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Laboratory and statistical evaluation of the microstructural characteristics of Sand Asphalt Mortar

Avaliação laboratorial e estatística das características microestruturais da Argamassa Areia Asfalto

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ABSTRACT
One of the problems encountered in asphalt pavements is fatigue damage, which is related to the type of aggregate, the rheology of the asphalt binder and its chemical composition, resulting in the emergence of microcracks and, subsequently, macrocracks. Therefore, this phenomenon needs to be studied from the asphalt binder scale to the complete asphalt mixture. In this sense, this paper focuses on the asphalt binder scale with the objective of investigating the microstructural characteristics of cylindrical samples of Sand Asphalt Mortar (SAM). These samples can be made up of standard sand and any type of asphalt binder. The use of SAM can contribute to obtaining laboratory rheological results representing the real binder film thickness that exists in an asphalt mixture. In this study, SAM samples composed of a polymer binder without aging were compacted with a manual press, resulting in cylindrical specimens with 40 mm of height and 12.5 mm of diameter. Its microstructure was investigated by determining the air voids (AV) and the binder film thickness (FT), considering different binder contents: 6%, 8% and 10% by weight. The results indicated that the samples made with 6% binder present homogeneity in terms of microstructural parameters, and represents more realistically the binder film thickness that will exist in a complete asphalt mixture, can be used in oscillatory tests carried out on the dynamic shear rheometer, and contribute to studies on the asphalt binders.

RESUMO
Um dos problemas encontrados nos pavimentos asfálticos é o dano por fadiga, que está relacionado com o tipo de agregado, a reologia do ligante asfáltico e sua composição química, resultando no surgimento de microtrincas e, posteriormente, em macrotrincas. Portanto, esse fenômeno precisa ser estudado desde a escala do ligante asfáltico até a mistura asfáltica completa. Nesse sentido, o presente artigo foca no estudo da escala do ligante asfáltico com o objetivo de investigar as características microestruturais de amostras cilíndricas de Sand Asphalt Mortar (SAM). Essas amostras podem ser constituídas de uma areia padrão e qualquer tipo de ligante asfáltico. O uso da SAM pode contribuir com a obtenção de parâmetros reológicos laboratoriais representando melhor a real espessura do filme de ligante que existe numa mistura asfáltica. Neste estudo, amostras de SAM constituídas de asfalto modificado por polímero sem envelhecimento foram compactadas com o auxílio de uma prensa manual, resultando em corpos de prova cilíndricos com 40 mm de altura e 12,5 mm de diâmetro. Os resultados obtidos indicaram que as amostras com 6% de ligante apresentam homogeneidade em termos de parâmetros microestruturais e representam de forma mais realista a espessura do filme de ligante que irá existir numa mistura asfáltica completa, podendo ser utilizada em ensaios oscilatórios realizados no reômetro de cisalhamento dinâmico e contribuir com os estudos sobre a fadiga de ligantes asfálticos.

1. INTRODUCTION

High traffic demand and the age of pavement can accelerate the deterioration process of the highways, increasing the cost of maintenance and rehabilitation (Chen, Jiang and Bahia, 2022). The distresses in Brazilian pavement are fatigue and permanent deformation, being fatigue damage a complex phenomenon.

Among the constituents that influence the asphalt mixture performance are the different characteristics of the shape and mineralogy of the aggregates, particle size distribution, content and type of binder. Therefore, an alternative for comparing and selecting materials based on the binder performance is to use tests and define binder parameters that can contribute to rheological analysis (Bernucci et al., 2022).

In order to guarantee the quality of pavements, it is essential to evaluate the binder's rheological parameters in different situations during their useful life. Kim, Little and Lytton (2003, 2004) and Kim, Little and Song (2003) proposed the use of cylindrical samples with Sand Asphalt Mortar (SAM) to carry out rheological tests using the Dynamic Shear Rheometer (DSR) and characterize the fatigue damage. The authors pointed out two advantages of the SAM: the repeatability of the rheological parameters evaluated in oscillatory DSR tests explained by the standardization of Ottawa sand, and the consequent elimination of the mineralogical and granulometric influence of the aggregates presented in Fine Aggregate Matrices (FAMs). Due to its constitution, SAM makes possible to simulate the real thickness of the binder film (FT) existing in asphalt concretes (AC), in the order of 2 μm in the fine fraction as indicated by Elseifi et al. (2008), when compared to the binder thickness samples used in the DSR parallel plate tests, between 1 and 2 mm (ASTM, 2015; 2020; AASHTO, 2019; 2020; DNIT, 2022a). Even with the adaptation of the rheological tests using the SAM, the parameters can be obtained quickly and economically.

Regarding the determination of the FT involving the AC aggregates, Radovskiy (2003) defines this parameter as the ratio between the volume of asphalt not absorbed by the aggregate particles and the aggregate surface area, which is considered uniform for all particles and is an important characteristic for guaranteeing the durability of asphalt mixtures. This author proposed an empirical equation, as shown in Equation 1:

$$FT_t = \frac{V_b}{SA \cdot W_{agg}} = \frac{P_{be}}{SA \cdot G_b} \cdot 10^{-5} \tag{1}$$

Where: FT_t = theoretical binder film thickness (m), V_b = effective binder volume (m^3), SA =specific aggregate surface (m^2/kg), W_{agg} = aggregate mass (kg), P_{be} = effective asphalt binder content in the mix by weight (%), G_b = asphalt binder density.

Elseifi et al. (2008) determined FT values using image analysis techniques obtained through Scanning Electron Microscope (SEM). They observed that in AC the FT value can be as low as 2 μm, considering only the binder, and over 100 μm for the mastic, highlighting the importance of laboratory studies to find this parameter at different scales.

Kommidi, Kim and Rezende (2020) and Rezende et al. (2021) carried out Linear Amplitude Sweep (LAS) and Time Sweep (TS) tests in the DSR with SAM samples with asphalt binders under different aging conditions, and conventional binder samples positioned between parallel plates 2 mm thick. SAM samples captured the micro-crack and macro-crack phases more clearly than the conventional method. The authors also highlighted the repeatability of the results obtained and pointed out that SAM can be considered suitable for assessing the properties and damage characteristics.

Similarly, Rezende et al. (2021) statistically observed the variability and sensitivity of FT values obtained using Laser Scanning Microscopy (LSM). In this study, SAM samples were made with different heights, 30, 40 and 50 mm, with different binder contents, 6%, 8% and 10%, and tested in the DSR at different temperatures, 10°C, 25°C and 45°C. The authors noted that the FT values were between 10 and 70 μm, and the test temperature had a significant impact on the results, where fatigue resistance decreased with FT increasing.

However, the small number of studies using SAM to evaluate the role of the binder in cracking on a microscopic scale is not enough to determine a protocol for producing, dosing and compacting these samples, ensuring that reliable and realistic FT values are obtained. Issues such as the homogeneity and repeatability of the microstructural characteristics of SAM samples need to be better evaluated, so that the rheological parameters obtained can be validated.

In this context, this paper aims to investigate the microstructural characteristics in terms of air voids (AV) and FT of SAM samples composed of Ottawa sand and polymer-modified asphalt, and to contribute to the safe use of SAM in rheology studies of asphalt binders and to a better understanding of asphalt binder fatigue phenomena.

2. MATERIALS AND METHODS

2.1. Materials

The materials used (Table 1) were Ottawa sand and polymer modified binder, identified as AMP 60-85 by Brazilian standards and as PG 70-XX in Superpave classification, to produce samples with three different binder contents by weight, 6%, 8% and 10%. The definition of these contents was based on the study presented by Kim, Little and Lytton (2003), in which was indicated that samples with 8% of binder would result in FT values around 10 μm, within the literature recommended range and expected as realistic value (Elseifi et al., 2008). In addition, these binder contents were used in previous studies (Kommidi, Kim and Rezende, 2020; Rezende et al., 2021; Gonçalves, 2021) which were also used in the analysis.

Table 1: Characteristics of the materials used in the mixtures.

OTTAWA SAND				
Grading, percent passing sieve (mm)	**Passing percentage criterion (%) [1]**	**Measured passing percentage (%)**		
1.18	100	100		
0.6	96 to 100	98		
0.425	60 to 75	61		
0.3	16 to 30	16		
0.15	0 to 4	1		
BINDER				
Tests	**Standard**	**Unit**	**Standard Criteria [2]**	**AMP 60/85**
Density Relative at 25°C	NBR 6296 (ABNT, 2012)	g/cm³	-	1.006
Softening point, min	ME 131 (DNIT, 2010a)	°C	60	67.5
Penetration index (100 g, 5 s, 25°C)	ME 155 (DNIT, 2010b)	0.1 mm	40-70	59
Brookfield Viscosity 135°C, max	NBR 15184 (ABNT, 2021)	cP	3000	753
Brookfield Viscosity 150°C, max	NBR 15184 (ABNT, 2021)	cP	2000	383
Brookfield Viscosity 177°C, max	NBR 15184 (ABNT, 2021)	cP	1000	148
Performance Grade	T 315 (AASHTO, 2021)	°C	-	70-XX

[1] according to ASTM C778 (ASTM, 2021). [2] according to ANP (2022).

The Ottawa sand was used in the composition of SAM mixtures, with granulometric characteristics (Table 1) and density, 2.652 g/cm³, supplied by the manufacturer, with ASTM C778 (ASTM, 2021). The particle size ranges were checked using a laser granulometer, confirming that the material complied with the established standard. The selected binder, without aging, was subjected to laboratory characterization tests according to Brazilian specifications (ANP, 2022) and also classified according to the maximum performance grade, PG (AASHTO, 2021).

2.2. Methods

The procedures used to obtain the SAM samples and analyze AV and FT consisted of six steps summarized in Figure 1. It should be noted that details of the equipment and method used can be found in patent application number BR 10 2023 025702-0 (Curado et al., 2023).

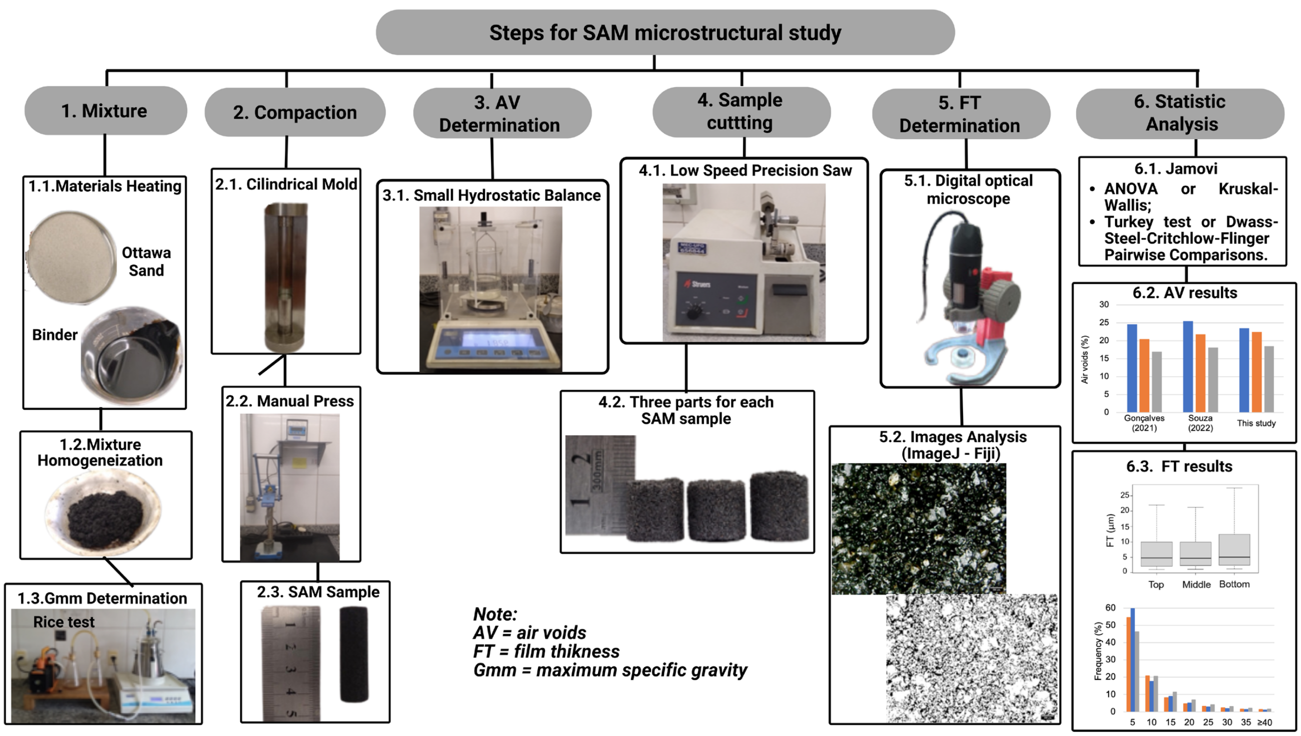


Figure 1. Steps to obtain and analyze SAM samples.

In this study, it is essential to define the sample preparation process in order to guarantee the repeatability of the data obtained. The samples were prepared as proposed by Kommidi, Kim and Rezende (2020), following the adaptation suggested by Gonçalves (2021). To prepare the mixtures, the materials and utensils used were heated in an oven at 135°C for 20 minutes. After this, the materials were homogenized until the binder completely covered the grains. Thus, the maximum measured relative density values were determined (G_{mm}), for subsequent determination of the volumetric variation with the Rice Test (DNIT, 2020). As proposed by Curado et al. (2021), 500 g of mixture was used to carry out the test, optimizing the laboratory study in view of the small amount of material needed to compact the specimens, approximately 10 g. In this way, the appropriate weight of aggregate and binder was used, depending on the binder content used, plus a margin of 25 g for possible losses during the process, resulting 525 g of mixture (e.g. 31.5 g of binder and 493.5 g of sand for 6% binder).

The compaction stage began with heating the amount of asphalt mixture needed to produce a specimen and the tools used at 150°C for 30 minutes. Afterwards, the heated mixture was inserted into the mold and this set was again kept in the oven (150°C for 15 minutes). Next, the compaction mold filled with the mixture went into the manual press specially developed for the study (Curado et al., 2023). The specimens were then compacted with five stages of loading, applying a constant load of 90.8 kgf for 20 seconds, with loading interspersed at the top and bottom, ensuring a uniform distribution of compaction energy.

After compaction, the mold was subjected to a cooling process at a temperature of -5°C for 15 minutes, facilitating the process of extracting. In this case, cooling the sample prevents any material from adhering to the mold and deforming the sample. In this study, eleven samples measuring 40 mm in height and 12.5 mm in diameter were produced for each binder content, in order to collect enough data to carry out statistical analysis of the parameters obtained. Thus, it was necessary to carry out laboratory tests and specific analyses to determine the microstructural characteristics of the samples in terms of AV and FT, in order to verify if these characteristics are homogeneous and suitable for use in rheological studies in the DSR in a more realistic, rapid and economical way.

To determine the AV values, it was necessary to carry out the hydrostatic balance test (DNIT, 2022b). As these are relatively small samples, it is recommended to use a balance with an accuracy of 0.001 g and a hydrostatic weighing device adapted to these dimensions. The tests were carried out in two situations: with the whole specimens and with the cut parts of the specimens.

Next, the specimens were cut into three equal parts approximately with 13 mm high, using a low-speed precision saw (maximum 400 rpm) with a 0.7 mm thick diamond blade. In order to achieve a more precise cutting process, the specimens were stored in the refrigerator at a temperature of -5°C for 30 minutes to reduce heating and, consequently, the change in viscosity of the binder and loss of material. In addition, chilled water was also circulated in constant contact with the cutting disc to minimize the impact of the process.

To determine FT, quadrants images of the specimens were taken using a Digital Optical Microscope (DM), with a magnification range of up to 1,600 times and brightness control. It was necessary to adjust the distance between the DM and the surface analyzed using a 3D support in order to obtain better quality images. Using this equipment is more economical than investing in high-resolution microscopes such as SEM. Six specimens were randomly selected with their respective sections cut out, resulting in eighteen different samples to be evaluated for each binder content. In order to find the best configuration to represent each section, isolated images were taken of the cross-section quadrants, with a resolution of 3264 × 2448 pixels.

The images obtained using DM were evaluated using ImageJ (Fiji) software. To analyze them, the procedure presented by Vieira et al. (2021) was used in an adapted way, enabling the extraction and processing of quantitative data that resulted in the FT values. Unlike Vieira et al. (2021) who performed digital processing of images generated by grouping the images collected in the SEM with the “Stitching” command, in this study the images collected with the DM were analyzed individually. The “Fill Holes” command was not applied in this study, because when it was used it considered most of the image as a single aggregate, without the presence of asphalt binder, which is an incoherent result.

The software generated several measurements of the horizontal distances between the aggregates filled with asphalt binder, ensuring the accuracy of the analysis. These distance measurements result in the line segments that represent FT. The data made it possible to obtain the parameters needed to determine FT, using Equation 2 as Ferreira and Rasband (2012) and Vieira et al. (2021). The FT value for an aggregate was calculated as half of each distance measured as indicated by Rezende et al. (2021).

$$FT = Feret * cos\left(180 - \theta\right) \tag{2}$$

Where: FT = film thickness parallel to the X axis (pixel), θ = angle formed between the line segment and the X axis of the image, Feret Angle (degree).

To obtain FT measurements, the samples were cut into 99 cross-sections which were used to capture images. As this is a meticulous, detailed and time-consuming process, six specimens were selected from each location, for each binder content, and four images were analyzed for each specimen. This resulted in 216 images that were subjected to automatic collection of the line segments to minimize operator interference. Only four images could not be analyzed automatically and were disregarded. In order to provide an independent evaluation and minimize the risk of imminent overlap, the analysis was carried out in pairs of diametrically opposed quadrants. Thus, the odd-numbered quadrants were called sample group 1 (G1) and the even-numbered quadrants were the group 2 (G2). The faces of the top and bottom samples analyzed were those that were in contact with the mold during the compaction process, thus obtaining clearer images in the DM. The middle faces were chosen according to the best quality and visibility of the images acquired.

All data was statistically analyzed to assess the homogeneity of AV and FT values obtained for whole and cut SAM samples, as well as the influence of the binder content on the microstructural characteristics. Jamovi software was used to analyze the data and perform statistical tests. In this study, the significance level (α) adopted was 5%, as this is a usual value in statistical analysis (Osmari et al., 2020; Rezende et al., 2021).

The results were evaluated using the Analysis of Variance (ANOVA) test to check for significant differences. The variations in FT and AV values as a function of binder content were compared in the middle, top and bottom samples to check the homogeneity and normality of the variances. The data distribution defined the type of ANOVA applied, parametric with normality or non-parametric Kruskal-Wallis without normality. The Tukey "*Post-Hoc*" test or Dwass-Steel-Critchlow-Flinger Pairwise Comparisons were used in situations with significant differences to identify between which groups the differences occurred according to the ANOVA.

3. RESULTS AND DISCUSSIONS

This section presents the results obtained for the characteristics, AV and FT, of the whole and cut specimens, and their statistical analyses for the three binder contents studied. The G_{mm} and AV results obtained for SAM with AMP 60-85E were compared with studies already carried out using the same SAM sample production protocol, but made up of other types of petroleum asphalt cement (CAP): Gonçalves (2021) with CAP 50-70, and Souza (2022) with CAP 30-45. For FT values, this comparison was not possible because the procedures used in this study were different from the others.

Table 2 summarizes the average values obtained for G_{mm}, AV and number of samples evaluated for each binder content. It can be seen that as the binder content increases, the density increases and the AV value decreases as expected. Even the highest coefficient of variation (CV) values were for AV in cut samples (< 3.34%), it still indicating little dispersion between the samples, as they are less than 15%. These findings point that AV values presented good repeatability and can be used as technological control of during the SAM production.

Table 2: Summaries of the values obtained for G_{mm} and AV of the SAM-type samples with AMP 60-85E.

	Gmm			**AV (%) Entire specimen (before cutting)**				**AV (%) Individual pieces (cut specimens)**			
Binder content (%)	**Average**	**SD**	**CV (%)**	**Number of samples**	**Average**	**SD**	**CV (%)**	**Number of samples**	**Average**	**SD**	**CV (%)**
6	2.43	0.003	0.12	11	25.24	0.39	1.53	33	24.00	0.006	2.49
8	2.36	0.007	0.30	11	21.68	0.25	1.17	33	23.01	0.008	3.34
10	2.30	0.002	0.08	11	18.06	0.28	1.53	33	18.54	0.006	3.09

Note: Gmm = maximum specific gravity; AV = air voids; SD = standard deviation; CV = coefficient of variation.

The average values obtained for G_{mm}, AV and FT were compared with previous studies, where Figure 2a shows the same trend in the behavior of the G_{mm}. Figure 2b shows the AV values obtained for whole samples, and regardless of the type of binder used, the values obtained were similar. The AV values could not be compared with international papers data because there are no publications using SAM samples compacted with this equipment and, consequently, with the methodology used in this paper.

It can be seen that the average AV values obtained were greater than 17%. Studies of FAM samples produced in the Superpave Gyratory Compactor (SGC) or in static presses without control of the applied load have aimed to produce cylindrical samples with an AV close to 4%, as this is a value commonly used in AC dosing (Fonseca et al., 2019; Vieira et al., 2021). However, this is not the logic of the dosage process for SAM samples, as it is not to simulate AC dosage, but to be homogeneous in terms of AV and to represent more realistically FT values. Thus, it is not mandatory to use low AV values.

In this study, the AV results were evaluated using ANOVA (Table 3) between the sample groups of the three different binder contents, showing different behaviors with significant differences (p-value < 0.05). When compared with other studies, it can be seen that the samples studied by Gonçalves (2021) and Souza (2022) showed similar results. This finding points to the influence of the binder content on the AV values.

For the AV analysis of the cut specimens, 99 samples resulting from the cutting process were used. Looking at the AV values (Figures 2c, 2d and 2e), it is possible to notice a trend in which the AV values at the top and bottom are slightly lower than those in the middle. These results were similar with others values already observed for SAM samples without polymer (Gonçalves, 2021; Souza, 2022), showing that this behavior is independent of the type of binder and the operator during the compaction process.

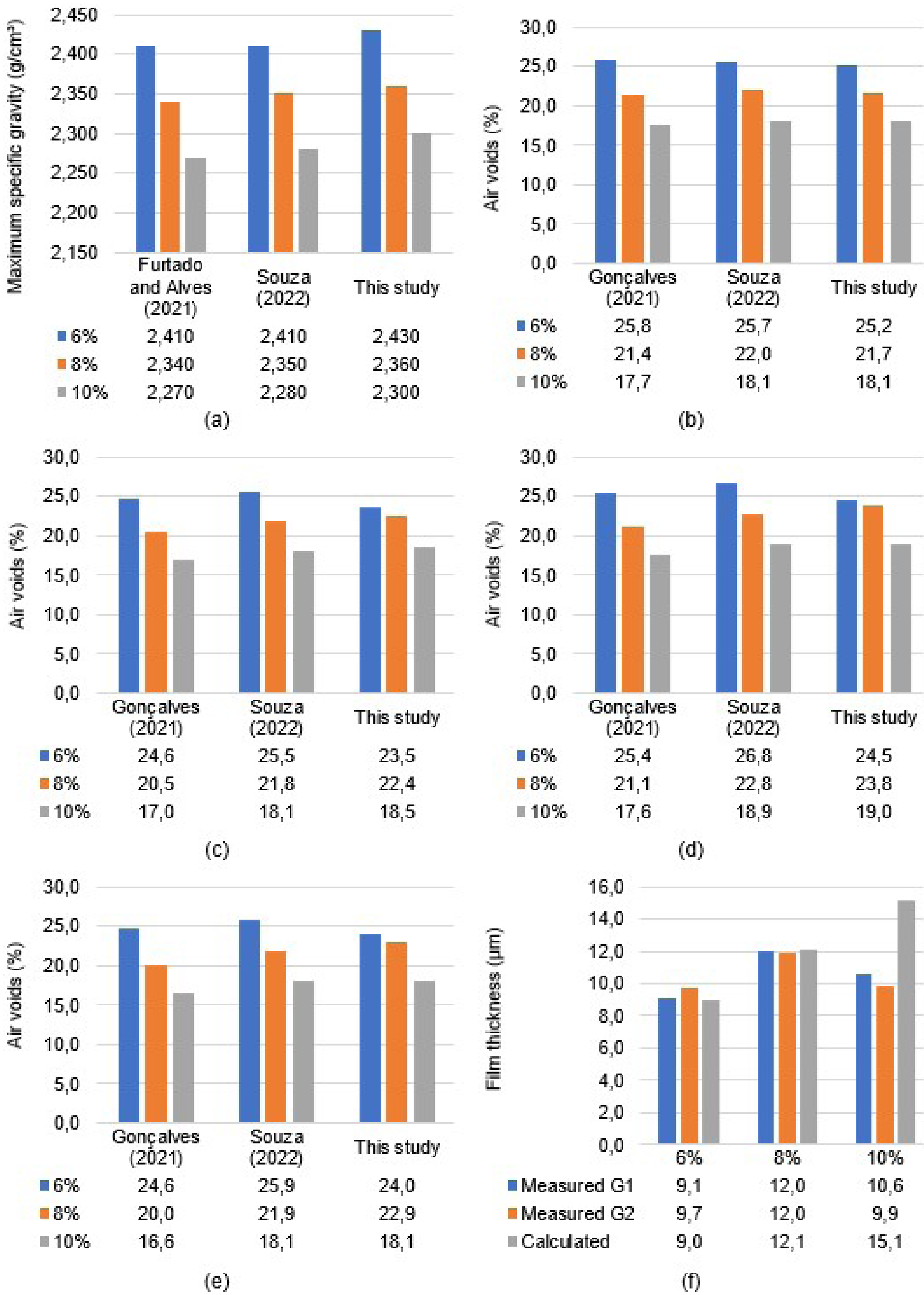


Figure 2. Average values obtained for: (a) G_{mm}; (b) AV entire specimen; (c) AV cut samples-top; (d) AV cut samples- middle; (e) AV cut samples-bottom; (f) FT.

Table 3: Statistical result for AV (p-value).

ENTIRE SAM SPECIMENS			
Binder content (%)	**6**	**8**	**10**
6	–	0.0356	0.0717
	–	< 0.001	< 0.001
8		–	0.0361
		–	< 0.001

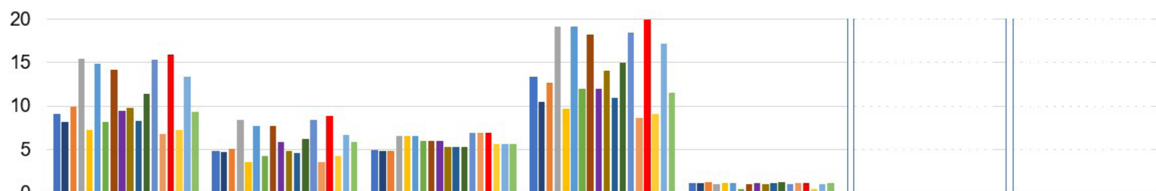
CUT SAM SPECIMENS						
***p*-value**	**6%**		**8%**		**10%**	
	Middle	**Bottom**	**Middle**	**Bottom**	**Middle**	**Bottom**
Top	< 0.001	0.064	< 0.001	0.088	0.052	0.149
Middle	–	0.022	–	< 0.001		< 0.001

ANOVA statistical analyses were carried out on the cut samples and indicated that there were significant differences in the AV values. Therefore, another evaluation was necessary using the Tukey test to identify the location of these differences (Table 3). The top/bottom ratio showed no significant differences (p-value < 0.05) in the contents studied. Compared to the results obtained by Gonçalves (2021) and Souza (2022), a trend of behavior can be identified for the 6% and 8% contents with the greatest significant differences between the ratios with the middle of the samples, indicating that this behavior is independent of the binder used. The only ratio involving the middle of the sample that did not show significant differences was for the middle/base ratio at 10%. There is a possibility that the data heterogeneity in the distribution of AV may be influenced by the cutting or compacting process. Therefore, considering that in rheological tests rupture tends to occur in the middle of the SAM sample as pointed out by Rezende et al. (2021), priority should be given to find the AV values in the sample middle instead of a single AV value for the entire specimen. In this case, the results of the fatigue parameters evaluated will be linked to the appropriate AV value.

For the FT values, the microscopic images showed whether or not the sand grains were completely covered and the influence of the cutting process on the quality of the images, where flatter surfaces contribute to better analysis. Figure 2f shows the average FT values obtained from the images analyzed and determined empirically, considering the density of the materials. It can be seen that the measured and calculated FT values were close to each other, especially for the 6% and 8% contents. It was not possible to observe a correlation trend between FT values and binder content. This can be explained by the complexity of the stages involved in obtaining FT measurements and by various factors that still need to be better investigated, but which were not the subject of this study, such as:

- Huang, Lv and Xiao (2016) indicated that the use of polymers improves the binder cohesion and can change its microstructural characteristics. Therefore, it is possible that SAM with polymer binder have increased cohesion depending on the binder content, which could interfere in the FT determinations;
- Although the use of a 3D support for the DM enabled better quality images to be captured, the images obtained from the samples with the highest binder content (10%) still showed glare interference compared to the lower contents (6% and 8%), which could result in erroneous FT values. In this sense, it is recommended to use binder contents lower than 8%.

There is still no standardized method for measuring FT values and, even guaranteeing accurate data, this analysis may not be conclusive, since the scale measured is too small (μm). However, this does not preclude the use of SAM in rheology studies since, regardless of the asphalt binder content, the average FT values obtained were less than 20 μm and can be considered realistic. The FT values were evaluated according to the three different cut sections (Figures 3). However, they were not compared with the results obtained by Gonçalves (2021) and Souza (2022), due to a difference in the method used to obtain FT. The results obtained do not allow the authors to establish a pattern of behavior that relates the position of the cut specimen to the FT value for each mixture analyzed. It can be seen that the average FT values varied between 6.78 and 15.85 μm, and the median values were between 3.57 and 8.89 μm, close to the values obtained by Vieira et al. (2021) in FAM samples, between 2.06 and 5.16 μm, and by Kommidi, Kim and Rezende (2020) in SAM samples, from 10.5 to 97.5 μm. Thus, the FT values obtained in this study and in the literature varying from 2 and 100 μm, and agree with the values indicated by Elseifi et al. (2008). These findings indicate that SAM samples can represent realistic values of FT and probably will contribute to better understand the rheological behavior of asphalt binders.



	Mean FT values (mm)	FT median values (mm)	Mean of median FT values	SD	Minimum FT (mm)	Maximum FT (mm)	CV (%)
G1-6%-Top	9,08	4,81	4,88	13,35	1,05	327,59	147,06
G1-6%-Middle	8,18	4,74	4,88	10,53	1,17	175,99	128,84
G1-6%-Bottom	9,95	5,08	4,88	12,71	1,23	177,96	127,71
G1-8%-Top	15,50	8,43	6,61	19,12	1,04	241,51	123,35
G1-8%-Middle	7,26	3,61	6,61	9,63	1,14	173,16	132,62
G1-8%-Bottom	14,91	7,78	6,61	19,16	1,10	302,03	128,56
G1-10%-Top	8,23	4,24	5,94	12,01	0,50	231,50	145,98
G1-10%-Middle	14,23	7,70	5,94	18,20	1,09	272,52	127,87
G1-10%-Bottom	9,45	5,87	5,94	11,98	1,17	190,47	126,70
G2-6%-Top	9,75	4,85	5,26	14,06	1,05	248,10	144,15
G2-6%Middle	8,26	4,66	5,26	10,94	1,17	182,72	132,49
G2-6%Bottom	11,43	6,27	5,26	14,95	1,23	233,50	130,83
G2-8%Top	15,31	8,43	6,96	18,48	1,04	224,56	120,66
G2-8%Middle	6,78	3,57	6,96	8,65	1,14	144,49	127,66
G2-8%Bottom	15,85	8,89	6,96	19,94	1,10	409,65	125,80
G2-10%Top	7,22	4,22	5,60	9,06	0,50	163,07	125,46
G2-10%Middle	13,37	6,67	5,60	17,22	1,09	244,37	128,74
G2-10%Bottom	9,35	5,90	5,60	11,49	1,18	211,08	122,91

Figure 3. FT variations in different locations.

The variations in FT for each binder content and specimen position were also analyzed, identifying the average FT, standard deviation (SD), minimum FT, maximum FT and CV. According to Ferreira (2015), some authors consider CV values greater than 30% to be a high dispersion for experimental data, as observed in Figure 3. Thus, different from AV, as FT is a complex microstructural characteristic to measure, it can be used as a secondary parameter to confirm the quality of SAM sample and AV can be chose as a primary parameter to control the sample repeatability.

When evaluating the data dispersion by using statistical boxplots (Figure 4), the boxplots with 200 µm, regardless of the asphalt binder content. By enlarging the scale of the graphs, it is possible to identify the values considered to be upper limits for each content and location analyzed, illustrating the trends in behavior for the same binder content analyzed in the different quadrants. Samples with 8% and 10% presented greater variations in the upper limits depending on the locations analyzed. This suggests that contents above 8% could be considered high and could jeopardize the homogeneity of the SAM samples in terms of FT.

Figure 5 shows the histograms obtained for FT values. These images showed a representative number of measurements with less than 20 µm. For the three different asphalt binder contents, a significant part of the data analyzed presented FT values between 1 and 5 µm. When assessing the trend of data dispersion by calculating the 1st, 2nd and 3rd quartiles, which correspond to 25%, 50% and 75% of the data respectively, Figure 6 shows that 75% of FT values were lower than 20.02 µm. The presence of outliers in all sample groups, contents and locations analyzed, together with the histograms presented, indicated that the data distribution is asymmetrical and is best represented by the median. It is known that the arithmetic mean is influenced by extreme values and should be used in situations where the data distribution is approximately symmetrical (Morettin and Bussab, 2010). In this way, the median FT values of the specimens were statistically analyzed.

Evaluating the homogeneity along the height of the specimen (top/middle, top/base, middle/base) for each binder content individually (Table 4), it can be seen that there are no significant differences between FT values for the 6% binder content in any of the ratios in the two sample groups. For the 8% and 10% contents, there were no significant differences between top and bottom, but the ratios evaluated involving the middle of the sample showed differences. These results coincide with the analysis obtained in terms of AV, where the middle of the SAM samples showed differences in relation to the top and bottom. This behavior is expected, given that for the same asphalt binder content, the AV and FT values will be correlated.

These results suggest that binder contents above 8% in SAM can compromise the homogeneity of FT values throughout their height. Contents of 6% and 8% showed close FT values, while 10% showed greater variability. Thus, the determination of AV may be a simpler, faster, more economical and reliable option for quality control in the production of SAM samples. On the other hand, using FT as a dosage or production control criterion can be more complex, although it is important for ensuring the representativeness of the sample to be used in the laboratory test. Determining AV in the laboratory combined with estimating FT using parametric equations derived from laboratory studies would be the ideal way forward. However, this would require further studies using the methodology presented in this paper, exploring different types of binder and ageing conditions.

In any case, the FT values obtained are consistent with those in the literature and may better represent the asphalt film that surrounds the aggregates in complete asphalt mixtures, making it possible to exclude the influence of fines in the mastic during the analysis of rheological parameters.

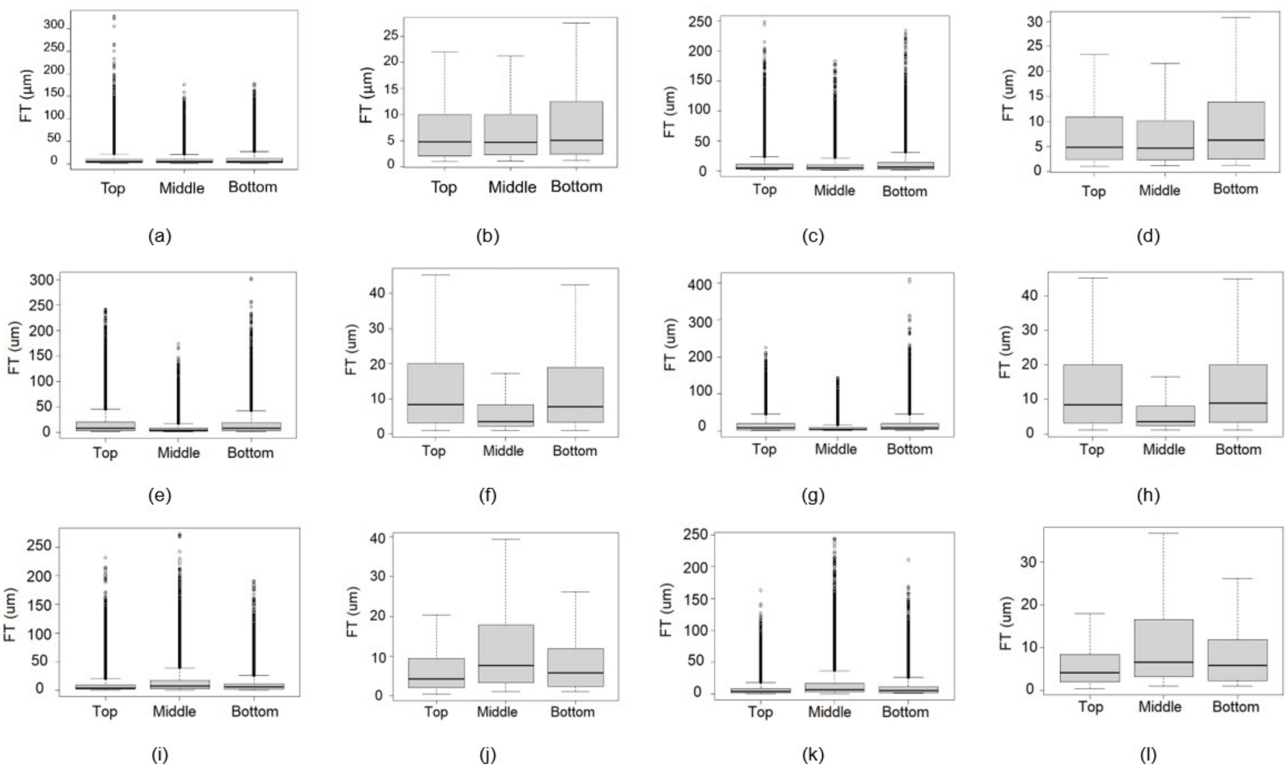


Figure 4. FT distribution: a) with outliers for G1-6%; b) with zoom for G1-6%; c) with outliers for G2-6%; d) with zoom for G2-6%; e) with outliers for G1-8%; f) with zoom for G1-8%; g) with outliers for G2-8%; h) with zoom for G2-8%; i) with outliers for G1-10%; j) with zoom for G1-10%; k) with outliers for G2-10%; l) with zoom for G2-10%.

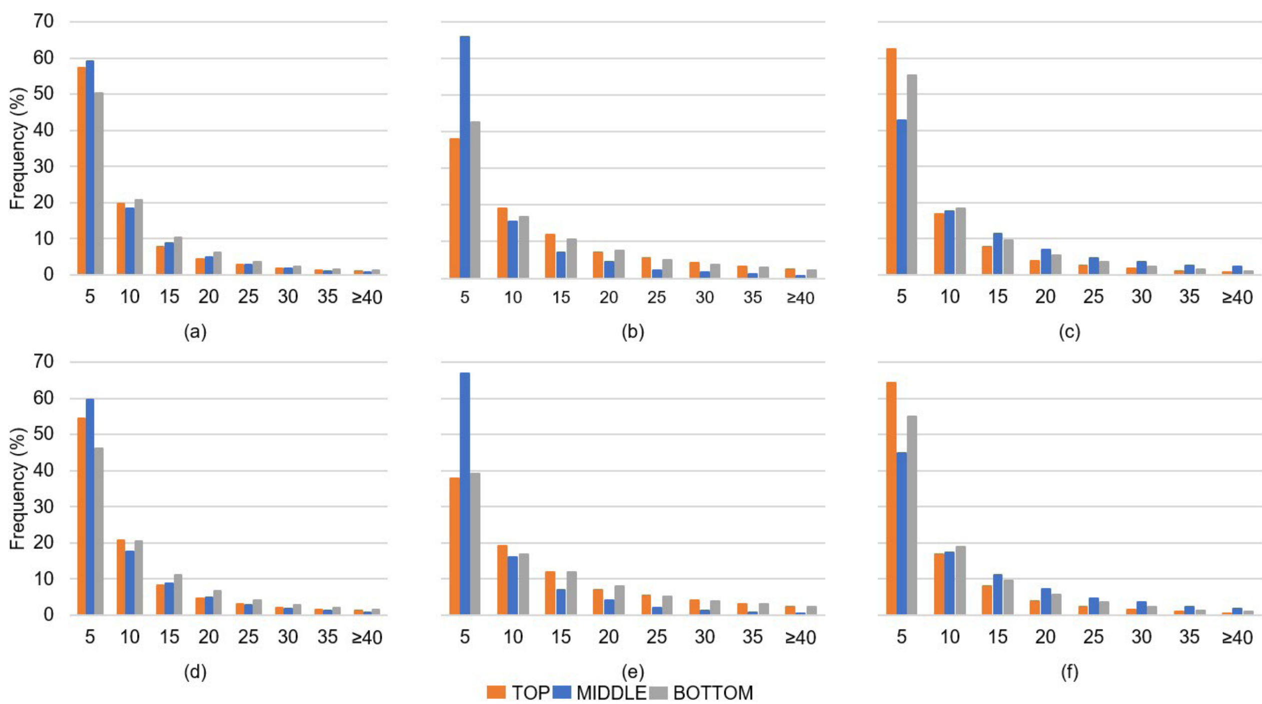
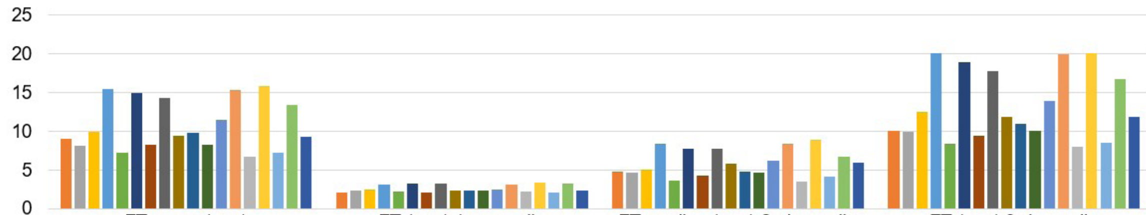


Figure 5. Distribution of FT through histograms at different binder contents: a) 6% binder - G1; b) 8% binder - G1; c) 10% binder - G1; d) 6% binder - G2; e) 8% binder - G2; f) 10% binder - G2.



	FT mean (mm)	FT (mm) 1st quartile	FT median (mm) 2nd quartile	FT (mm) 3rd quartile
G1-6%-Top	9,08	2,09	4,81	10,04
G1-6%-Middle	8,18	2,37	4,66	9,95
G1-6%-Bottom	9,95	2,50	5,08	12,49
G1-8%-Top	15,50	3,16	8,43	20,02
G1-8%-Middle	7,26	2,29	3,61	8,33
G1-8%-Bottom	14,91	3,31	7,78	18,89
G1-10%-Top	8,23	2,10	4,24	9,46
G1-10%-Middle	14,23	3,30	7,70	17,78
G1-10%-Bottom	9,45	2,38	5,87	11,90
G2-6%-Top	9,75	2,40	4,85	10,92
G2-6%Middle	8,26	2,33	4,66	10,10
G2-6%Bottom	11,43	2,51	6,27	13,88
G2-8%Top	15,31	3,15	8,43	19,95
G2-8%Middle	6,78	2,29	3,57	8,06
G2-8%Bottom	15,85	3,33	8,89	20,00
G2-10%Top	7,22	2,10	4,22	8,50
G2-10%Middle	13,37	3,30	6,67	16,67
G2-10%Bottom	9,35	2,38	5,90	11,90

Figure 6. FT variations.

Table 4: Statistical result for FT of SAM samples.

Binder content (%)	*p*-value	G1		G2	
		Middle	Bottom	Middle	Bottom
6	Top	0.992	0.663	0.870	0.257
	Middle	-	0.408	-	0.087
8	Top	<0.001	0.927	<0.001	0.767
	Middle	-	<0.001	-	<0.001
10	Top	0.002	0.094	0.003	0.032
	Middle	-	0.030	-	0.062

4. CONCLUSIONS

This study verified that the microstructural characteristics analyzed for SAM samples composed of Ottawa sand and polymer-modified asphalt presented homogeneity in terms of air voids (AV) and represented film thickness (FT) values realistically. These findings point to the safe use of SAM samples in rheological tests as an option to a better understanding of asphalt binder fatigue phenomena.

There was a behavioral trend in terms of AV values and binder content for the whole SAM specimen. The results obtained for cut samples indicate that the average values of AV in the middle were a little higher than the values obtained for the top and bottom. Possible hypotheses include the fact that the compaction may not be homogeneous throughout the specimen, as well as the possibility of interference from the cutting process. In terms of FT values, it was not possible to observe a definite behavioral trend when related to binder content. Although, samples with 6% binder can be considered homogeneous in terms of FT with realistic values less than 20 µm.

More studies are needed to help obtain results with less variability in the parameters analyzed, studying the SAM microstructural characteristics with binder contents less than 6%, using the same method for producing and compacting, with the same method for obtaining and analyzing the images to determine FT, as well as obtaining these parameters with advanced techniques that do not damage the samples. Finally, this paper contributed to part of this process and can help to discuss the standardization of the SAM samples to be used in DSR tests, produced with different types and aging conditions of asphalt binders. Thus, the use of SAM samples with many types of asphalt binders with different aging conditions in rheological studies will contribute to the advancement of multiscale evaluation, as well as providing a deeper understanding of the influence of the asphalt binder on the asphalt concrete fatigue phenomenon.

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