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Zinc Oxide as a Filler in a Hot-Mix Asphalt: Impact on Mechanical Properties

Hugo Alexander Rondón-Quintana ^{1,*} , Karem Tatiana Forero-Rubiano ¹, Yohan Sebastián Valderrama-Agudelo ¹, Juan Gabriel Bastidas-Martínez ² and Carlos Alfonso Zafra-Mejía ³

¹ Facultad del Medio Ambiente y Recursos Naturales, Universidad Distrital Francisco José de Caldas, Bogotá 110321, Colombia; ktforeror@udistrital.edu.co (K.T.F.-R.); ysvalderramaa@udistrital.edu.co (Y.S.V.-A.)

² Facultad de Estudios a Distancia, Universidad Militar Nueva Granada, Cajicá 250240, Colombia; juan.bastidas@unimilitar.edu.co

³ Grupo de Investigación en Ingeniería Ambiental-GIIAUD, Facultad del Medio Ambiente y Recursos Naturales, Universidad Distrital Francisco José de Caldas, Bogotá 110321, Colombia; czafra@udistrital.edu.co

* Correspondence: harondonq@udistrital.edu.co

Abstract: Zinc oxide (ZnO) exhibits promising thermochemical properties when used as an asphalt binder modifier. Its micrometric size further enhances its potential as a substitute for natural fillers (NFs) in hot-mix asphalt (HMA). This study evaluates the effect of partially and fully replacing NFs with ZnO on the mechanical performance of HMA, addressing a research gap since the influence of ZnO as a filler in asphalt mixtures has not been previously investigated. NFs were replaced by ZnO at weight-based proportions of ZnO/NF = 25, 50, 75, and 100%. Initially, the morphology of NF and ZnO particles was analyzed using Scanning Electron Microscopy (SEM). Asphalt mastics were then produced with the same ZnO/NF proportions and subjected to conventional characterization tests, including penetration, softening point, and viscosity. In the next phase, HMA samples were designed using the Marshall method, incorporating ZnO at 0, 25, 50, and 100% replacement levels (designated as Control, HMA-25, HMA-50, and HMA-100, respectively). The mechanical performance of these mixtures was assessed through indirect tensile strength (ITS) and Cantabro tests. Based on the initial results, further evaluations were conducted on the Control, HMA-50, and HMA-100 mixtures to determine their resilient modulus, fatigue behavior under stress-controlled conditions, and resistance to permanent deformation (static creep test). The findings indicate that ZnO can replace NF in HMA without compromising Marshall stability or Cantabro strength. Additionally, ZnO-modified HMAs exhibit increases in stiffness under cyclic loading, and improvements in resistance to permanent deformation, fatigue performance, and moisture damage. These enhancements occur despite a 0.5% reduction in binder content compared to the Control HMA and a slight increase in porosity.

Keywords: filler; zinc oxide; ZnO; hot-mix asphalt; asphalt mastic; mechanical performance



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1. Introduction

In a hot-mix asphalt (HMA), the filler (mineral aggregate smaller than 0.075 mm) makes up approximately 3–10% of the aggregate mass, but volumetrically it makes up 70–90% [1–4]. The combination of viscoelastic asphalt binder and solid filler forms a material called asphalt mastic [5,6], which is credited with increasing HMA stiffness, cohesion, and adhesion properties, and much of its mechanical performance and durability [3,5,7–13]. The type of filler, its geometry, size, surface area, porosity, texture, specific gravity, physico-chemical and mineralogical properties, etc., influence the workability, compactability, and

short- and long-term mechanical response of asphalt mastic and HMA [10,11,14–22]. The most used fillers are conventional natural powders, which are mainly obtained from the crushing process of natural mineral aggregates (e.g., granite, limestone, marble). Obtaining natural filler (NF) is costly [23] and impacts the environment (e.g., alteration of the ecosystem, loss of vegetation and water retention layers, lowering of the water table, etc.). For such reason, the use of alternatives other than traditional NFs, such as lime [17,18,24]; Portland cement [25,26]; and wastes from construction, industry, agriculture, mining, among others [3,11,20,22,27–30].

One promising material that could be used as a filler in HMA is zinc oxide (ZnO). ZnO is the main chemical product obtained from metallic zinc, which is the fourth most widely used metal in the world [31]. It is a colorless material with low toxicity to humans [32–34]. ZnO can aid in the photodegradation of pollutants, mineralization of toxic organic compounds, and removal of acidic pollutants from rainwater [35–37]. It is a wide-gap semiconductor with high thermo-chemical stability and piezoelectric properties [38–40], high resistance to deformation, low electrical resistivity, and photocatalytic behavior. It improves the ductility of certain materials when incorporated as nanoparticles in polymeric matrices [41] and exhibits a low coefficient of expansion and high resistance to ultraviolet (UV) radiation [42]. These properties, together with its nanometer size and high compatibility with asphalts, have promoted its study as an asphalt binder modifier [43–49]. Most of the studies on the subject conclude that ZnO helps to improve the UV aging resistance of asphalt binders (e.g., [40,45,46,48–60]). ZnO may even help to improve the resistance to thermo-oxidative aging of these binders [41,61,62]. ZnO increases stiffness and rutting resistance and tends to improve rheological properties at high temperatures [41,48,56,62,63]. It also exhibits resistance to moisture damage [40,53,64–68] and fatigue cracking [40,56–59,64,69,70].

To date, in asphalt pavements, ZnO has only been used as a binder modifier. Additionally, the influence of these modified binders on the properties of HMAs has been little studied [48]. The influence of ZnO as a filler replacement in HMAs has not been investigated. Therefore, as an innovative aspect and to contribute to the state of knowledge on the subject, the main objective of this study was to evaluate the influence of this material when used as a filler in a HMA. To achieve this objective, HMAs were initially designed by the Marshall method, replacing the NF fraction with ZnO in proportions of ZnO/NF = 25, 50, and 100% by weight. Indirect tensile strength (ITS) and Cantabro tests were also performed on these mixtures. Based on the results obtained, in a second experimental phase, cyclic loading tests (resilient modulus—RM, and fatigue strength) and static creep tests were carried out to evaluate the resistance to permanent deformation of HMAs made with ZnO/NF = 50 and 100%. Additionally, Scanning Electron Microscope (SEM) visualizations of ZnO and conventional characterization tests (ASTM D5 penetration, ASTM D36 softening point, and ASTM D4402 viscosity) were performed on five asphalt mastics (ZnO/NF = 0, 25, 75, 50, and 100%). To evaluate whether ZnO as a filler influences the mechanical properties of HMA, an ANOVA analysis of variance with 95% confidence was performed. In this analysis, when F_T (quotient of the variances of the two analysis groups) is less than the critical value ($F_T < F_{0.05}$), it means that the changes in the evaluated parameters are not statistically significant; otherwise ($F_T > F_{0.05}$), the variations are considered significant.

2. Materials and Methods

2.1. Materials

The gradation and aggregate properties of the Control HMA (consisting of 100% natural filler; ZnO/NF = 0%) are shown in Figure 1 and Table 1, respectively. An AC 60/70 (penetration range in dmm) was used as a binder (Table 2). The performance grade (PG)

of the AC is 64–22. Both the aggregate and the AC meet the requirements established by IDU [71] and INVIAS [72] to produce HMAs.

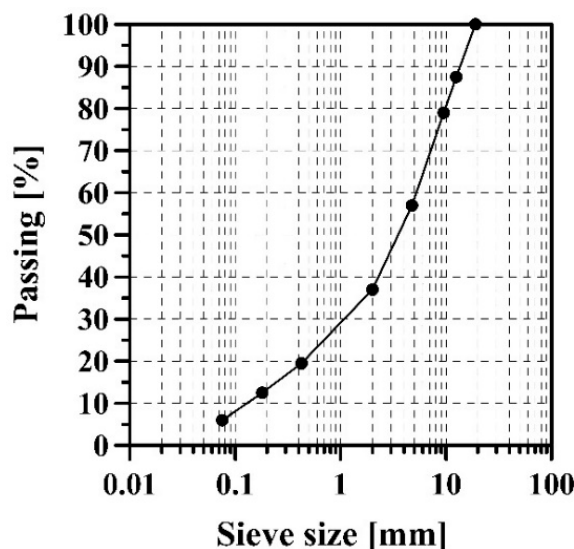


Figure 1. Aggregate gradation of HMA.

Table 1. Properties of mineral aggregates.

Test (Standard), Unit	Requirement	Value
Abrasion in Los Angeles machine (AASHTO T96), in %	25% maximum	22.7
Micro-Deval (AASHTO T327), %	20% maximum	18.6
10% of fines—dry (DNER-ME 096), kN	110 kN minimum	122.5
10% of fines—wet (DNER-ME 096), kN	82.5 kN minimum	108.9
Soundness of aggregate (AASHTO T104), in %	18.0% maximum	5.30
Specific gravity (fine aggregate; AASHTO T84), in -	-	2.652
Absorption (fine aggregate; AASHTO T84), in %	-	1.65
Specific gravity (coarse aggregate; AASHTO T85), in -	-	2.671
Absorption (coarse aggregate; AASHTO T85), in %	-	1.88
Fractured particles (ASTM D5821), in %	85% minimum	92.7

Table 2. Properties of binder AC 60/70.

Test (Standard), Unit	Requirement	Value
Virgin asphalt		
Penetration (ASTM D5), in dmm	60–70	61.6
Softening point (ASTM D36), in °C	48–54	48.7
Ductility (ASTM D113), in cm	100 minimum	128
Viscosity at 135 °C (ASTM D 4402), in Poises	4 minimum	4.72
Penetration Index (NLT 181), in -	−1.2 at +0.6	−1.05
Specific gravity (AASHTO T 228), in -	-	1.024
Flash and fire points (ASTM D92), in °C	230 minimum	288
After Rolling Thin Film Oven Test		
Mass loss (ASTM D2872), in %	0.8 maximum	0.22
Penetration (ASTM D5), in %	50 minimum	82.8
Increase in softening point (ASTM D36), in °C	9 maximum	2.3

The aggregate of natural origin is limestone. X-ray diffractometry (XRD) and X-ray fluorescence (XRF) tests were performed on the NF obtained from this aggregate (Figure 2a) [27]. The chemical composition of the NF showed a predominance of SiO_2 (79%), which is consistent with its mineralogical composition (61.0% Quartz, 25.1% Albite, 5.3% Illite, and 8.7% Chlorite). The filler also showed low CaO content (1.09%), and proportions of Al_2O_3 , Fe_2O_3 , MgO, and Na_2O of 7.9, 3.7, 2.3, and 1.8%, respectively, among other elements. ZnO is commercially available and sold over the counter. It was provided by “Químicos Campota & Cía. Ltda.” (Bogotá D.C., Colombia). On the NF and the white-colored ZnO (Figure 2b), visualizations were performed in a SEM, performing magnifications between 30 and 40,000 $\times$ in a JEOL JSM-6700F (Japan electron optics laboratory Co., Ltd., Tokyo, Japan) with an accelerating voltage of 4 to 20 kV and working distance between 9 and 15 mm (Figure 3). The elemental chemical composition of the particles, their size, and pore diameter were measured. The latter varied between 6 and 36 μm for NF and between 300 and 500 nm for ZnO. ZnO particles are smaller in size (less than 20 μm) compared to NF (particles varying between 30 and 70 μm). Each ZnO particle is composed of agglomerated nanoparticles of tubular shape (Figure 3c) with dimensions between approximately 200 and 600 nm. The use of tubular nanoparticles as fillers in asphalt mixtures has shown considerable potential for enhancing various properties of asphalt mastic, such as viscosity, stiffness, and thermal stability. However, when these nanoparticles form agglomerates, their behavior can change significantly, affecting both the rheological properties and the long-term performance of asphalt mixtures [73]. This is the main reason why some researchers recommend dispersing these nanoparticles using a dispersing agent or mechanism or doping the ZnO surface [41,48,64,74,75]. In the present study, ZnO was used as supplied by the provider (without any prior treatment or modification). The elemental chemical composition of NF in the SEM is like the one reported in the XRF test. For the case of ZnO, each particle showed contents of 80% Zn and 20% O, which agrees with the information provided by the supplier (80.28% Zn, and low bacterial and trace organic content). ZnO is an alkaline material ($\text{PH} > 7$), and its specific gravity (SG) is 5.6.

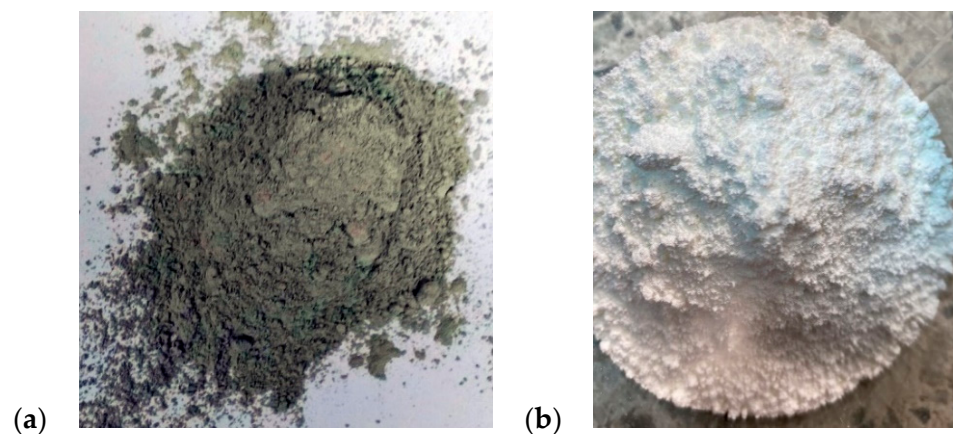


Figure 2. (a) NF; (b) ZnO.

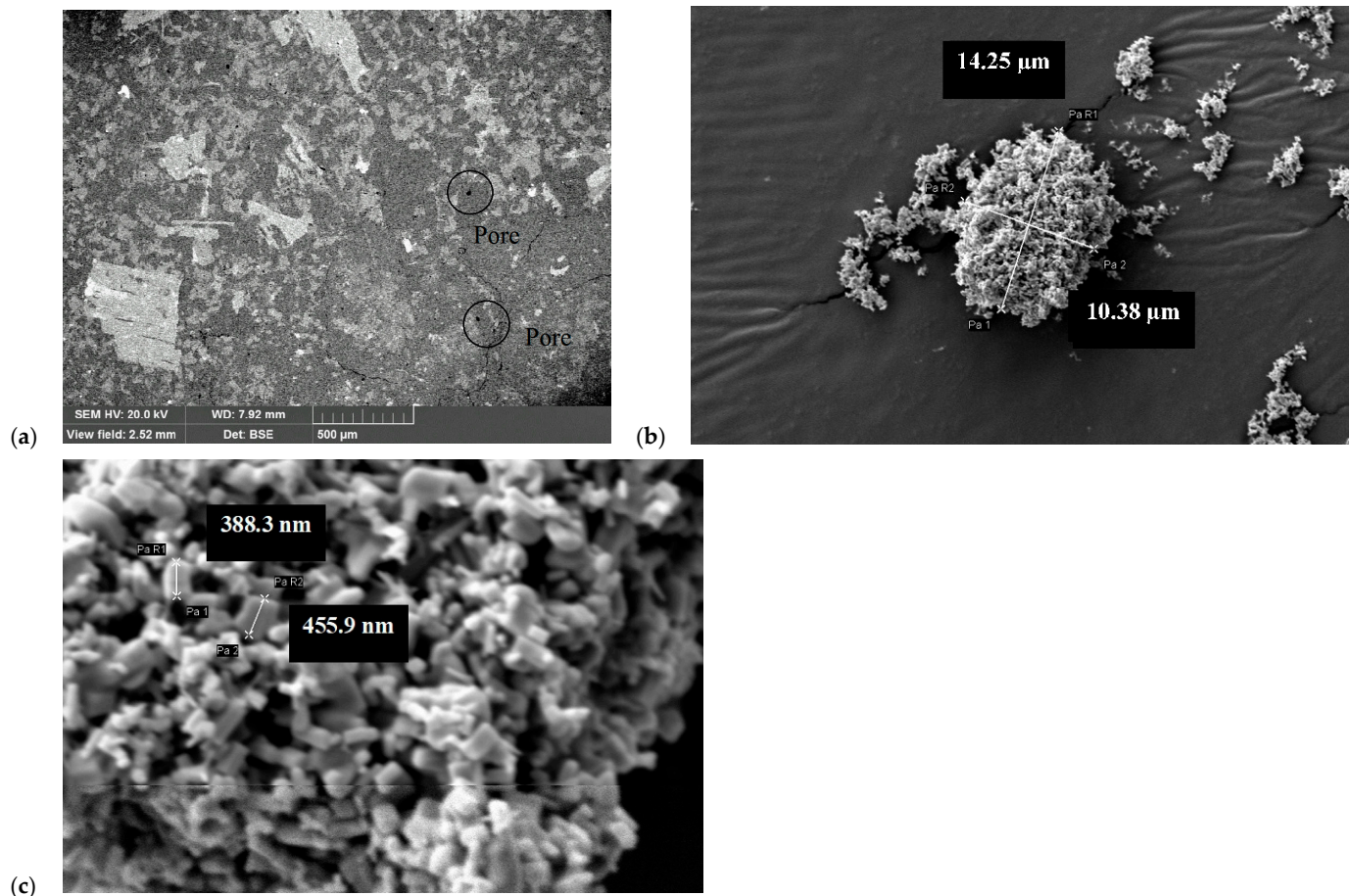


Figure 3. (a) NF to 30 increases; (b) ZnO to 6000 increases; (c) ZnO to 40,000 increases.

2.2. Asphalt Mix Design

Four types of HMA specimens were produced and designed following the guidelines established by AASHTO T 245: Control (HMA with 100% NF; ZnO/NFs = 0%), HMA-25 (HMA with 25% NFs replaced by ZnO; ZnO/NFs = 25% by weight); HMA-50 (HMA with 50% NFs replaced by ZnO; ZnO/NFs = 50% by weight), and HMA-100 (HMA with total or 100% NFs replaced by ZnO; ZnO/NFs = 100% by weight). For the design, three samples (compacted at 75 blows per face) were produced per mix type and per binder content, ranging from 4.5 to 6.0%. The mixing and compaction temperatures of the samples were 150 ± 3 and 145 ± 3 °C, respectively, and were chosen based on the viscosity criterion (170 ± 20 cP and 280 ± 30 cP, respectively). The maximum load that the specimens withstood under monotonic loading at a rate of 50.8 mm per minute (stability— S), as well as the displacement from load initiation to material failure (flow— F), were measured in a Marshall press. To measure S and F , the samples were previously conditioned in a water bath at 60 °C for one hour. With these parameters, the Marshall Quotient (S/F) was calculated. The volumetric composition (air voids in volume— V_a , voids in mineral aggregate— VMA , and voids filled with asphalt— VFA) and Bulk specific gravity of the samples were calculated following the guidelines of ASTM D2726. The design criteria to determine the optimum asphalt binder content (OAC) were those established in [71,72].

2.3. Asphalt Mastics

To contribute to the analysis of the results, five types of asphalt mastic were produced: ZnO/NFs = 0, 25, 50, 75, and 100%. The filler-to-binder ratio (by weight) was 1.09 for ZnO/NF = 0% and 1.20 for ZnO/NF = 25, 50, 75, and 100%. These ratios were chosen

based on the results of the previous experimental phase (the 6% filler content according to Figure 1 was divided by the OAC obtained from the HMA design). The fillers (NF and ZnO) were dried in an oven for 24 h at 105 °C and then mixed at 150 °C and 200 rpm for five minutes with AC 60/70 [3] (Figure 4). To avoid problems of sedimentation of the filler, small samples of approximately 50 g were made. Conventional characterization tests such as penetration (ASTM D5), softening point (ASTM D36), and rotational viscosity (ASTM D4402; measured at 135, 145, 150, 165, and 180 °C) were performed on these samples.



Figure 4. Mixture of AC, ZnO, and NF.

2.4. Mechanical Resistance Tests

On samples produced with the OAC obtained from the design of each HMA (Control, HMA-25, HMA-50, and HMA-100), ITS tests (AASHTO T283) and Cantabro tests (Tex-245-F) were initially performed. Based on the results obtained from this initial phase, RM (UNE-EN 12697-26), static creep/flow time (NCHRP, 2001; Appendix C) and fatigue strength (UNE-EN 12697-24) tests were then performed on the Control, HMA-50, and HMA-100 mixtures.

The ITS test was carried out on six Marshall-type specimens per type of mix produced with the V_a obtained from the design of each mix (i.e., the criterion recommended by AASHTO T283 of $V_a = 7 \pm 0.5\%$ was not considered). Three were tested in the dry state (*ITSD*) and three in the wet state (*ITSC*). Monotonic loading in indirect tension was applied at a displacement rate of 50 mm per minute in a Marshall press. The wet or partially saturated samples were previously conditioned or immersed in water at 60 °C for one day. The resistance to moisture damage was evaluated by calculating the *ITSC/ITSD* ratio in percent (*TSR*).

Abrasion and raveling resistance were evaluated by measuring the *CL* (mass or Cantabro Loss; Equation (1) parameter of the Cantabro test on a Los Angeles machine (without steel spheres). Three Marshall-type dry samples for each type of HMA were tested at 20 °C.

$$CL = \frac{mi - mf}{mi} \cdot 100 \quad (1)$$

where *mi* and *mf* are the initial and final mass of the specimen after 500 revolutions in the Los Angeles machine.

RM is a parameter used to evaluate the stiffness under cyclic loading of HMAs when a recess period is applied between loads. The test was performed at laboratory temperature (20 °C), applying a half-sine cyclic load of 1200 N with frequencies of 2.5 Hz (load application time—*t_c* = 125 ms and recess period—*t_r* = 275 ms), 5.0 Hz (*t_c* = 63 ms

and $t_r = 137$ ms), and 10.0 Hz ($t_c = 31$ ms and $t_r = 69$ ms). The test was carried out on a Universal Testing Machine—UTM 30. The RM in MPa was obtained using Equation (2).

$$RM = \frac{P \cdot \mu}{t \cdot \Delta H} \quad (2)$$

where P is the cyclic load in N, μ is Poisson's ratio (0.35 for asphalt mixes), t is the sample thickness in mm, and ΔH is the recoverable horizontal deformation in mm measured with a LVDT (Linear Variable Differential Transformer).

The static creep test is used as a performance criterion indicator of the resistance to permanent deformation of HMAs. The general guidelines recommended by NCHRP [76] (Appendix C) were followed for the test, but some changes were made. For example, the specimens were of Marshall type, and the test temperature was 20 °C (laboratory temperature). The axial stress was 100 kPa and was applied for 7200 s. During load application, axial and radial displacements were measured using LVDTs on the UTM 30. The average of the evolution of axial displacements with the loading time of three specimens per mix type was plotted to evaluate the resistance to permanent deformation.

Fatigue strength can be performed under stress or strain control. In this study, an indirect tensile stress-controlled mode was used. The loading frequency was 2 Hz (loading time 125 ms and unloading time 375 ms). Three stress magnitudes (σ) were used in the UTM: 150, 250, and 350 kPa. Nine Marshall-type specimens were tested per HMA type (three specimens for each stress). The failure criterion was the complete rupture of the specimens. With the results obtained, the number of failure cycles (N_f) that the HMAs resisted under the three applied stresses was obtained.

In this experimental phase, a total of 81 specimens were prepared, as shown in Table 3. Including the specimens fabricated for the mix design using the Marshall method (48 specimens corresponding to 4 asphalt mixtures, 4 binder contents, and 3 specimens per binder content), a total of 129 specimens were prepared.

Table 3. Number of samples for each test and type of mixture.

Test	Parameter	Asphalt Mix Type			
		Control	HMA-25	HMA-50	HMA-100
ITS	ITSD (kPa)	3	3	3	3
	ITSC (kPa)	3	3	3	3
Cantabro	CL (%)	3	3	3	3
RM	RM (MPa)	3	-	3	3
Static creep	Displacement (mm)	3	-	3	3
Fatigue	N_f (-) to $\sigma = 150$ kPa	3	-	3	3
Fatigue	N_f (-) to $\sigma = 250$ kPa	3	-	3	3
Fatigue	N_f (-) to $\sigma = 350$ kPa	3	-	3	3

3. Results

3.1. Marshall Test

The results of the Marshall test are shown in Figure 5. The Control HMA exhibits its maximum Marshall strength (higher S and S/F values; Figures 5a and 5c, respectively) at 5.5% AC. For the case of the mixtures with the replacement of NF by ZnO, this maximum strength is achieved when the AC content is 5.0%. At these AC percentages, S shows statistically non-significant increases ($F_T < F_{0.05} = 7.71$; Table 4) of 1.4, 6.1, and 3.2% with respect to the Control mix when 25, 50, and 100% of NF is replaced by ZnO, respectively. For the case of the S/F ratio, the HMA-50 mixture shows a similar magnitude compared to the Control mixture, but statistically insignificant decreases ($F_T < F_{0.05} = 7.71$; Table 4) of 7.9%

and 4.1% were observed when 25% and 100% of the NFs were replaced by ZnO, respectively. In summary, by replacing NFs with ZnO, the mixtures exhibit similar resistance under monotonic loading with respect to the Control mixtures, with an AC content lower by 0.5%. This is even though the mixtures with replacement of NF by ZnO present higher porosity (V_a is higher and statistically significant, $F_T > F_{0.05} = 7.71$; Table 4). The higher porosity of the HMA with ZnO is mainly due to the difference in specific gravity of the fillers ($SG = 5.60$ and 2.65 for ZnO and NF, respectively). When replacing NF with ZnO by mass, the number of particles to be covered with AC is lower (lower specific surface area), generating larger intergranular spaces not covered with AC (Figure 5d–f) [11,77,78]. Additionally, the stiffness and viscosity of asphalt mastic increase (as will be shown in the next section), making the mixing and compaction process more difficult. The loss of strength (S or S/F) due to the higher porosity of the HMAs with ZnO as a filler had to have been compensated by the lower AC content and the higher stiffness exhibited by the mastic with the inclusion of ZnO, as will be shown in the next section.

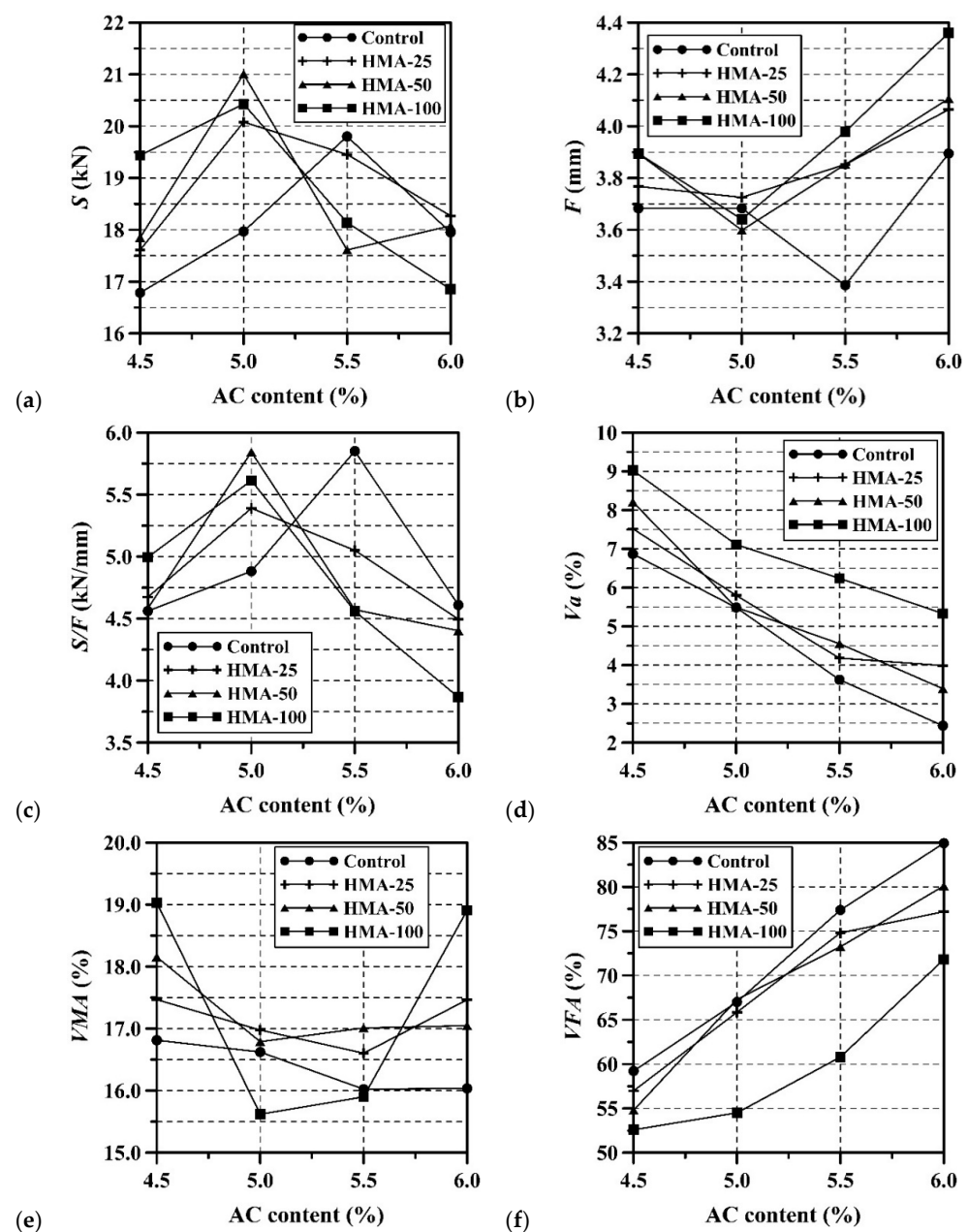


Figure 5. Marshall test results. (a) S ; (b) F ; (c) S/F ; (d) V_a ; (e) VMA ; (f) VFA .

Table 4. ANOVA—Marshall test.

Comparison	<i>S</i>	<i>S/F</i>	<i>Va</i>
		<i>F_T</i>	
Control (AC = 5.5%) vs. 25% (AC = 5.0%)	0.92	5.26	187.7 *
Control (AC = 5.5%) vs. 50% (AC = 5.0%)	1.57	0.001	105.9 *
Control (AC = 5.5%) vs. 100% (AC = 5.0%)	3.51	1.91	1316.3 *

* Significant values ($F_T > F_{0.05} = 7.71$).

Based on the results obtained, an asphalt binder content of 5.5% was selected as the OAC for the Control HMA, as this percentage satisfies the design criteria established by standards in [71] and/or [72]. In the case of the mixtures in which NFs were partially or fully replaced by ZnO, an OAC of 5.0% was selected. This decision was made despite the fact that the HMA-100 mixture (with 100% NFs replacement) does not fully comply with the recommended volumetric ranges. Specifically, HMA-100 would require a higher binder content (approximately 5.7%) to meet volumetric specifications; however, such an increase would compromise mechanical performance, as evidenced by a significant reduction in *S* and the *S/F* ratio. Additionally, it would increase the overall cost of the mixture due to the greater binder demand. Conversely, at a binder content of 5.0%, all three modified mixtures (HMA-25, HMA-50, HMA-100) exhibited the highest strength values. These factors were the main reasons for selecting 5.0% as the binder content for HMA-100.

3.2. Conventional Properties of Asphalt Mastics

The results of the penetration tests, softening point, and viscosity curves of the asphalt mastics are shown in Figure 6a, b, and c, respectively. The penetration and softening point results show that the stiffness of mastic asphalt tends to increase as the NFs replacement content with ZnO grows (softening point increases, and penetration decreases). However, in the viscosity results, the trend is different, as the highest viscosity is achieved at a ZnO/NF ratio of 100%, followed by 50%, 75%, and 25%. Despite this, in general terms, ZnO increases the viscosity and stiffness of the binder. This increase in stiffness is mainly due to the higher asphalt–filler interaction between AC and ZnO due to the smaller size of ZnO nanoparticles (Figure 3) and its higher alkalinity compared to NFs (NFs feature inert minerals such as quartz that are chemically neutral and of low asphalt–filler interaction compared to other minerals) [4,79–81]. The ZnO nanotube agglomerates observed under SEM exhibit a higher effective mass and occupy a larger volume compared to individually dispersed particles. This phenomenon may increase the viscosity of the asphalt mastic due to the physical interaction between the nanotubes, which restricts the mobility of the asphalt binder [73,82]. Additionally, in ZnO/NF = 0%, the higher binder content with respect to the filler could contribute to a decrease in the stiffness of the asphalt mastic (the filler-to-binder ratio is lower). On the other hand, the surface texture of NFs is predominantly smooth (Figure 3), which could affect the shear resistance of the asphalt–filler interface [83].

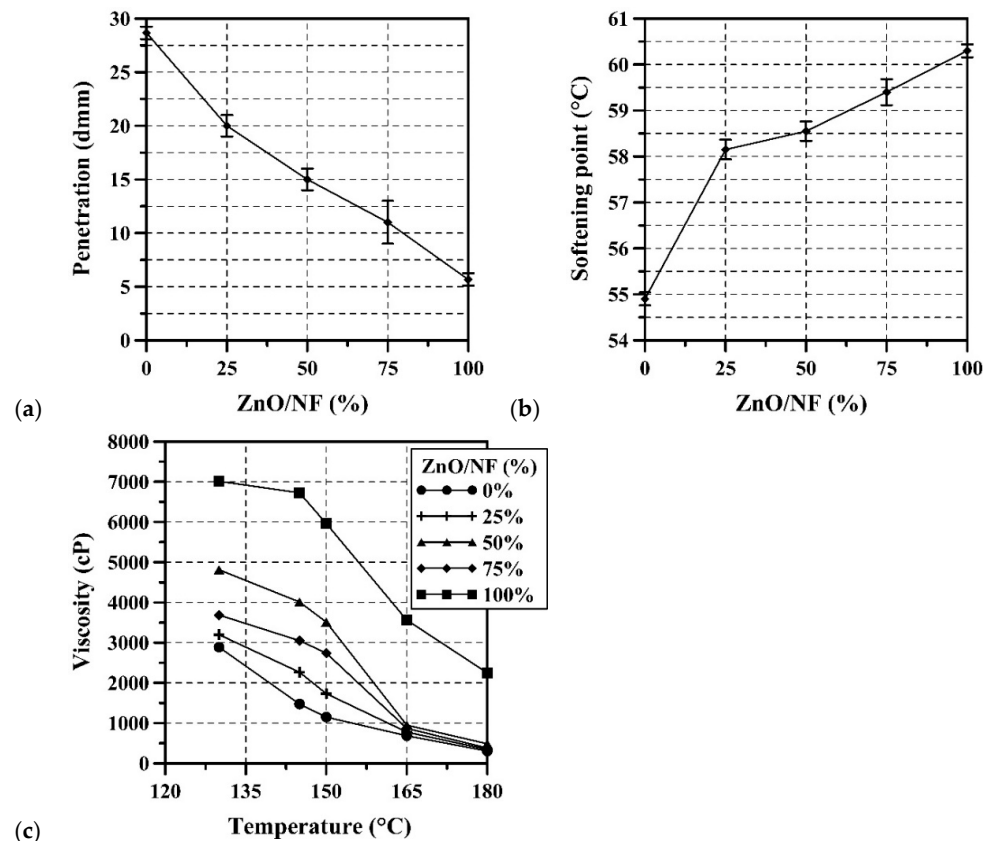


Figure 6. (a) Penetration; (b) softening point; (c) viscosity of asphalt mastics.

3.3. ITS and Cantabro Tests

The results of the ITS and Cantabro tests are shown in Figures 7 and 8, respectively. The *ITSD* and *ITSC* parameters of the HMAs with NFs replacement by ZnO exhibit higher and statistically significant values ($F_T > F_{0.05} = 7.71$; Table 5) with respect to the Control HMA. Moreover, both parameters tend to increase markedly (Table 5) with increasing replacement percentage (except for the *ITSC* of HMA-100, which is lower with respect to HMA-50, but this decrease is not statistically significant according to Table 5). The increase in *ITSD* is 5.8, 10.5, and 21.0% when replacing 25, 50, and 100% of NF by ZnO. For the case of *ITSC*, these increases are 9.0, 25.5, and 21.1%, respectively. These increases are consistent with the increase in stiffness exhibited by the asphalt mastics when NF was replaced by ZnO (Figure 6). The *TSR* parameter is also higher in the HMAs using ZnO as a filler (*TSR* = 86.8, 89.4, 98.6, and 86.9% for Control, HMA-25, HMA-50, and HMA-100%, respectively). The decrease in the *TSR* of HMA-100 with respect to HMA-50 is mainly due to the higher porosity of HMA-100. On the other hand, the abrasion and raveling resistance of the HMA with ZnO is similar with respect to the Control mixture (statistically similar *CL*, see Table 5). All the above indicate that ZnO helped to improve binder-aggregate adhesion and resistance to moisture damage, even though the HMA with ZnO has lower asphalt binder content and higher porosity (higher *Va*). This could be because replacing NFs with nano-ZnO (alkaline material) reduces the acidic component and increases the basic component of the binder, improving its adhesion with the aggregate [5,11,69,84]. Additionally, NF presents high quartz content, low CaO content (low ratio CaO/SiO_2), low absorption, and smooth surface texture that can induce loss of adhesion with the binder [77,85–87]. On the other hand, ZnO has been shown to improve the aging resistance of binders and HMAs [41,48,49,60–62] and is a material that can be incorporated into polymeric matrices, improving ductility [41], which could impact the results of the ITS

and Cantabro test (less brittle binder and mixtures; [88]. In addition, ZnO tends to interact ionically with other materials, increasing the hydrophobicity and interfacial tension of the samples, which, together with a high surface roughness of the ZnO nanoparticles, could help to improve the resistance to moisture damage [89].

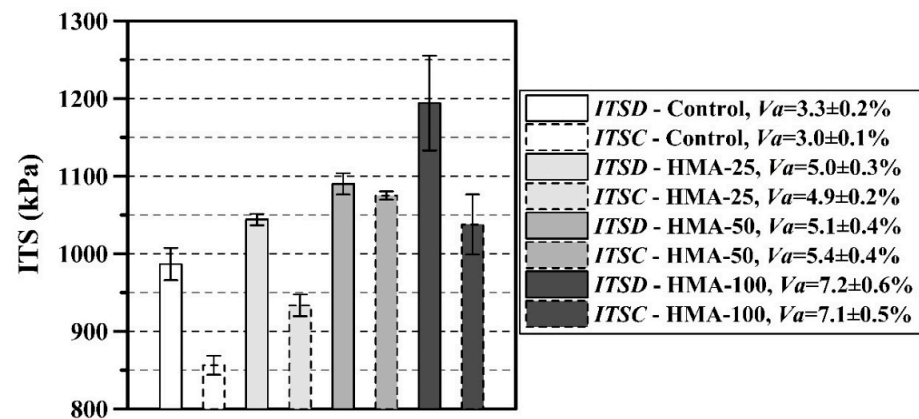


Figure 7. ITSD and ITSC parameters.

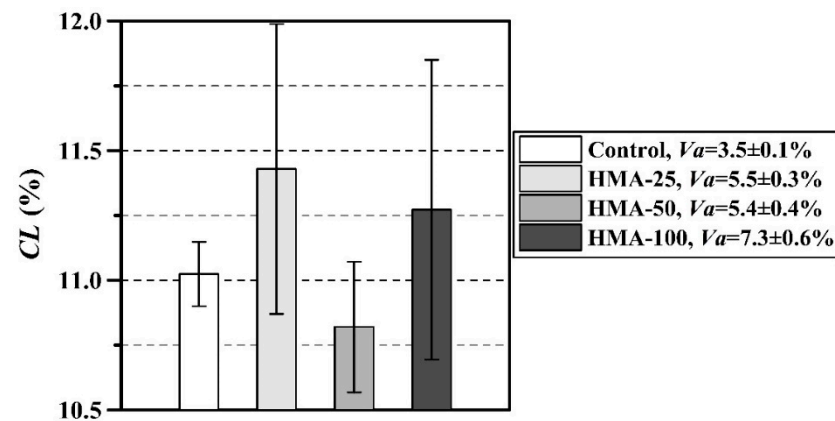


Figure 8. Cantabro test results.

Table 5. F_T values of ANOVA—ITS and Cantabro tests.

Comparison	ITSD	ITSC	CL
Control (AC = 5.5%) vs. HMA-25 (AC = 5.0%)	20.4 *	51.5 *	1.50
Control (AC = 5.5%) vs. HMA-50 (AC = 5.0%)	52.0 *	794.4 *	1.59
Control (AC = 5.5%) vs. HMA-100 (AC = 5.0%)	31.0 *	59.5 *	0.53
HMA-25 (AC = 5.0%) vs. HMA-50 (AC = 5.0%)	26.0 *	270.1 *	2.96
HMA-25 (AC = 5.0%) vs. HMA-100 (AC = 5.0%)	17.8 *	19.2 *	0.11
HMA-50 (AC = 5.0%) vs. HMA-100 (AC = 5.0%)	8.3 *	2.7	1.54

*Significant values ($F_T > F_{0.05} = 7.71$).

3.4. Resilient Modulus and Static Creep Tests

The results of the RM and static creep tests are shown in Figures 9 and 10, respectively. The mix that shows the highest stiffness under cyclic loading was HMA-50. The RM of HMA-50 increased by 46.2, 29.9, and 20.7% over Control HMA for loading frequencies of 2.5, 5.0, and 10 Hz, respectively, and these increases were statistically significant ($F_T > F_{0.05} = 7.71$; Table 6). Compared to HMA-100, HMA-50 also exhibited higher RM (5.7, 18.0, and 19.8% higher for 2.5, 5.0, and 10 Hz loading frequencies, respectively). HMA-100 tends to experience higher RM than the Control HMA (increases of 38.2, 10.1, and 1.0% for 2.5, 5.0, and 10 Hz frequencies, respectively); however, only for the 2.5 Hz frequency was the change in stiffness statistically significant (Table 6). Generally, more porous mixtures tend to experience lower RM. However, the higher RM of the HMA with ZnO could be due

to the lower OAC content, the higher values obtained in the ITS test [90], and the increase in stiffness of the asphalt mastics when NF is replaced by ZnO [3].

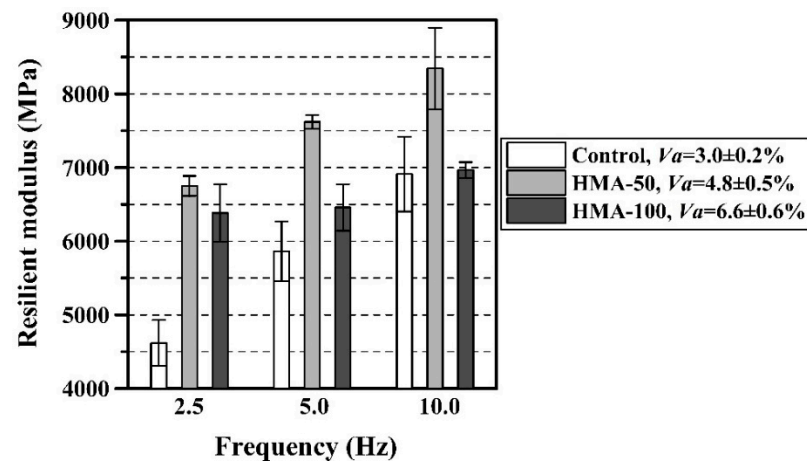


Figure 9. Resilient modulus test results.

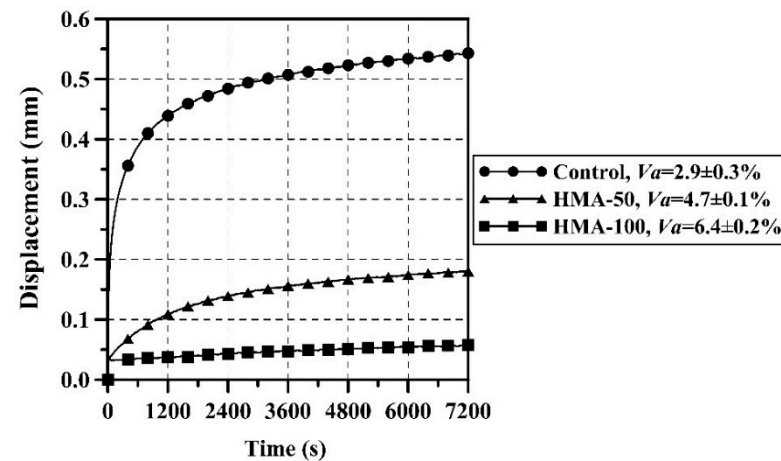


Figure 10. Displacement vs. time (creep static test).

Table 6. ANOVA—resilient modulus test.

Comparison	2.5 Hz	5.0 Hz	10.0 Hz
	F_T		
Control vs. HMA-50	118.1 *	53.1 *	11.0 *
Control vs. HMA-100	37.5 *	4.0	0.03
HMA-50 vs. HMA-100	2.3	37.4 *	18.0

* Significant values ($F_T > F_{0.05} = 7.71$).

The static creep test shows that the HMAs with ZnO exhibit higher resistance to permanent deformation compared to the Control HMA. This is mainly due to the higher *RM* exhibited by the HMA-50 and HMA-100, and the increased stiffness of the asphalt mastic (Figure 6). The displacement (*D*) at 7200 s was 0.543, 0.180 and 0.058 mm for the Control, HMA-50, and HMA-100 mixtures, respectively, while the displacement rate (*DR*) measured between 3600 and 7200 s was 10×10^{-6} , 6.67×10^{-6} , and 3.06×10^{-6} mm/s, respectively. Likewise, it is observed that HMA-100 shows the highest resistance despite being more porous and presenting a lower *RM* and *S/F* ratio compared to HMA-50. There is no clear explanation for this. However, perhaps the higher surface roughness and amorphous geometry of the ZnO agglomerations (generated by the granular skeleton formed by its rod-like shape tubular nanoparticles; Figure 3) could have influenced the higher resistance

to permanent deformation. The agglomerates of tubular ZnO nanoparticles observed under SEM may form reinforcement structures within the asphalt matrix. These three-dimensional reinforcements can enhance the stiffness of the mixture, contributing to greater resistance to plastic deformation or rutting [73,82]. On the other hand, the addition of a nanometal such as ZnO could increase the toughness of a polymer matrix [91], especially considering that ZnO nanoparticles tend to possess high hardness, structural stiffness, and compressive strength [92,93]. The incorporation of ceramic nanoparticles such as ZnO into the polymeric network could improve the mechanical and thermal stability [94].

3.5. Fatigue Resistance Test

The results of the fatigue test under the stress-controlled mode are shown in Figure 11. Under the stresses analyzed, the HMAs with ZnO demonstrate higher fatigue strength, with respect to the Control mix, and the increases in N_f are statistically significant (except for the 250 kPa stress when comparing the Control mix and the HMA-100; Table 7). Statistically, HMA-50 shows similar fatigue strength at 150 and 250 kPa, but higher strength at 350 kPa than HMA-100. It is interesting to note that the HMA-50 and HMA-100 mixtures exhibit better performance in the fatigue test despite presenting higher V_a and lower OAC with respect to the Control HMA. However, the increase in fatigue strength of HMA-50 and HMA-100 is consistent with the ITS test and RM results (increases in $ITSD$ and RM tend to increase fatigue strength under stress-controlled [78,89,95–98].

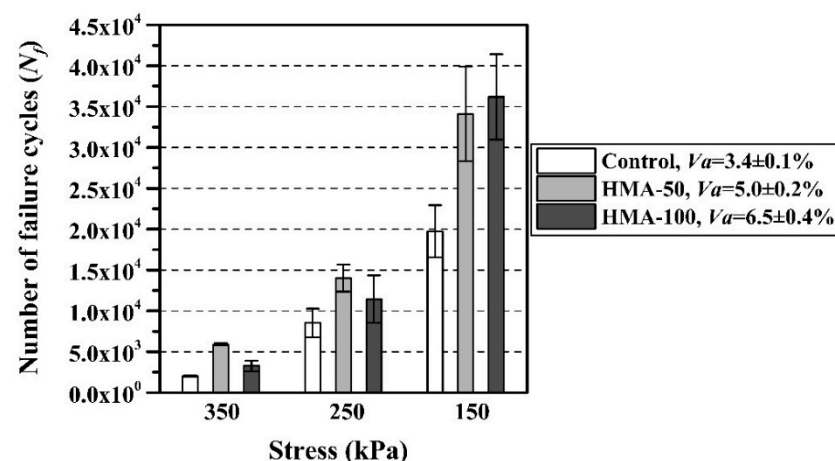


Figure 11. N_f evolution with stress (fatigue test).

Table 7. ANOVA—fatigue resistance test.

Comparison	350 kPa	250 kPa	150 kPa
	F_T		
Control vs. HMA-50	3523.9 *	31.10 *	28.20 *
Control vs. HMA-100	21.71 *	4.49	43.36 *
HMA-50 vs. HMA-100	91.66 *	3.56	0.43

* Significant values ($F_T > F_{0.05} = 7.71$).

3.6. Summary of Results

To help evaluate the influence of ZnO as a replacement for NF on the mechanical performance of HMA, the parameters obtained in the tests were qualitatively characterized on a scale of 1 to 3 (Table 8), where 1 and 3 correspond to the magnitude of the lowest and highest value, respectively. In Table 8, if S , S/F , $ITSD$, $ITSC$, TSR , RM, and N_f , tend to 3, it means that the HMA tends to exhibit better performance, since the strength parameters under monotonic load in the Marshall test (S and S/F) and in indirect tension ($ITSD$ and

ITSC) are higher, as well as the resistance to moisture damage (*TSR*) and the stiffness under cyclic load (*RM*). The opposite is true for parameters *D*, *DR*, and *CL*, since a value of 1 would indicate better performance (less displacement and mass loss in static creep and Cantabro tests). In summary, Table 8 shows that the best performance is exhibited by the HMA-50 and HMA-100 mixtures. That is, ZnO has a positive influence on the mechanical performance of HMA when used as a replacement for NF. Despite these findings, it is important to highlight that the higher porosity of the HMA-50 and HMA-100 mixtures could potentially affect their long-term performance (e.g., binder stripping, premature aging, internal structural weakening, freeze–thaw damage in cold climates, etc.), a factor that should be considered in future studies.

Table 8. Qualitative characterization of the results.

Test	Parameter	Qualitative Value		
		Control	HMA-50	HMA-100
Marshall	<i>S</i> (kN)	1	3	2
	<i>S/F</i> (kN/mm)	2	3	1
ITS	<i>ITSD</i> (kPa)	1	2	3
	<i>ITSC</i> (kPa)	1	3	2
	<i>TSR</i> (%)	1	3	2
Cantabro	<i>CL</i> (%)	2	1	3
Resilient Modulus	<i>RM</i> (MPa)	1	3	2
Static creep	<i>D</i> (mm)	3	2	1
	<i>DR</i> (mm/s)	3	2	1
Fatigue	<i>N_f</i> (-) to $\sigma = 150$ kPa	1	2	3
Fatigue	<i>N_f</i> (-) to $\sigma = 250$ kPa	1	3	2
Fatigue	<i>N_f</i> (-) to $\sigma = 350$ kPa	1	3	2

4. Conclusions

The present study evaluated the influence that ZnO has when used as a replacement for NF in HMA at ZnO/NF = 25, 50, and 100% ratios. The conclusions obtained are presented below.

- When using ZnO as a replacement for NF, the OAC of the HMA decreases by 0.5%. At this percentage, the HMAs with ZnO (HMA-25, HMA-50, and HMA-100) are more porous (*V_a* increases), but exhibit the following with respect to the Control HMA:
 - Similar resistance under monotonic loading in the Marshall test (similar *S* and *S/F* ratio).
 - Increased *ITSD*, *ITSC*, and *TSR*, indicating increased binder–aggregate adhesion, and increased resistance to moisture damage.
 - Similar *CL* or resistance to abrasion and raveling.
- Under cyclic loading, HMA-50 and HMA-100 exhibit the following:
 - Higher stiffness under cyclic loading (higher *RM*). However, from a statistical point of view, HMA-50 exhibited higher *RM* with respect to HMA-100 and Control HMA, while the latter two mixtures tended to experience a similar *RM*.
 - Higher resistance to permanent deformation measured in the static creep test. This resistance tends to increase with the increasing replacement of NF by ZnO.
 - Higher fatigue resistance under stress-controlled conditions; the highest resistance was obtained with HMA-50.

In summary, most of the evaluated mechanical properties of the HMA improved when ZnO was used as a replacement for NF, even though *V_a* increases. That is, ZnO is a promising material as a replacement for NFs in HMAs. However, it is recommended that

future studies take into account the potential impact of increased porosity in HMA-50 and HMA-100 mixtures on their long-term performance.

Some recommendations for future work: (i) evaluate RM and resistance to permanent deformation at higher temperatures; (ii) evaluate rheological properties of asphalt mastics; (iii) evaluate low-temperature properties and resistance to aging; (iv) use different types of NF, gradations of HMAs, types of ACs, etc.; (v) perform simulations and evaluations of environmental and economic impact; and (vi) perform tests to assess the distribution and dispersion of ZnO within the asphalt mastic and the asphalt mixture.

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