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Assessment of asphalt binder content, temperature and loading rate in indirect tensile strength and resilient modulus tests of a hot-mix asphalt – Comparison with Marshall design method

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ABSTRACT

The optimum asphalt content (OAC) in a hot-mix asphalt (HMA) is an important parameter that affects pavement performance and durability. To obtain it, it is essential to consider variables associated with the action of the climate and the rate of load application. The traditional way of obtaining the OAC through the Marshall test does not consider these variables. Additionally, this test is more appropriate to evaluate the performance of HMAs in high-temperature climates (mainly to evaluate rutting resistance) and does not consider the types of distresses that predominate in temperate and/or cold climates (e.g., fatigue cracking). This study analyzed the influence of asphalt binder content on the performance of an HMA by performing indirect tensile strength (ITS) and resilient modulus (RM) tests, considering temperatures of 10, 20, and 40 °C, with load application rates of 10, 30 and 50 mm/min for the ITS test, and loading frequencies of 2.5, 5.0 and 10.0 Hz for the RM test. From the ITS test, the peak load (P), the slope at the inflection point (m), the stiffness (St), and the work of fracture (Wf) were obtained. The performance of the HMA based on these parameters was evaluated and an OAC was defined and compared with that obtained traditionally by the Marshall test. Additionally, these values were compared with the results obtained from resilient modulus (RM) tests. A mathematical correlation between the ITS and RM tests was reported. Temperature, speed of load application in the ITS test, and frequency of load in the RM test affected the performance of the HMA. P, ITS, St, Wf, and RM increased when temperature decreased and loading speed or frequency increased. The opposite was true for parameter m. For the HMA evaluated in the present study, the results show that the OAC obtained from ITS and RM tests is 0.5% higher than that obtained from the Marshall test. In other words, HMA design methods should include ITS and RM tests to help reduce susceptibility to cracking in temperate climates and rutting in high-temperature climates. It's suggested to conduct similar studies using different aggregates, asphalts, and gradations.

1. Introduction

1.1. Problem statement

Colombia is a country where high-temperature climate predominates in most of its regions. The annual average temperature (AAT) can vary between 26 and 27°C, and in this type of climate, the main type of distress in hot-mix asphalt (HMA) is rutting. However, there are important cities such as Bogotá (capital of the country with 14,536.7 km-lane as of June 2021 [1]) where the AAT varies between 13 and 15°C, with minimum and maximum temperatures varying between 8 and 10°C, and 18–20°C, respectively [2,3]. In this type of climate, the predominant type of distress in asphalt mixtures is fatigue cracking. In the world, the main method used for the design (HMA) is the Marshall method [4]. This is mainly due to its wide knowledge and use in the world, low cost, and ease of implementation. Its rationale is based on determining the volumetric composition (e.g., air voids - Va, voids in mineral aggregate - VMA and voids filled with asphalt - VFA) and strength parameters of the asphalt mixture (stability - S, flow – F, and S/F ratio). S and F can be understood as the resistance under monotonic load and the maximum displacement in the failure state, respectively, that a sample undergoes when subjected to elevated temperature (60°C). In other words, it can be understood as an indicator of rutting resistance. Additionally, the S/F ratio is an indirect indicator of shear strength

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Table 1
Characterization of aggregates.

Test	Standard	Requirement	Result
Abrasion in Los Angeles machine (%)	AASHTO T96 2002	maximum 25%	21.6
Micro-Deval (%)	AASHTO T327 2005	maximum 20%	13.5
Soundness of aggregate using magnesium sulphate (%)	AASHTO T 104 1999	maximum 18%	7.6
Fractured particles	ASTM D 5821–2001	maximum 85	89
Sand equivalent (%)	AASHTO T 176 2002	minimum 50%	85
Methylene blue (ml/g)	ASSHTO T 330–2007	maximum 10	1.75
Specific gravity of filler	AASHTO T 85 1991	-	2.71
Specific gravity of fine aggregate (real/ apparent)	AASHTO T 84 1991	-	2.64/ 2.58
Specific gravity of coarse aggregate (real/ apparent)	AASHTO T 85 1991	-	2.65/ 2.60
Coarse aggregate absorption (%)	AASHTO T 85 1991	-	0.8

Table 2
Results of physical characterization tests of asphalt binder.

Test	Standard	Requirement	Result
Original asphalt			
Penetration 25°C, 100 g, 5 s (0.1 mm)	ASTM D-5	60–70	65
Softening point (°C)	ASTM D36–95	48–54	52
Viscosity 60°C (Poises)	ASTM D-4402	1500 minimum	2600
Ductility 25°C, 5 cm/min (cm)	ASTM D-113	100 minimum	150
Flash and fire points (°C)	ASTM D-92	230 minimum	305
After RTFO (Rolling Thin Film Oven)			
Mass loss (%)	ASTM D-2872	0.8 maximum	0.55
Penetration, in % of original penetration	ASTM D-5	50 minimum	55
Increase in the softening point (°C)	ASTM D36–95	8 maximum	7

Table 3
Particle size distribution of HMA-19.

Sieve	3/4 in	1/2 in	3/8 in	N. 4	N. 10	N. 40	N.80	N. 200
Passing (%)	100	87.5	79.0	57.0	37.0	19.5	12.5	6.0

provided by the aggregate matrix and by the cohesion offered by the asphalt binder. This cohesion of the mix depends on the loading speed, the viscosity of the asphalt, and the temperature, among others. In summary, the Marshall test is based on the plastic deformation damage that occurs at a temperature of 60°C, which is perhaps relevant in hot climate zones, where temperatures above 20°C are present. However, in cities such as Bogotá, which has cold weather temperatures at night (below 13°C) and mild weather during the day (between 13 and 20°C), the main failure mechanisms of asphalt mixtures are associated with cracking. When designing HMAs by the Marshall method, relatively low optimum asphalt contents (OAC) can be obtained, since as the asphalt binder content increases, the stiffness of these mixtures tends to decrease, promoting the generation of rutting. This OAC obtained from the Marshall test could be insufficient to adequately cover the aggregates and produce a sufficiently ductile mix to adequately resist fatigue cracking. That is, in cities such as Bogotá, where temperate climate prevails, HMAs could demand higher OAC than that obtained in the Marshall test. In this sense, an inexpensive and easy-to-perform test that could be used to evaluate cracking resistance is the indirect tensile strength (ITS). The fracture parameters obtained from the area under the curve (load versus displacement) of this test could allow analyzing the performance of HMA mixtures to more appropriately determine the OAC. In conjunction with the Marshall and ITS test results, a physical parameter that could also be used to evaluate the performance of HMAs and obtain a more accurate OAC is the resilient modulus (RM), as it measures the stiffness of the material under cyclic loading at different temperatures and a loading frequency. Additionally, this parameter can be correlated with rutting and cracking resistance, since HMAs with higher RM tend to be more resistant to rutting but could experience brittle behavior at low and intermediate service temperatures, decreasing their resistance to cracking. Furthermore, this stiffness under cyclic loading can be correlated with stiffness parameters obtained from ITS providing additional and novel criteria for asphalt mixtures design.

1.2. Background

Different distresses of asphalt pavements are associated with fracture (e.g., thermal, fatigue, reflective cracking). These in turn can be related to moisture damage. This results in high maintenance and repair costs and reduced pavement service life. [5]. Generally, cracks are associated with the action of traffic and occur at low and intermediate service temperatures [6]. Several studies analyze fracture in HMA to understand the cracking phenomenon. Usually, the most common tests to analyze it are Semi-Circular Bending (SCB), disc-shaped compact tension (DCT), single-edge notched beam (SEB), Thermal Stress Restrained Specimen Test (TSRST), and ITS tests, among others. [7–9]. These tests make it possible to simulate the behavior of the mixture through the evolution of cracking with time and temperature [10]. The SCB, DCT, and SEB tests induce cracking. That is, they consider that the mixture is fractured. On the other hand, the TSRST test induces direct stress in the mix, which does not represent the real traffic stress on the pavement. Traditionally, these tests are performed using the OAC and are not considered in the mix design. It is important to highlight that the performance of HMA depends fundamentally on the OAC. Low asphalt binder contents result in loss of cohesion and adhesion, which is reflected in cracking. On the contrary, high asphalt binder contents generate a loss of stiffness, increasing the deformation of the mixture, and promoting rutting.

In the literature, several studies attempt to analyze cracking at low and intermediate temperatures, i.e. <13ºC and between 13 and 20°C, respectively. The ITS and SCB tests provide tensile properties at intermediate temperatures, which can be correlated with load cracking [11–13]. In addition, these tests can be included in mechanistic pavement designs. [14]. Given the simplicity of these laboratory tests, some studies are correlated with mechanical characteristics of the mixtures, to determine rutting, and fatigue cracking, among others [15,16]. However, it is necessary to analyze the type of load (monotonic, dynamic, static), speed of load application, and temperature, among other aspects, in order to establish valid correlations between the tests and the parameters obtained. Likewise, it is necessary to consider the crack propagation rate, which can be adequately evidenced by dynamic load tests [17]. [18,19] established correlations between fatigue resistance from ITS and SCB tests. At intermediate temperatures, it was shown that asphalt mixtures with higher ITS are more resistant to fatigue cracking. [20] established a correlation between ITS and fatigue strength using a parameter called Fatigue Index (FI). FI is determined from the ITS test as the ratio of the fracture energy and slope of the load vs displacement curve at the inflection point. Higher values of FI indicate higher fatigue strength. Some correlations were also evidenced in novel HMA composite fabricated with a combination of crumb rubber (CRM) and polyvinyl alcohol (PVA) fiber [21,22]. Also, [23,24] established models to predict rutting resistance from tests under monotonic loading. Several studies analyze the effect of load application rate and temperature by SCB and ITS testing. [25–28]. The temperature ranges comprise both

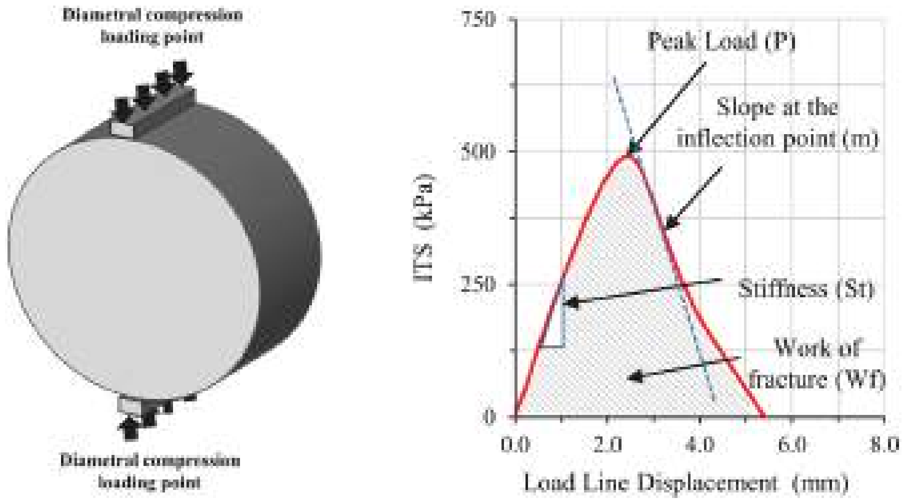

Fig. 1. Test configuration and parameters obtained.

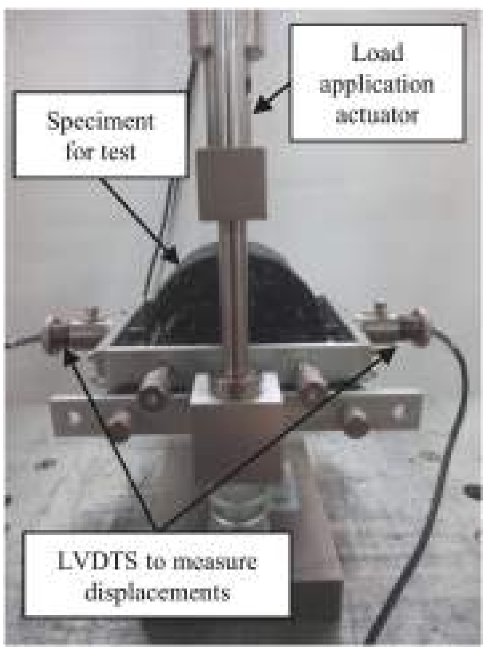

Fig. 2. Resilient modulus test.

low and high temperatures (between −10 and 40°C). The load application rate ranges from 0.1 to 200 mm/min. Generally, results and statistical analyses indicate that: i) Fracture parameters such as peak load, stiffness, and fracture energy of the mixtures depend on the rate of load application; ii) These parameters increase with the rate of load application and decrease with increasing temperature. In addition, it is recommended that tests at intermediate temperatures near 25°C be run at a load application rate between 5 and 20 mm/min. [29] iii) The results presented in fine asphalt mixtures (FAM) can be correlated to understand the behavior of HMA since cracking initially develops in the asphalt mastic; iv) At low temperatures, the behavior of the mixture depends on the aggregate; v) At elevated temperatures between 30 and 40°C, the performance depends mainly on the asphalt viscosity [30] analyzed the effect of aging of an HMA on fracture and fatigue characteristics at intermediate temperatures (25°C). In the laboratory, they simulated different levels of aging. Experimental findings suggest that aging reduces ductility, increases stiffness, and generates increased cracking. A reduction in fatigue strength was also observed with increasing aging. Other studies have been carried out to numerically simulate SCB and ITS tests using finite element techniques, and discrete element techniques, among others [26,31,32]. Simulations using the laws of fracture mechanics and viscoelastic models allow predicting the initiation and dissipation of cracks in HMAs with high accuracy concerning those evidenced in the field [33]. The above could help to predict the behavior of the mixture numerically, allowing savings in costs and experimental time [26].

On the other hand, mixture design methods seek to establish the OAC to ensure aggregate particle coverage. [34] analyzed the asphalt sheet thickness coating the aggregates in an HMA, obtaining that the best performance occurs between 9.5 and 10.5 μm. This is in search of an adequate balance or interaction between the materials that make up the mixture (air, asphalt binder, and aggregates). Some parameters that allow controlling the balance are Va, VMA, and VFA. Additionally, some design methodologies determine the mechanical stability of the mix under failure conditions. The main tests for mix design are Hveem, Marshall, and Superior Performing Asphalt Pavements (SUPERPAVE), among others. However, for the design of asphalt mixtures with modified asphalts or alternative aggregates, analyses with stiffness tests (resilient and dynamic modulus) are also included, which are correlated with the ITS test [35,36]. New studies report the determination of OAC from artificial intelligence using neural networks. However, a large database is necessary for its calibration [37,38].

1.3. Study objective

Based on the literature review, it became evident that numerous studies analyze fracture properties at low and intermediate temperatures, applying different load application rates. One of the most widely used fracture tests is the SCB. However, this test considers cracking of the material, which does not represent the initial conditions of the mixture in the pavement. On the other hand, most fracture studies evaluate the behavior of HMAs manufactured with the OAC obtained from Marshall or Superpave tests. Few studies have been conducted to determine the OAC from fracture parameters, which are more suitable for evaluating the performance of HMAs in temperate climates. Additionally, few studies on the subject include in the evaluation the stiffness under cyclic loading through the resilient modulus.

The objective of this study includes:

- Evaluate the influence of asphalt binder content, temperature, and loading rate on the fracture parameters obtained in ITS tests in an HMA. To this end, samples with four asphalt binder contents were manufactured and ITS tests were performed under three

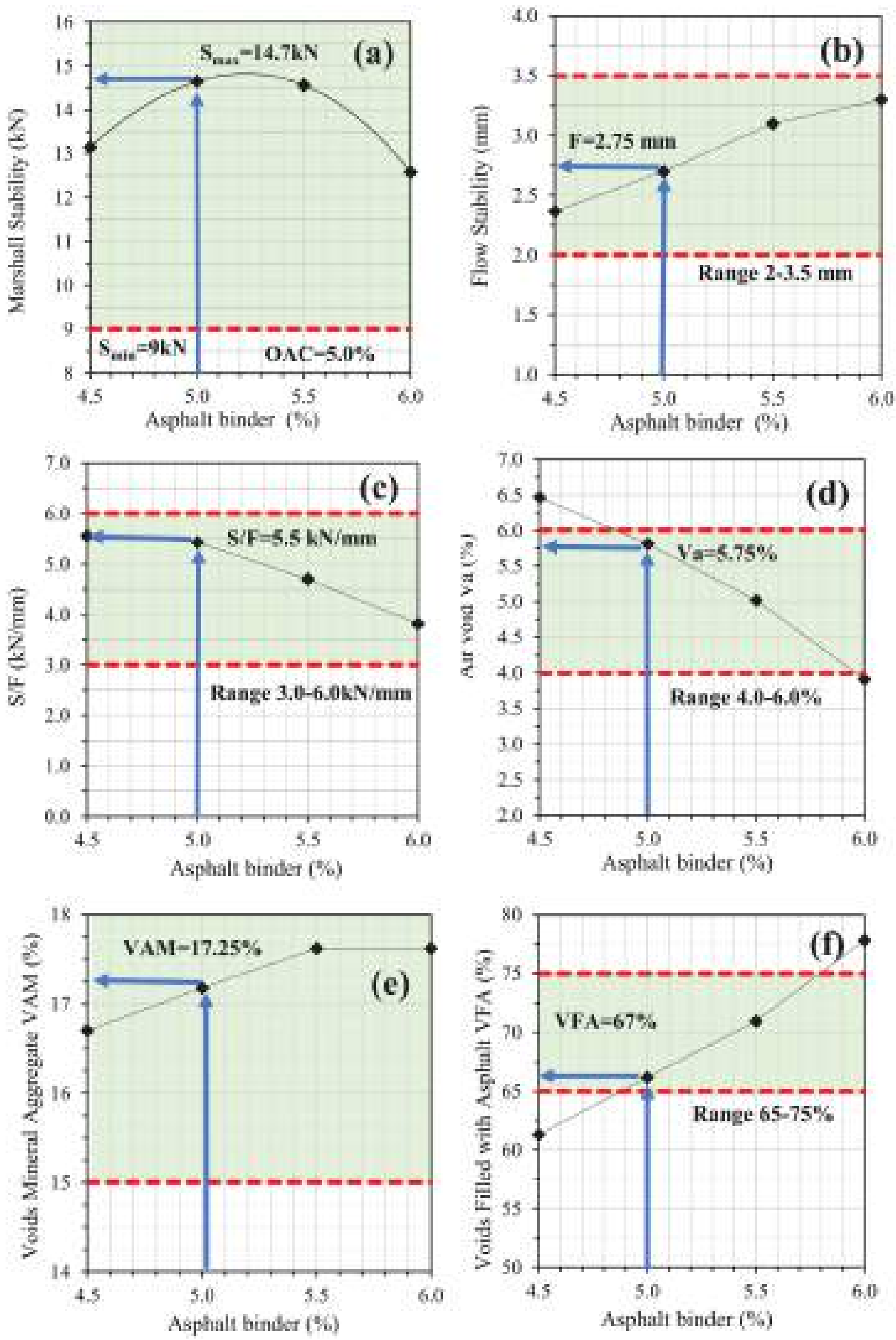

Fig. 3. Marshall Test results: a) Stability Marshall; b) Flow Marshall; c) S/F ratio; d) Va; e) VMA; f) VFA.

temperatures (10, 20, and 40°C) and three loading rates (10, 30, and 50 mm/minute).

- Compare the results obtained from the first experimental phase with those obtained from RM Tests Also performed on samples manufactured with the Same Four Asphalt Binder content. The temperatures of the RM tests were the same and three loading frequencies (2.5, 5.0, and 10.0 Hz) were used.
- Finally, the OAC obtained from the Marshall test was compared with that obtained from ITS and RM tests.

2. Materials and methodology

The Marshall method was used for the design of the HMA considering the conditions of Colombia of the INVIAS [39]. AC contents of 4.5%, 5.0%, 5.5% and 6.0% were used. Then, Indirect tensile strength (ITS) and resilient modulus (RM) tests they were made using the same AC contents. The tests were performed at temperatures of 10, 20 and 40 °C to simulate the action of the weather. load application rates of 10, 30 and 50 mm/min for the ITS test, and loading frequencies of 2.5, 5.0 and 10.0 Hz for the RM test, purpose to analyze the effect of the speed loading in the mixture. From the ITS test, peak load (P), the slope at the

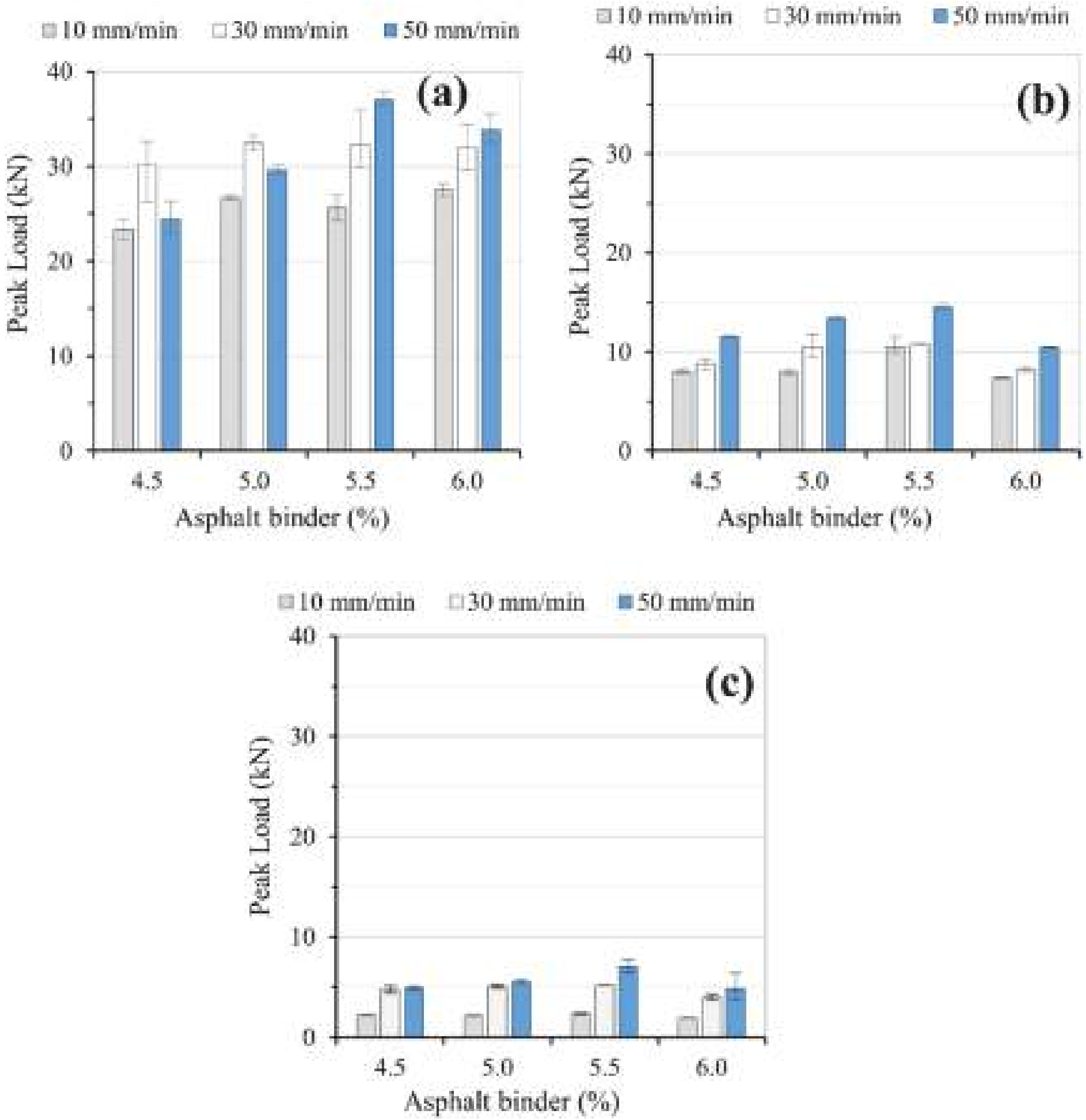

Fig. 5. Peak Load at temperatura of: a) 10 °C; b) 20 °C; c) 40 °C.

inflection point (m), the stiffness (St) and the work of fracture (Wf), Fracture energy (Gf), Flexibility Index (FI) and Cracking Index (CRI). Statistical analysis ANOVA and the coefficients of variation (CV) were calculated to analyze the variability of the results. A mathematical correlation between the ITS and RM tests was reported. The performance of the HMA based on these parameters was evaluated and an OAC was defined and compared with that obtained traditionally by the Marshall test. Additionally, these values were compared with the results obtained from resilient modulus (RM) tests.

2.1. Materials

The main physical characteristics of the materials used are shown in Tables 1 and 2. The aggregates are sandstones from the city of Bogotá (Colombia). The asphalt cement corresponds to an asphalt cement AC 60–70 according to its penetration grade in dmm. The results indicate that the materials meet the quality requirements of the construction specifications of the "Instituto Nacional de Vías" – INVIAS [33].

2.2. Mix design - Marshall test

For the HMA design by the Marshall method (AASHTO T 245), the aggregates were adjusted to the particle size required by the INVIAS specification for HMA with a nominal maximum size of 19 mm (Table 3) [33]. Samples with four AC contents were analyzed: 4.5%, 5.0%, 5.5%, and 6.0% to the total mass. These percentages were chosen because they are the most used by asphalt plants for the design of HMAs in the city of Bogotá. In total, 12 samples were manufactured, three for each asphalt binder content. Each sample was manufactured with 1200 g and compacted with 75 blows on both surfaces. The dimensions of each sample correspond to approximately 101 mm and 63.5 mm in diameter and height, respectively. According to the asphalt equi-viscosity method, the temperature ranges for mix manufacture and compaction were 150±3°C and 140±3°C, respectively. To determine the OAC, the volumetric composition was analyzed: Va, VMA, and VFA. Additionally, the strength parameters S, F, and S/F ratios were determined.

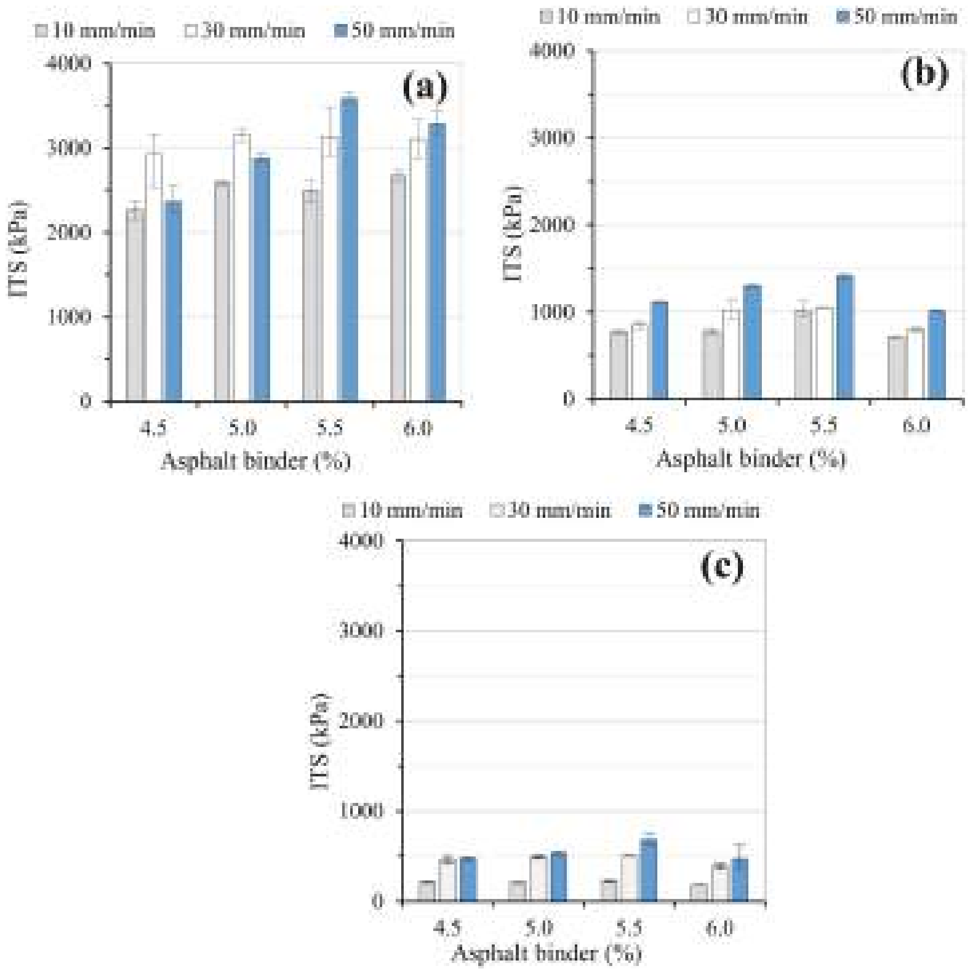

Fig. 6. ITS at temperature of a) 10 °C; b) 20 °C; c) 40 °C.

2.3. Indirect Tensile Strength (ITS) tests

For the four asphalt binder contents evaluated in the Marshall test, the ITS test was performed. The tests were performed at temperatures of 10°C, 20°C and 40°C. Previously, the samples were subjected to a temperature chamber for 24 hours at 10°C and 20°C, respectively. For the 40°C temperature, the samples were subjected to a water bath for 2 hours. Loading speeds of 10, 30, and 50 mm/minute were applied. Each sample was subjected in a Material Testing System (MTS) to the action of the monotonic load until rupture. During the test, the following data were obtained, plotting the evolution of ITS with vertical displacement (Fig. 1):

- Peak Load (P): Corresponds to the maximum point in kN.
- Stiffness (St): It is estimated as the slope of the secant line between 0% and 50% of the peak load (kN/mm) [28].
- ITS: Corresponds to the indirect tensile strength, determined according to Eq. (1). P corresponds to a Peak Load in N, d, and h to the diameter and height of the specimen in mm, respectively.
- Work of fracture (Wf): This is the area under the curve (J), which was determined by the numerical integration technique.
- Fracture energy (Gf): Corresponds to the ratio between Wf and fracture area in (J/m2). In this study, the fracture area corresponds to the failure cross-section, which was determined by the diameter and height of each sample.
- Flexibility Index (FI): It is established as the ratio between the fracture energy Gf and the slope (m) of the load curve after the maximum point (unloading stiffness). Parameter obtained in J-mm/kN.
- Cracking Index (CRI): the ratio between Gf and P (J/(kN-m2)).

A total of 108 tests were performed (4 asphalt binder contents, 3 temperatures, 3 loading rates, and 3 repetitions per condition).

$$ITS = \frac{2000 \cdot P}{\pi \cdot d \cdot h} \quad (1)$$

2.4. Resilient modulus test

For the asphalt binder contents described in the Marshall test, resilient modulus tests were performed by diametral compression according to ASTM D 4123–82 (Fig. 2). The tests were carried out in a Universal Testing Machine - UTM, applying a cyclic load of 1200 N at temperatures of 10, 20 and 40 °C. The load application frequencies correspond to 10 Hz, 5 Hz, and 2.5 Hz, respectively. In total, 36 tests were performed (4 asphalt binder contents, 3 temperatures, and 3 repetitions).

2.5. Statistical analysis of the results

To analyze the variability of the results, coefficients of variation (CV) were calculated for the parameters measured. ANOVA analyses of variance with a 95% confidence level were also performed to evaluate whether the variations in the results and calculated parameters were statistically significant. The analyses performed correspond to i) a Comparison of the parameters obtained for each asphalt binder content; and ii) a Comparison of the parameters for each load application speed.

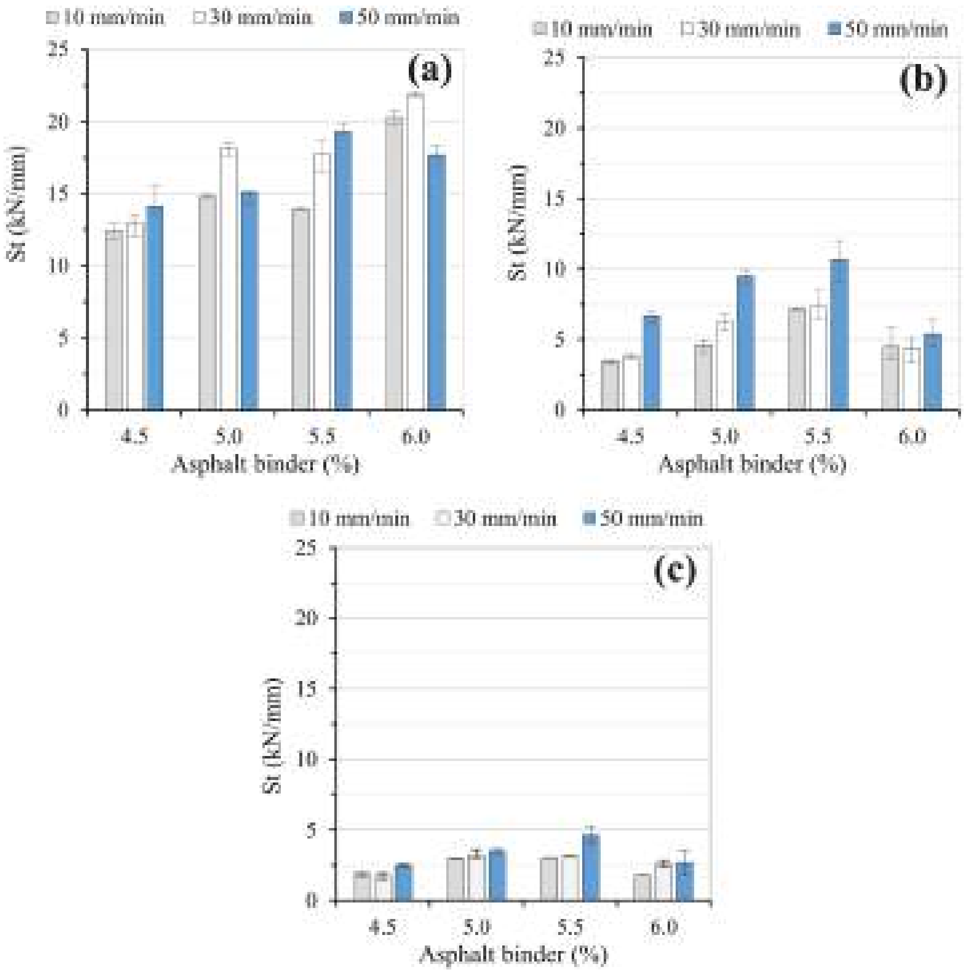

Fig. 7. Stiffness at: a) 10 °C; b) 20 °C; c) 40 °C.

3. Results and discussions

3.1. *Asphalt mixture design – Marshall test*

The OAC obtained in the Marshall test (Fig. 3) corresponds to 5.0%. The criteria established by INVIAS [33] are: Va between 3% and 6%; VMA minimum 15%; VFA between 65% and 75%; S Minimum 9 kN; F between 2.0% and 3.5% and S/F between 3 and 6 kN/mm. In other words, the 5.0% content provides adequate Marshall stability and S/F ratio to withstand the action of traffic in high-temperature climates, increasing rutting resistance.

3.2. *ITS test – fracture parameters*

Figs. 5 to 7 present the results of the ITS versus Load Line Displacement curves at loading rates of 10, 30, and 50 mm/min and temperatures of 10, 20, and 40 °C. The parameters P, St, Wf, and m were obtained from each curve. Higher P and St correspond to mixtures that undergo higher strength and stiffness, respectively, under monotonic loading in indirect tension. High and low values of m correspond to mixtures with brittle and ductile behavior, respectively. Wf can be understood as the energy required for the mixture to fail in indirect tension. It is also an indirect indicator of ductile behavior when accompanied by low values of m (the area under the curve increases). Higher values of Gf, CRI, and FI are understood as higher cracking resistance [40,41].

Based on the results schematized in Fig. 4, the indirect tensile strength and stiffness decrease with increasing temperature. For loading rate, the general trend is to increase strength and stiffness with an increasing rate of load application. At lower temperatures, the samples tend to exhibit brittle behavior, while at elevated temperatures the tendency is the opposite (ductile). This is consistent with previous studies [28,29,42,43]. Regardless of temperature and loading rate, the highest values of P and ITS are found with the highest asphalt binder contents (5.5 and 6.0%), while St is observed at 5.5%. That is, the lowest ITS and stiffnesses are observed at low asphalt binder contents (especially at 4.5%). Based on ANOVA analysis, the changes in P, ITS, and St tend to be statistically significant when evaluating the effect of asphalt binder content (Table 4) and loading rate (Table 5).

The load values P, ITS, and St are shown in Fig. 5, Fig. 6, and Fig. 7, respectively. In general, at 10°C, without considering the asphalt binder content and the rate of load application, higher P (between 22 and 38 kN), ITS (between 3670 and 2153 kPa) and St (between 11.8 and 22 kN/mm) are reported. At 20°C (ambient), P values between 7.3 and 14.9 kN, ITS between 703 and 1441 kPa, and St between 3.3 and 12 kN/mm are reported. While at 40°C lower values of P (between 1.9 and 7.7 kN), ITS (between 181 and 744 kPa), and St (between 1.5 and 5.3 kN/mm) were recorded. Taking as reference the values obtained from the tests at 20°C, an average increase of approximately 150% and a reduction of 57% in strength and stiffness are generated for the minimum (10°C) and maximum (40°C) temperatures, respectively. A possible relationship between P, ITS, and St is evidenced, indicating that the higher the P or ITS, the higher the displacement at peak load and consequently the higher the stiffness (St).

The results of Wf for the three temperatures and loading rates are presented in Fig. 8. The results indicate that Wf increases with decreasing temperature and increases with the rate

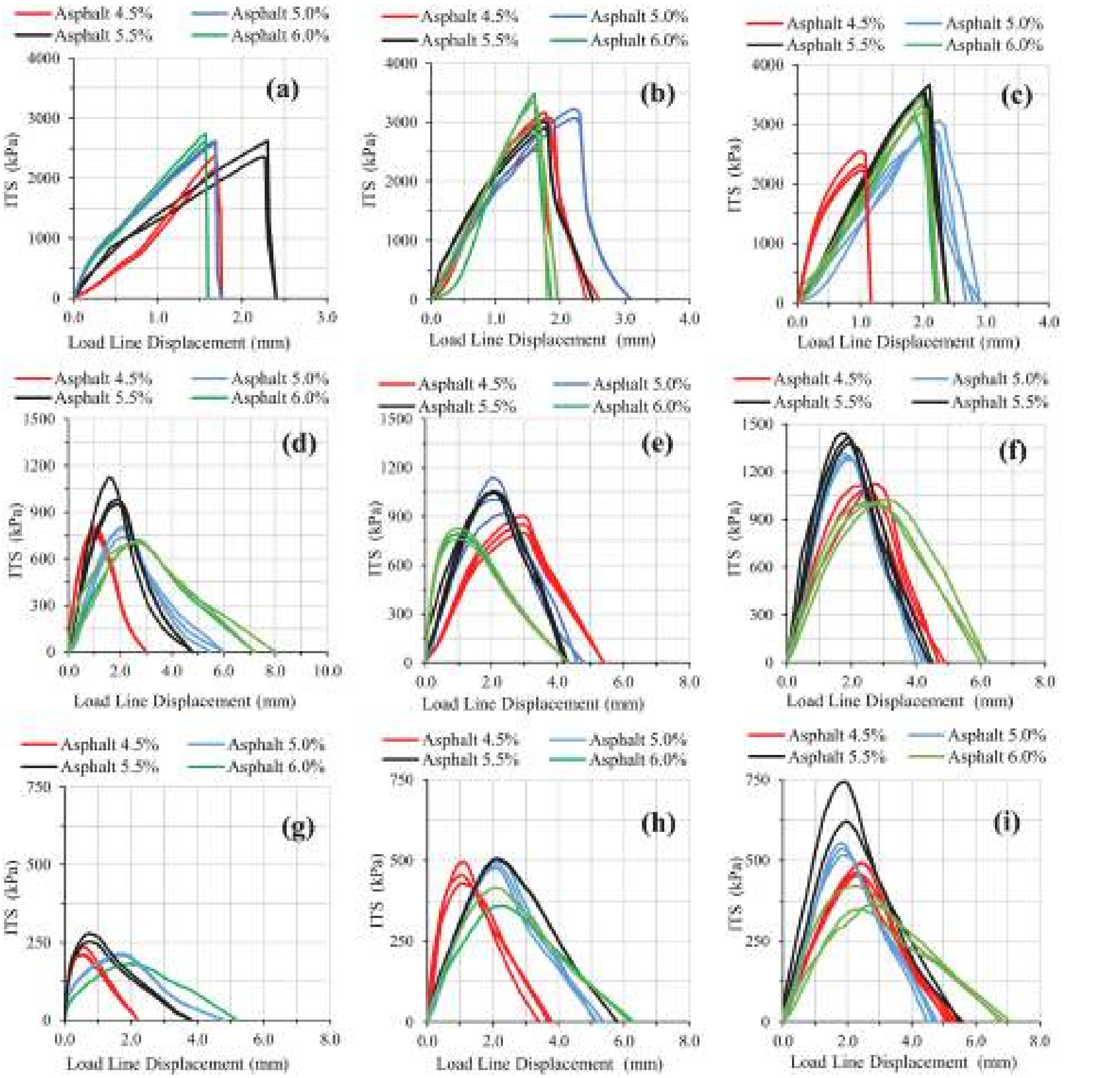

Fig. 4. ITS test: a) 10 mm/min to 10 ºC; b) 30 mm/min to 10 ºC; c) 50 mm/min to 10 ºC; d) 10 mm/min to 20 ºC; e) 30 mm/min to 20 ºC; f) 50 mm/min to 20 ºC; g) 10 mm/min to 40ºC; h) 30 mm/min to 40ºC; i) 50 mm/min to 40ºC.

Table 4
ANOVA for the significate of Peak Load, ITS and St at different asphalt binder content.

	Loading Rate (mm/min)	P-máx			ITS			St		
		F	p-value	Sig	F	p-value	Sig	F	p-value	Sig
Temperature 10ºC	10	11.1	3.18×10^{-3}	Yes	11.1	3.18×10^{-3}	Yes	223.6	4.73×10^{-8}	Yes
	30	0.5	7.09×10^{-1}	No	0.5	7.09×10^{-1}	No	70.6	4.26×10^{-6}	Yes
	50	73.8	3.59×10^{-6}	Yes	73.8	3.59×10^{-6}	Yes	41.3	3.25×10^{-5}	Yes
Temperature 20ºC	10	21.3	3.58×10^{-4}	Yes	21.3	3.58×10^{-4}	Yes	16.1	9.52×10^{-4}	Yes
	30	12.1	2.45×10^{-3}	Yes	12.1	2.45×10^{-3}	Yes	14.7	1.27×10^{-3}	Yes
	50	300.7	1.46×10^{-8}	Yes	300.7	1.46×10^{-8}	Yes	19.8	4.61×10^{-4}	Yes
Temperature 40ºC	10	9.2	5.66×10^{-3}	Yes	9.2	5.66×10^{-3}	Yes	47.5	1.93×10^{-5}	Yes
	30	15.0	1.21×10^{-3}	Yes	15.0	1.21×10^{-3}	Yes	25.9	1.80×10^{-4}	Yes
	50	5.2	2.84×10^{-8}	Yes	5.2	2.84×10^{-8}	Yes	9.9	4.59×10^{-3}	Yes

Table 5
ANOVA for the significate of Peak Load, ITS and S at different Loading Rate.

	Asphalt binder content (%)	P-máx			ITS			St		
		F	p-value	Sig	F	p-value	Sig	F	p-value	Sig
Temperature 10°C	4.5	7.52	2.3×10^{-2}	Yes	7.52	2.3×10^{-2}	Yes	3.39	1.0×10^{-1}	No
	5.0	77.69	5.1×10^{-5}	Yes	77.69	5.1×10^{-5}	Yes	62.38	9.7×10^{-5}	Yes
	5.5	23.75	1.4×10^{-3}	Yes	23.75	1.4×10^{-3}	Yes	45.22	2.4×10^{-4}	Yes
	6.0	11.75	8.4×10^{-3}	Yes	11.75	8.4×10^{-3}	Yes	94.27	2.9×10^{-5}	Yes
Temperature 20°C	4.5	85.15	3.9×10^{-5}	Yes	85.15	3.9×10^{-5}	Yes	91.00	3.3×10^{-5}	Yes
	5.0	45.68	2.3×10^{-3}	Yes	45.68	2.3×10^{-3}	Yes	69.10	7.2×10^{-5}	Yes
	5.5	46.40	2.2×10^{-4}	Yes	46.40	2.2×10^{-4}	Yes	7.42	2.4×10^{-2}	Yes
	6.0	176.58	4.7×10^{-6}	Yes	176.58	4.7×10^{-6}	Yes	0.93	4.5×10^{-1}	No
Temperature 40°C	4.5	103.22	2.3×10^{-5}	Yes	103.22	2.3×10^{-5}	Yes	8.73	1.7×10^{-2}	Yes
	5.0	454.21	$2.8. x10^{-7}$	Yes	454.21	$2.8. x10^{-7}$	Yes	5.17	5.0×10^{-2}	Yes
	5.5	108.52	1.9×10^{-5}	Yes	108.52	1.9×10^{-5}	Yes	14.64	4.9×10^{-3}	Yes
	6.0	10.55	1.1×10^{-2}	Yes	10.55	1.1×10^{-2}	Yes	2.66	1.5×10^{-1}	No

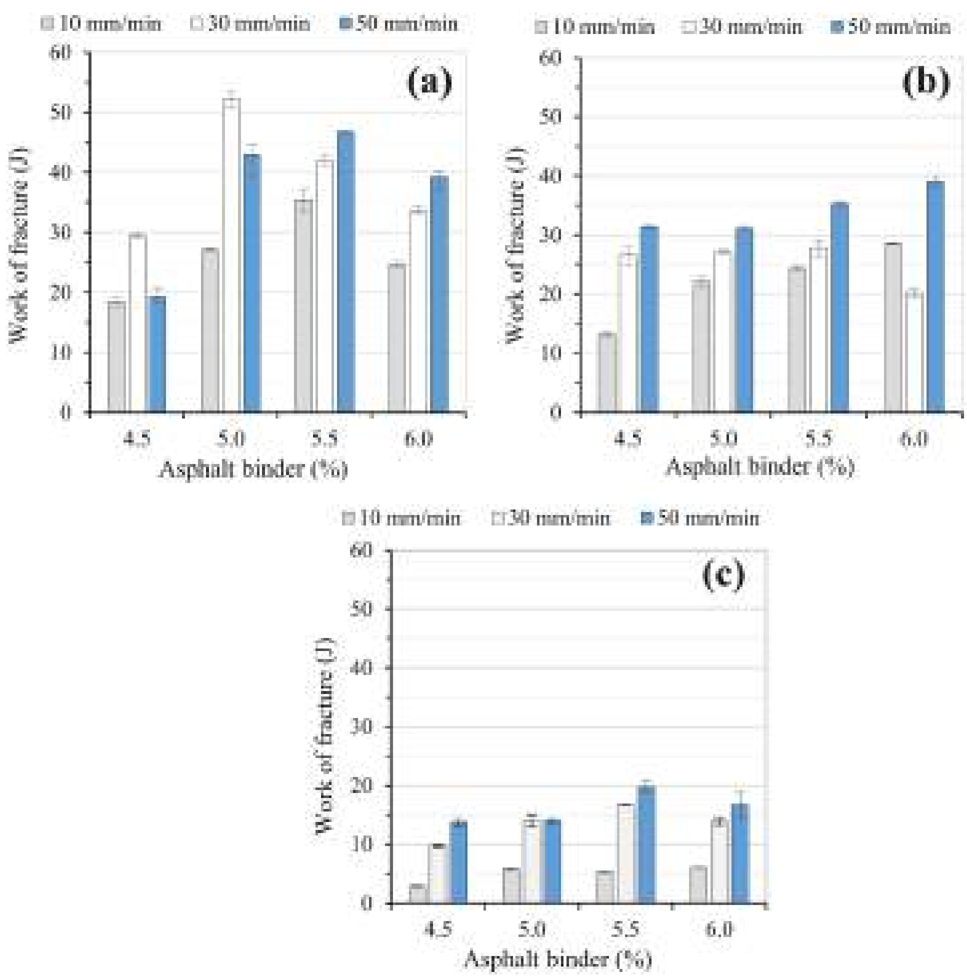

Fig. 8. Work of fracture: a) 10 °C; b) 20 °C; c) 40 °C.

of load application, except for the mixes with 4.5%, 5.0% asphalt at 30 mm/mm at 10°C, as well as for the 6.0% content at 30 mm/mm at 20°C. This may be related to similar behaviors when analyzing maximum load, ITS, and St. The ANOVA results indicate that Wf for each temperature depends on the asphalt binder content and the speed of load application. The results show that the highest Wf is obtained with 5.5% asphalt, except for the mix tested at a speed of 30 mm/min at 10°C and with 10 and 50 mm/min at 20°C, respectively. The above allows us to conclude that, for the temperatures of this study, the asphalt binder content that provides the highest Wf corresponds to 5.5%. The FI and CRI parameters are shown in Figs. 9 and 10, respectively. At temperatures of 20°C and 40°C, it is evident that the highest cracking resistance is presented with 6% asphalt for the three speeds analyzed, except for the mix at 20°C and 30 mm/min. At 10°C, asphalt binder contents between 5.0% and 5.5% exhibit higher values of FI and CRI. That is, at lower temperatures, higher asphalt binder contents are required to obtain an adequate performance of the mixture [44]. This confirms that a higher asphalt binder content contributes to increasing the capacity of the material to resist crack propagation by increasing the amount of energy dissipated in the plastic zone. On the other hand, mixtures with higher than optimum asphalt binder contents are not necessarily more resistant to cracking [45]. The above may be associated with cracking that can occur when

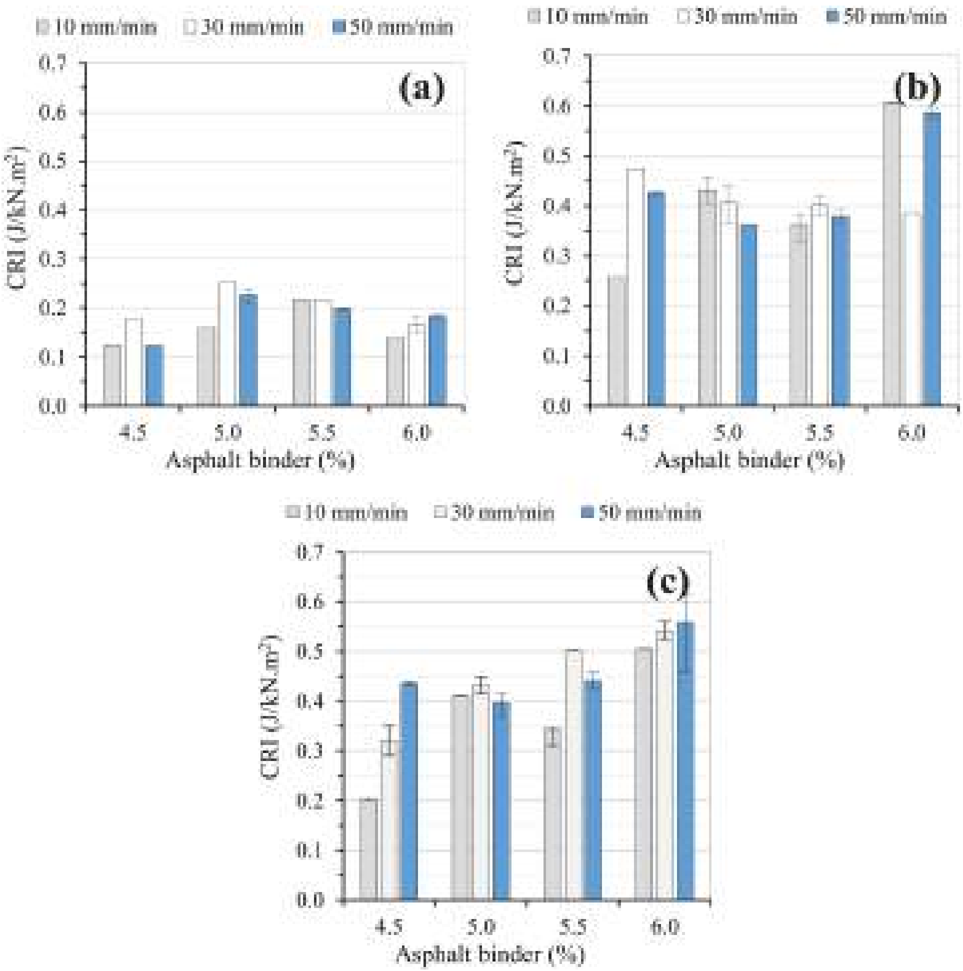

Fig. 9. CRI: a) 10 °C; b) 20 °C; c) 40 °C.

coarse aggregate particles fracture at low temperatures, whereas at elevated temperatures, the performance of the mixture depends mainly on the asphalt mastic [28]. Overall, the ANOVA results (Tables 6 and 7) show significant changes in the measured parameters with varying asphalt binder content, loading rate, and temperature, with a few exceptions.

The mixtures that developed higher P, ITS, and St, undergo greater displacement at failure and consequently higher Wf. In addition, statistical analyses indicate that the behavior of the mix depends on the asphalt binder content. The evolution of ITS with Wf for different asphalt binder contents is shown in Fig. 11. In summary, the best performance in the ITS test was achieved with 5.5% asphalt. Additionally, the results indicate that there is less scatter and better correlation (R^2 =0.93) between ITS and Wf at this asphalt binder content. Asphalt binder contents of 4.5% and 6.0% indicate higher dispersion and, consequently lower coefficient of determination values (R^2 =0.40 and R^2 =0.35, respectively). This behavior may be associated with the fact that for the ITS at a temperature of 10 °C, Wf is lower than the trend, which may be related to the brittle behavior of the samples. In other words, the results of the load versus displacement curves indicate that, at 10 °C, there is a rapid drop in the maximum load, resulting in a smaller area under the curve. This behavior is contrary to that evidenced at 40ºC, which indicates that there is a higher Wf than that of the trend. This can be explained by the ductile behavior of the mixture (curve with greater opening). Similar behavior also occurs for 5% asphalt, although with less dispersion (R^2 =0.70). Therefore, it is possible to show that the asphalt binder content of 5.5% adequately correlates the ITS behavior with the Wf at temperatures between 10 and 40 °C.

3.3. Resilient modulus tests

The results of the RM tests for each asphalt binder content are shown in Fig. 12. The results are consistent with those widely evidenced in the reference literature. [46] since the RM increases with decreasing temperature and increasing loading frequency. For all frequencies and temperatures, the maximum RM value was presented for the mixture with 5.5% asphalt, which is consistent with the results obtained from the ITS test and the Wf analyses.

Since the way of load application and temperatures are the same in the ITS and RM tests, it is possible to obtain a correlation including Wf. Mixtures with higher ITS and Wf values lead to higher stiffness and consequently higher RM. Eq. (2) shows a parameter (K) that empirically relates ITS, Wf, and loading frequency (f). This parameter in turn can be linearly correlated with the RM (Fig. 13) for the various asphalt binder contents. There are higher coefficients of determination (R^2) for higher asphalt binder contents, mainly for OAC=5.5%. In other words, there is a positive and direct proportional relationship between RM and K (Eq. (3)). Where a and b are regression parameters that depend on the asphalt binder content.

$$K = \frac{ITS \cdot Wf}{10 + 0.43f} \tag{2}$$

$$RM = aK + b \tag{3}$$

3.4. Mixture performance analysis

To analyze the performance of the mixes with different asphalt

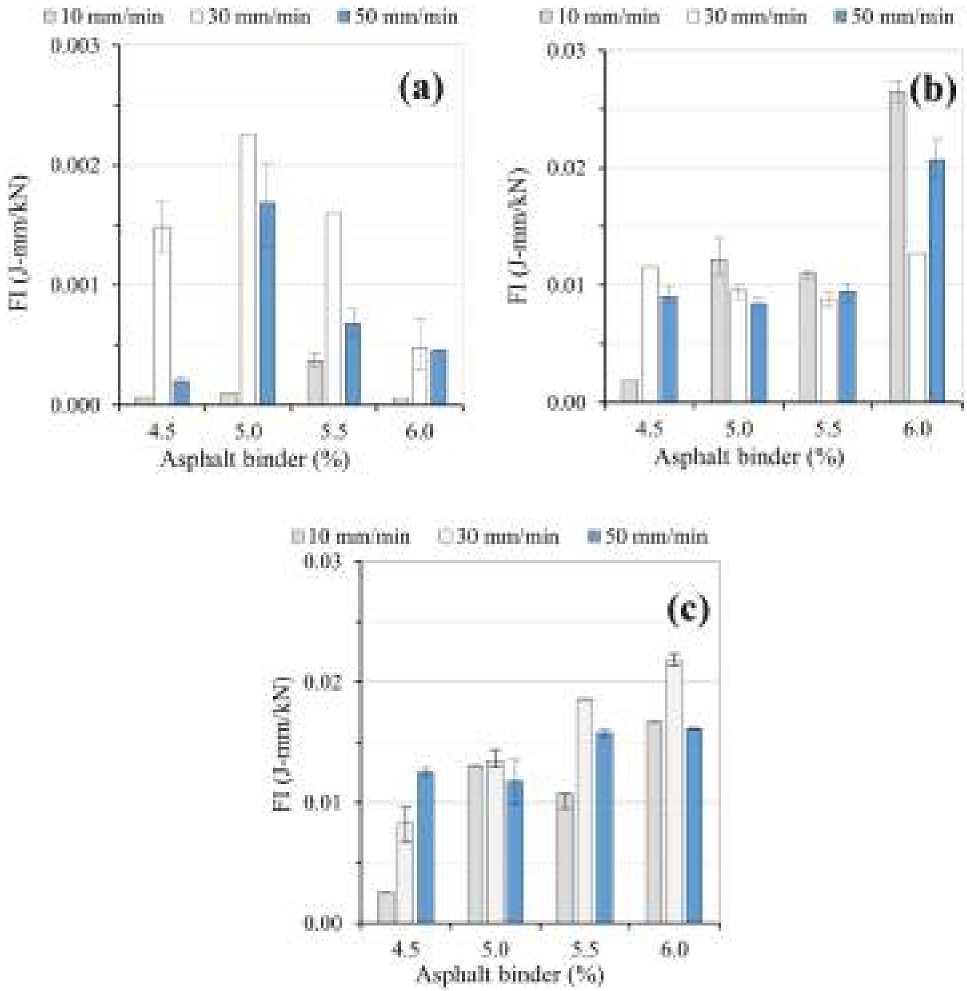

Fig. 10. FI: a) 10 °C; b) 20 °C; c) 40 °C.

Table 6
ANOVA for the significate of work of fracture, CRI and FI at different asphalt binder content.

	Loading Rate (mm/min)	Work fracture			CRI			FI		
		F	p-value	Sig	F	p-value	Sig	F	p-value	Sig
Temperature 10°C	10	126.1	4.49×10^{-7}	Yes	11.1	3.18×10^{-3}	Yes	94.9	1.36×10^{-6}	Yes
	30	7.1	1.20×10^{-2}	Yes	0.5	7.09×10^{-1}	No	70.4	4.31×10^{-6}	Yes
	50	130.4	3.94×10^{-7}	Yes	73.8	3.59×10^{-6}	Yes	37.0	4.90×10^{-5}	Yes
Temperature 20°C	10	89.3	1.72×10^{-6}	Yes	21.3	3.58×10^{-4}	Yes	11.4	2.92×10^{-3}	Yes
	30	27.3	1.49×10^{-4}	Yes	12.1	2.45×10^{-3}	Yes	48.4	1.80×10^{-5}	Yes
	50	106.1	8.80×10^{-7}	Yes	300.7	1.46×10^{-8}	Yes	84.1	2.16×10^{-6}	Yes
Temperature 40°C	10	387.5	5.35×10^{-9}	Yes	9.2	5.66×10^{-3}	Yes	289.4	1.70×10^{-8}	Yes
	30	76.1	3.20×10^{-6}	Yes	15.0	1.21×10^{-3}	Yes	137.4	3.21×10^{-7}	Yes
	50	15.0	1.20×10^{-3}	Yes	5.2	2.84×10^{-2}	Yes	3.4	7.27×10^{-2}	No

binder content, temperature, and loading rate, each of the parameters was rated on a scale of 1–4, where 1 and 4 correspond to the highest and lowest values, respectively. That is, if P, ITS, St, Gf, Gf, CRI, and FI tend to 1, the HMA presents a better performance in terms of fracture. The opposite case occurs with the m parameter, where the best performance of the mixture is referred to when it tends to 4. The qualifications of the results and parameters obtained from the ITS test are shown in Table 8. It is observed that the worst performance is obtained when an asphalt binder content of 4.5% is used. This is mainly because this content is insufficient to cover the aggregates and adhere to them. In general, it is evident that at 10°C (lower temperature), the best responses were obtained for asphalt binder contents of 5.0% and 5.5% (except at 10°C and 10 mm/min), while at temperatures of 20°C and 40°C, the best responses were obtained for contents of 5.5% and 6.0% (except CRI at 20°C and 30 mm/min).

The qualification of the RM test results are shown in Table 9. 1 and 4 correspond to the highest and lowest values, respectively. It is observed that the highest stiffness values under cyclic loading or RM are presented when the HMA employs 5.5% asphalt, regardless of temperature and loading frequency.

Based on the qualification of the ITS and RM tests, it is observed: i) The best performance is obtained with 5.5% asphalt; ii) at intermediate and high temperatures the mixes require higher asphalt binder contents than that obtained by the Marshall method.

Table 7
ANOVA for the significate of work of fracture, CRI and FI at different Loading Rate.

	Loading Rate (mm/min)	Work fracture			CRI			FI		
		F	p-value	Sig	F	p-value	Sig	F	p-value	Sig
Temperature 10°C	4.5	19.61	2.3×10^{-3}	Yes	7.52	2.3×10^{-2}	Yes	122.63	1.4×10^{-5}	Yes
	5.0	131.54	1.1×10^{-5}	Yes	77.69	5.1×10^{-5}	Yes	90.35	3.3×10^{-5}	Yes
	5.5	5.71	4.1×10^{-2}	Yes	23.75	1.4×10^{-3}	Yes	226.89	2.2×10^{-6}	Yes
	6.0	140.98	9.0×10^{-6}	Yes	11.75	8.4×10^{-3}	Yes	10.83	1.0×10^{-2}	Yes
Temperature 20°C	4.5	289.14	1.1×10^{-6}	Yes	85.15	3.9×10^{-5}	Yes	356.14	5.8×10^{-7}	Yes
	5.0	148.73	7.7×10^{-6}	Yes	45.68	2.3×10^{-4}	Yes	10.18	1.2×10^{-2}	Yes
	5.5	113.76	1.7×10^{-5}	Yes	46.40	2.2×10^{-4}	Yes	7.98	2.0×10^{-2}	Yes
	6.0	112.63	1.7×10^{-5}	Yes	176.58	4.7×10^{-6}	Yes	3.18	1.1×10^{-1}	No
Temperature 40°C	4.5	460.29	2.7×10^{-7}	Yes	103.22	2.3×10^{-5}	Yes	98.57	2.6×10^{-5}	Yes
	5.0	174.57	4.8×10^{-6}	Yes	454.21	2.8×10^{-7}	Yes	1.59	2.8×10^{-1}	No
	5.5	486.25	2.3×10^{-7}	Yes	108.52	1.9×10^{-5}	Yes	91.54	3.2×10^{-5}	Yes
	6.0	56.91	1.3×10^{-4}	Yes	10.55	1.1×10^{-2}	Yes	1.38	3.2×10^{-1}	No

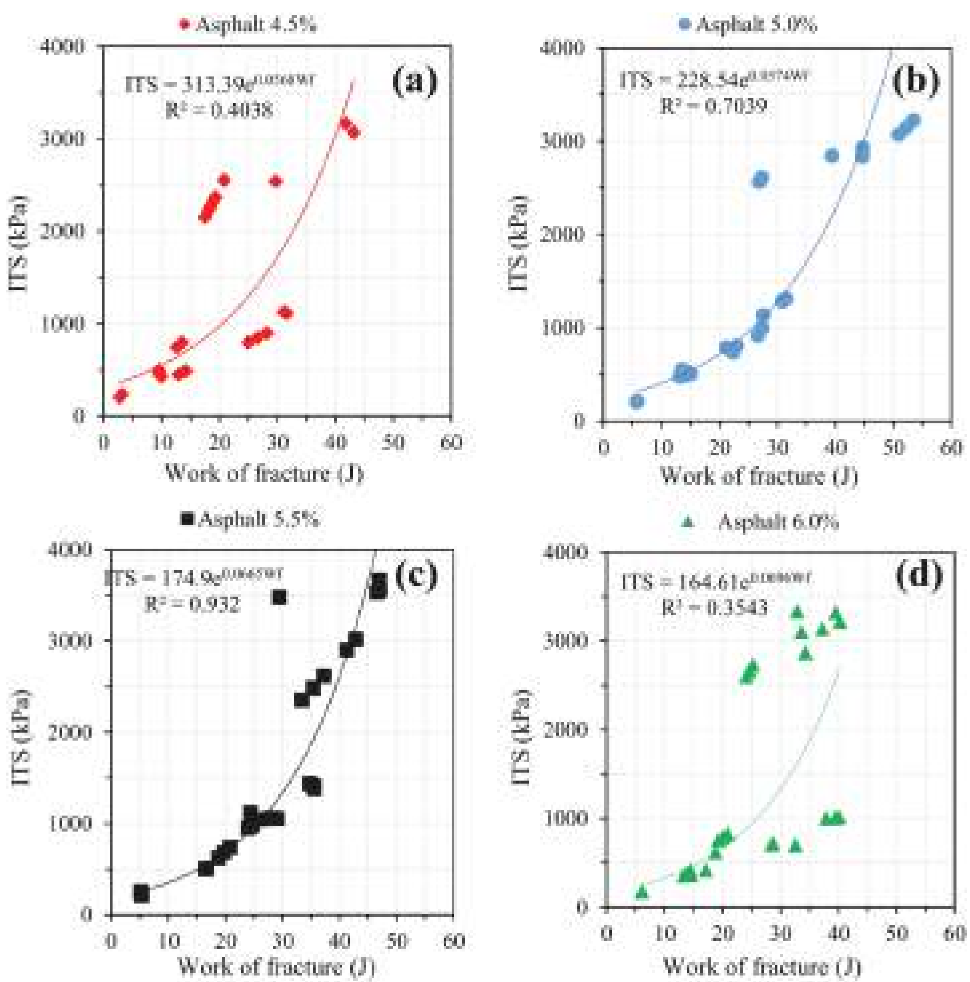

Fig. 11. Evolution of ITS with the Wf for asphalt binder content of: a) 4.5%; b) 5.0%; c) 5.5% y d) 6.0%.

3.5. Results variability analysis

ANOVA statistical analyses were used to determine if there are significant variations between the results as a function of asphalt binder content and loading rate. However, to determine the variability of the results, the CV (%) was analyzed. The CV obtained for the results of the Marshall test, ITS (fracture parameters), and RM are presented in Tables 10, 11, and 12, respectively. Concerning the Marshall test, it is observed that the CVs are lower than 10%. The highest values are presented for the highest asphalt binder content (6%), where there is greater deformability of the material. On the other hand, in the ITS tests and fracture parameters, the highest CV values are presented mainly for high temperatures and high asphalt binder contents. On the other hand, in the RM test, there is no clear trend of CV as a function of temperature or loading frequency.

4. Conclusions

Based on the results obtained, it is concluded:

- The Marshall test reported an OAC of 5.0%. The ITS strength analysis led to the conclusion that the adequate OAC corresponds to 5.5%

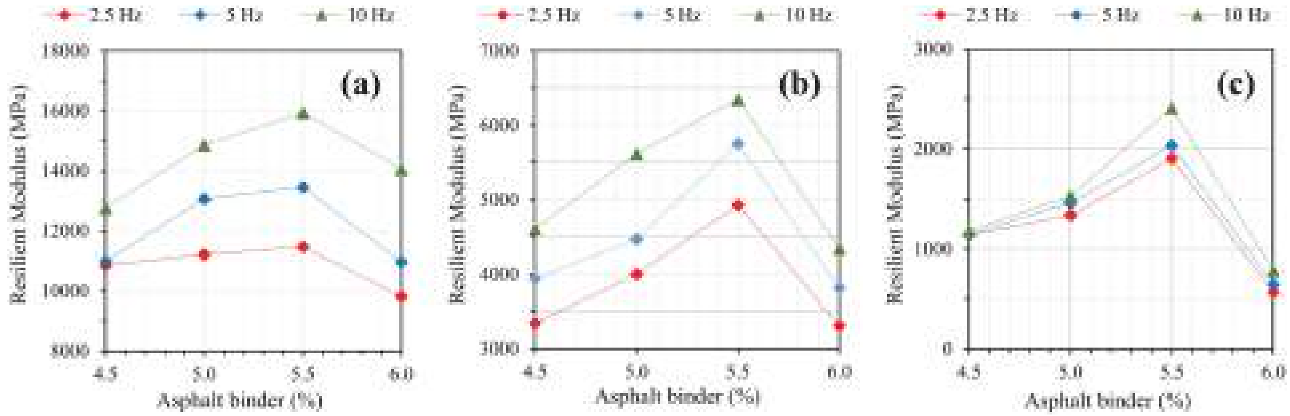

Fig. 12. Results of resilient modulus tests: a) 10 °C; b) 20 °C; c) 40 °C.

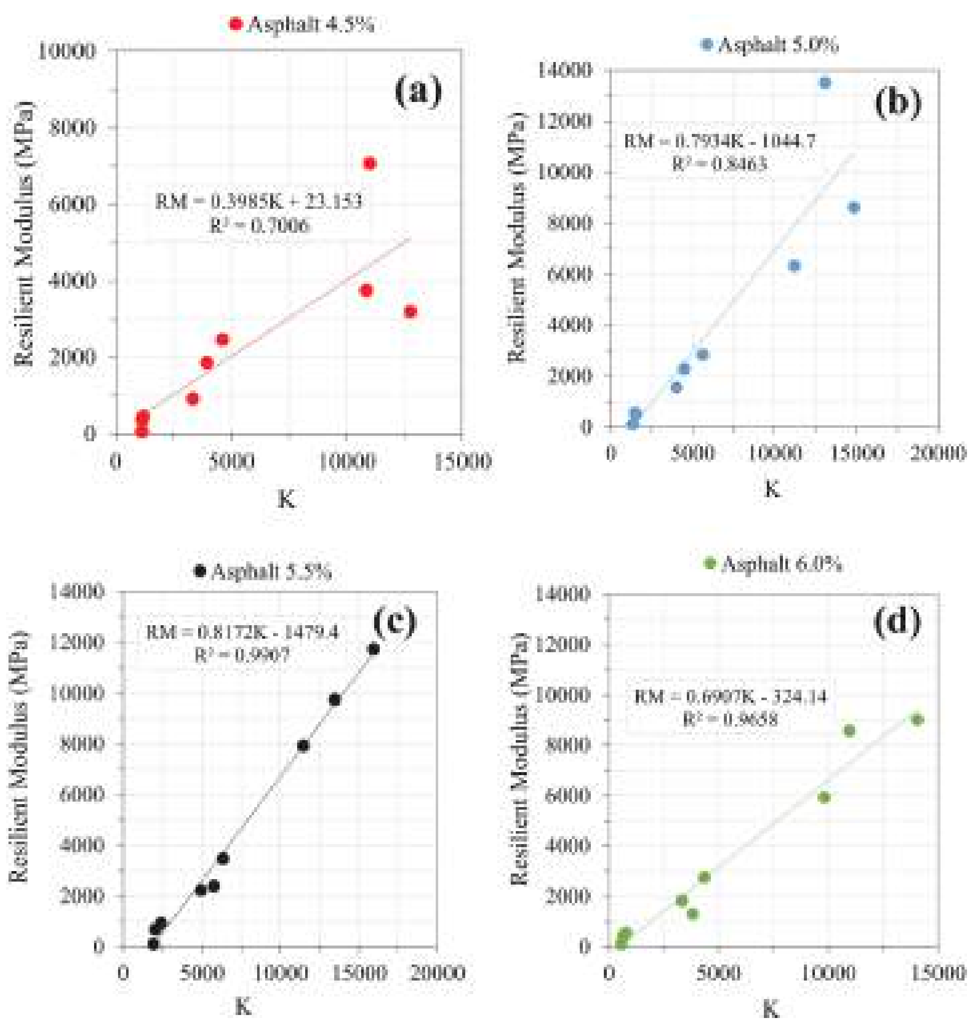

Fig. 13. Evolution of the RM with parameter K for asphalt binder content: a) 4.5%; b) 5.0%; c) 5.5% y d) 6.0%.

since it presents higher magnitudes of P, ITS, St, Wf, and lower values of m for most of the temperature and load application rates contemplated in this study. The difference between the OAC obtained by the Marshall and ITS methods is 0.5%. That is when designing HMA by the Marshall method, the OAC could be insufficient to adequately cover the aggregates and improve the fracture properties (crack resistance, adhesion with aggregates, and cohesion of the mixture, among others).

- Temperature, speed of load application in the ITS test, and frequency of loading in the RM test influence the HMA behavior. The highest and lowest values of P, ITS, St, Wf, and RM were reported at 10 °C to 40 °C, respectively. An opposite trend is reported for the m parameter. These values were higher with increasing load application rate in the ITS test and frequency in the RM test.
- According to the results of the fracture parameters, increasing the OAC obtained in the Marshall test by +0.5% leads to greater

Table 8
Qualification of asphalt mixtures depending on the ITS parameters.

Parameters	Loading rate (mm/min)	10 °C				20 °C				40 °C			
		4.5	5.0	5.5	6.0	4.5	5.0	5.5	6.0	4.5	5.0	5.5	6.0
P (kN)	10	4	2	3	1	3	2	1	4	2	3	1	4
	30	4	1	2	3	3	2	1	4	3	2	1	4
	50	4	3	1	2	3	2	1	4	3	2	1	4
ITS (kPa)	10	4	2	3	1	3	2	1	4	2	3	1	4
	30	4	1	2	3	3	2	1	4	3	2	1	4
	50	4	3	1	2	3	2	1	4	3	2	1	4
St (kN/mm)	10	4	2	3	1	4	2	1	3	3	2	1	4
	30	4	2	3	1	4	2	1	3	4	1	2	3
	50	4	3	1	2	3	2	1	4	4	2	1	3
m (kN/mm)	10	2	3	4	1	1	3	2	4	1	3	2	4
	30	3	4	2	1	3	2	1	4	1	2	3	4
	50	1	4	3	2	3	2	1	4	3	2	1	4
Wf (kJ/m2)	10	4	2	1	3	4	3	2	1	4	2	3	1
	30	4	1	2	3	3	2	1	4	4	2	1	3
	50	4	2	1	3	3	4	2	1	4	3	1	2
Gf (J)	10	4	2	1	3	4	3	2	1	4	2	3	1
	30	4	1	2	3	3	2	1	4	4	2	1	3
	50	4	2	1	3	3	4	2	1	4	3	1	2
CRI (J/(kN-m2).	10	4	2	1	3	4	2	3	1	4	2	3	1
	30	3	1	2	4	1	2	3	4	4	3	2	1
	50	4	1	2	3	2	4	3	1	3	4	2	1
FI (J-mm/kN)	10	3	2	1	4	4	2	3	1	4	2	3	1
	30	3	1	2	4	2	3	4	1	4	3	2	1
	50	4	1	2	3	3	4	2	1	3	4	2	1

Table 9
Qualification of asphalt mixtures depending on the RM test.

Parameters	Frequency	10 °C				20 °C				40 °C			
		4.5	5.0	5.5	6.0	4.5	5.0	5.5	6.0	4.5	5.0	5.5	6.0
RM	2.5	3	2	1	4	3	2	1	4	4	2	1	3
	5.0	3	2	1	4	3	2	1	4	3	2	1	4
	10.0	3	2	1	4	3	2	1	4	3	2	1	4

Table 10
Marshall test results CV.

Parameter	Asphalt binder content (%)			
	4.5	5.0	5.5	6.0
E (kN)	1.2	0.7	1.0	2.9
F (mm)	4.9	6.4	5.6	9.1
Va (%)	3.4	3.5	5.3	7.8
VMA (%)	1.2	1.0	1.3	1.5
VFA (%)	1.4	1.3	1.6	1.8

resistance to cracking and possibly greater durability of the pavement. This was also consistent with the results obtained with the RM test.

- The analyses carried out, based on the experimental campaign and the parameters obtained, provide a new approach in the selection of the OAC for HMAs concerning the traditional design using the Marshall methodology. Therefore, it is proposed to combine the Marshall method and the results obtained from ITS and RM tests to determine the OAC of HMAs that will be exposed to temperate climates.
- Taking the laboratory ambient temperature (20°C) as a reference, the results of this study allow us to conclude that P, ITS, and St increase approximately 1.5 times when the temperature is lowered to 10°C, but decrease by half when the temperature is increased to 40°C. This could have a direct impact on the structural performance of the pavement. Further studies should be carried out to predict the effect of temperature variation and the traffic loading spectrum to calibrate the mechanistic-empirical equations for pavement design.
- There is a direct and positive proportionality correlation between RM and fracture parameters that are grouped in a K variable established in this study. Establishing the physical significance of this variable requires a broader experimental phase where different types of asphalts, aggregates, and granulometry, among others, are analyzed.
- ANOVA statistical analyses indicate that the response of the material to monotonic loading action and fracture parameters depend significantly on the asphalt binder content and the rate of load application for each temperature. The same conclusion applies to the case of mixture performance in RM tests. On the other hand, the CV values can be considered valid and representative from the point of view of replicability.

The results obtained in this study are representative only of the HMA analyzed. Further studies are recommended to understand the mechanical behavior of other mixtures including different granulometries and asphalt materials. Also, the use of alternative materials (e.g., reclaimed asphalt pavement - RAP, recycled concrete aggregates - RCA, construction and demolition wastes - CDW, etc.) could be included. Finally, comparison with other tests that directly measure the resistance to permanent deformation and fatigue of the mixes (evaluating aspects associated with durability such as resistance to moisture damage, aging, and abrasive wear, among others) is recommended, as well as larger scale laboratory and field tests to obtain more definitive conclusions.

CRediT authorship contribution statement

Juan Gabriel Bastidas-Martínez: Writing – review & editing, Writing – original draft, Methodology, Formal analysis, Data curation,

Table 11
CV test ITS and fracture parameters.

Parameter	Temperature (°C)	Loading Rate 10 (mm/min)				Loading Rate 30 (mm/min)				Loading Rate 50 (mm/min)			
		Asphalt binder content (%)				Asphalt binder content (%)				Asphalt binder content (%)			
		4.5	5.0	5.5	6.0	4.5	5.0	5.5	6.0	4.5	5.0	5.5	6.0
P max (kN)	10	6.7	1.3	7.5	3.4	11.5	3.4	9.8	10.6	7.1	1.8	2.0	4.0
	20	3.5	4.5	8.9	1.2	6.0	10.7	0.6	4.0	0.7	1.3	2.2	1.1
	40	7.3	2.4	11.6	0.0	7.1	3.3	0.7	10.3	4.2	3.4	12.9	28.6
ITS (kPa)	10	6.7	1.3	7.5	3.4	11.5	3.4	9.8	10.6	7.1	1.8	2.0	4.0
	20	3.5	4.5	8.9	1.2	6.0	10.7	0.6	4.0	0.7	1.3	2.2	1.1
	40	7.3	2.4	11.6	0.0	7.1	3.3	0.7	10.3	4.2	3.4	12.9	28.6
St (kN/mm)	10	6.7	1.3	1.0	3.4	6.5	3.4	6.5	1.0	7.1	3.4	2.1	2.3
	20	3.5	10.2	12.8	26.6	6.0	9.3	14.2	19.5	7.4	7.1	13.7	18.0
	40	13.6	2.3	9.3	0.0	13.4	9.7	0.7	12.2	2.8	3.5	19.2	32.9
Wf (J)	10	6.7	1.3	7.5	3.4	1.6	3.4	2.8	2.9	7.1	7.1	0.5	3.6
	20	3.5	4.3	1.5	8.1	6.0	1.7	5.3	4.0	0.7	1.5	1.2	2.8
	40	7.3	2.4	2.2	0.0	2.2	6.9	0.7	5.8	4.3	3.4	7.4	12.7
Gf (kJ/m2)	10	4.8	0.9	5.3	2.4	1.6	2.4	2.8	2.1	7.1	7.1	0.5	3.6
	20	3.5	4.3	1.5	8.1	6.0	1.7	5.3	4.0	0.7	1.5	1.2	2.8
	40	7.3	2.4	2.2	0.0	2.2	6.9	0.7	5.8	4.3	3.4	7.4	12.7
CRI	10	3.3	2.1	6.3	4.1	10.3	3.1	23.4	13.5	0.1	6.6	1.8	3.0
(J/(kN-m2).	20	7.1	6.3	8.4	9.1	1.1	8.9	5.0	1.1	1.4	0.2	3.3	2.1
	40	3.1	4.1	13.8	0.1	9.2	3.7	2.1	4.6	0.6	6.4	5.5	15.6
FI	10	2.3	1.1	14.4	3.1	20.1	2.1	1.1	45.6	13.1	20.8	17.0	2.5
(J-mm/kN)	20	4.1	13.6	3.3	5.0	0.1	6.6	7.1	1.1	8.9	8.6	9.5	12.3
	40	0.8	4.4	16.0	0.0	17.9	6.1	2.1	3.2	2.5	15.8	2.1	41.7

Table 12
RM test results CV.

Parameter	Frequency 2.5 Hz				Frequency 5.0 Hz				Frequency 10 Hz			
	Asphalt binder content (%)				Asphalt binder content (%)				Asphalt binder content (%)			
	4.5	5.0	5.5	6.0	4.5	5.0	5.5	6.0	4.5	5.0	5.5	6.0
Temperature 10°C	4.0	2.2	6.7	4.5	8.3	5.6	3.7	5.8	7.9	7.2	6.0	14.6
Temperature 20°C	38.1	24.0	1.2	32.4	36.5	24.8	7.8	13.6	34.8	6.1	10.9	25.0
Temperature 40°C	31.3	6.2	9.6	9.5	30.0	3.7	5.8	41.8	26.6	21.4	6.7	7.4

Conceptualization. **Hugo Alexander Rondón-Quintana:** Conceptualization, Methodology, Supervision, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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