addition of CNTs on the rheological behavior of the asphalt binder. For all of the nanomodified samples and temperatures tested higher values were verified for the parameter |G*|/sin δ due to an increase in the complex shear modulus (|G*|) and a reduction in the phase angle (δ).

Table IV shows the gains in the performance obtained for each asphalt nanocomposite in relation to the conventional binder, for both the virgin samples and the residues obtained by RTFOT. The gains are evaluated in terms of temperature (^{o}C) and considered in relation to a |G*|/sin δ value of 1.0 kPa for the virgin samples and 2.2 kPa for the aged samples.

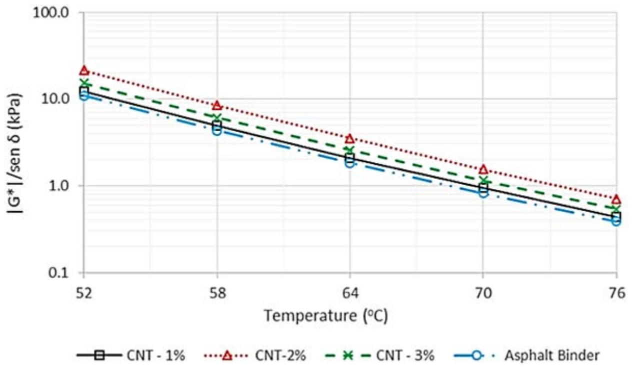

Fig. 7 Relation between |G*|/sin δ and temperature for aged residues (RTFOT)

TABLE VI
PERFORMANCE OF ASPHALT BINDERS IN RELATION TO THE PARAMETER |G*|/SIN δ

Asphalt Binders	Temperature (°C)		Gain in Performance (°C)	
	Virgin $\|G^*\|/\sin\delta = 1.0$ kPa	RTFOT $\|G^*\|/\sin\delta = 2.2$ kPa	Virgin	RTFOT
Conventional	63.0	62.7	-	-
CNT-1%	64.8	63.4	+ 1.8	+ 0.7
CNT-2%	69.6	67.3	+ 6.6	+ 4.6
CNT-3%	67.2	65.0	+ 4.2	+ 2.3

The data in Table VI verify that the incorporation of CNTs resulted in an improved performance of the asphalt binder in terms of permanent deformation. In this regard, the best performance is observed when the CNT content is increased from 1% to 2%. However, this gain is reduced with an increase in the content to 3%. The reduced performance could be related to the dispersion of the CNTs in the asphalt matrix, indicating that for contents of around 3% the dispersion is hindered, leading to the formation of agglomerates of CNTs in the matrix. In this regard, Biercuk et al. [30] and Liu and Wagner [31] reported that good nanometric dispersion not only allows a better interaction with the matrix but also ensures that the agglomerates do not concentrate stresses, which affect the mechanical performance of the nanocomposites. In this context, several authors [32]-[34] have noted that a CNT concentration higher than the critical concentration leads to a reduction in the mechanical characteristics of the nanocomposites, which in some cases can be lower than those of the pure matrix.

The rheological results show an optimum incorporation of around 2%. The asphalt binder with 2% addition of CNTs shows a maximum performance gain in the resistance to permanent deformation of 6.6°C for the virgin samples and 4.6°C for the aged samples when compared with the conventional asphalt.

B. Mix Design of Asphalt Mixtures

Prior to the verification of the susceptibility to permanent deformation of the asphalt mixtures, the Superpave mix design of the asphalt mixture and the molding of the asphalt sheets were carried out. In this regard the mix design of the mixture was performed using the conventional asphalt binder. Fig. 8 shows the graphs obtained from the gyratory compaction of the asphalt mixture. The contents defined in the mix design study were obtained by varying the estimated binder content (4.0%) by ± 0.5% and +1.0%.

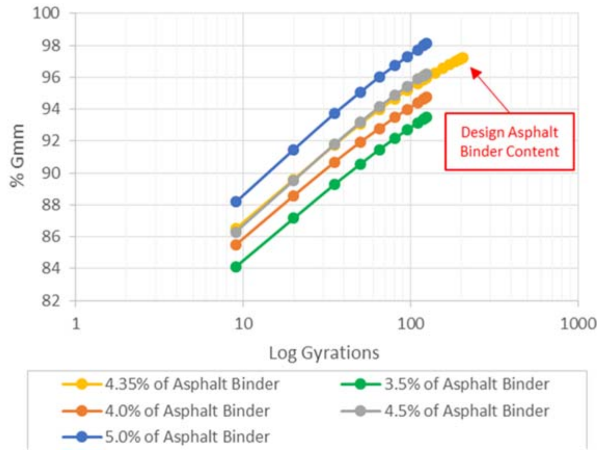

Fig. 8 Compaction curves for the asphalt mixture

Based on the compaction curves (Fig. 8), taking as the suitable binder content that which led to 4% of pores at 125 gyrations (N_{design}), the design content of the mixture was defined as 4.35% of asphalt binder. The volumetric criteria obtained in the mix design are shown in Tables VII and VIII.

TABLE VII
VOLUMETRIC CRITERIA FOR THE DEFINITION OF THE DESIGN CONTENT

% Binder	G_{mb} @ N_{design} (g/cm^3)	% G_{mm} @		
		$N_{initial} < 89\%$	$N_{design} = 96\%$	$N_{maximum} < 98\%$
3.50	2.548	84.1	93.5	-
4.00	2.560	85.4	94.7	-
4.35	2.577	86.5	95.9	97.2
4.50	2.577	86.3	96.2	-
5.00	2.605	88.1	98.1	-

TABLE VIII
VOLUMETRIC PROPERTIES OF MIXTURES WITH DIFFERENT ASPHALT BINDER CONTENTS

% Binder	G_{mm} (g/cm^3)	Vv (%)	VMA (%)	VFA (%)	Dust Proportion
Criterion	-	4.00	13.00 min.	65-75	0.8-1.6
3.50	2.725	6.50	14.35	54.66	1.74
4.00	2.701	5.22	14.38	63.68	1.50
4.35	2.685	4.01	14.12	71.57	1.37
4.50	2.678	3.78	14.27	73.52	1.32
5.00	2.655	1.89	13.79	86.29	1.17

It can be observed in Tables VII and VIII that for the design content obtained (4.35%) the dosed asphalt mixture satisfies all of the volumetric requisites of the specifications.

As mentioned above, a design content of 4.35% was arrived at for the conventional asphalt binder and in a complementary study (data not shown) a design content of 4.10% was obtained for the asphalt nanocomposite with 2% CNTs. However, the adoption of different contents of asphalt binders in the mixtures in order to investigate the changes in the mechanical characteristics due to the use of different types of binders introduces a variable which affects the results. In this regard, a variation in the proportion of materials is inherent to the use of different contents of asphalt binder. Therefore, in order to guarantee the same proportion of the materials (content of binder and granulometric distribution) in the asphalt mixtures studied, the design binder content of 4.35% was fixed for the conventional and nanomodified asphalt mixtures in the evaluation of permanent deformation.

C. Evaluation of Permanent Deformation Performance of Asphalt Mixtures

After the volumetric parameters of the Superpave mix design had been attained, two asphalt sheets were molded in the LCPC compactor for each mixture. After a period of 15 days of compaction the sheets were submitted to permanent deformation tests. Fig, 9 shows the results for the resistance to permanent deformation of the asphalt sheets tested in the laboratory traffic simulator.

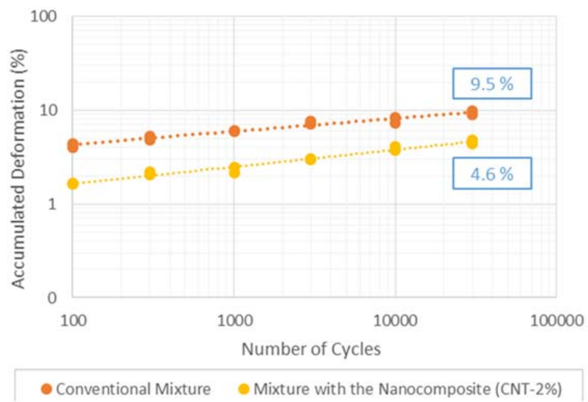

Fig. 9 Performance of permanent deformation of asphalt mixtures produced

In general, as shown in Fig. 9, the nanomodified asphalt mixture obtained better performance than the conventional mixture. After 30,000 cycles the accumulated deformation of the conventional mixture was 9.5% in relation to the thickness of the sheet while the mixture with the nanocomposite containing 2% of CNTs showed an accumulated deformation of 4.6%. Thus, the reduction in the permanent deformation at 30,000 cycles is 51.6% with the addition of 2% of CNTs. The results verify the beneficial effect of the addition of nanometric loads on the mechanical performance of the asphalt mixture, reducing the permanent deformation.

The permanent deformation of an asphalt coating layer is associated with several factors, mainly the formulation of the granulometric composition and the appropriate mix design of the mixture (binder content). However, the properties of the asphalt binder will also influence the behavior of the mixture. In this regard, the different responses of the behavior verified in Fig. 9 are associated with the characteristics of the asphalt binder, since the binder content and the granulometric composition of the two mixtures did not change.

The improved behavior of the permanent deformation of the nanomodified asphalt mixtures is directly associated with the CNTs, which form an elastic network, and to the high modulus inside the asphalt matrix. Thus, with each passing of traffic the total deformation and also the viscous deformation of the asphalt binder are smaller.

V. Conclusion

This paper presents the results of research carried out to develop asphalt nanocomposites containing CNTs with a high potential for the resistance of permanent deformation. The main conclusion of this study is that the nanomaterials had a beneficial effect on the rheological behavior of asphalt binders and on the mechanical performance of the asphalt mixtures with regard to permanent deformation. The obtainment of asphalt mixtures with a high resistance to wheel track rutting based on the incorporation of CNTs was verified.

The incorporation of nanomaterials into the conventional asphalt binder led to an improvement in the empirical and rheological properties of these high temperature materials. With the addition of CNTs the softening point of the asphalt binder increased and the penetration decreased, leading to better thermal susceptibility, which is dependent on the content incorporated. The reduction in the sensitivity to temperature is greater with an increase in the CNT content incorporated. The complex shear modulus increased and the phase angle decreased at high temperatures with the addition of CNTs. The results show that the optimum content added is around 2%. However, it should be noted that the addition of around 3% of CNTs leads to a reduction in the rheological characteristics of the nanocomposites, possibly due to the formation of larger agglomerates of CNTs and a lack of interaction with the asphalt matrix.

For the mixture studied, the incorporation of 2% of CNTs in the asphalt binder reduced the permanent deformation of the mixture by 51%, indicating that in the field, particularly in tropical developing countries, a significant reduction in the appearance of this defect on highways could be achieved.

It is recommended that in future investigations an evaluation of the effect of the addition of CNTs to nanocomposites on the rheological behavior and fatigue resistance of the asphalt mixtures is carried out. In this regard, it is important to know the rupture characteristics of the materials involved in the construction of pavements, bearing in mind that the behavior of asphalt coatings under bending demands is characterized by specific laws, such as the law of fatigue, which should be addressed in the structural design of the pavement.

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