



ANALYSIS OF PORE GEOMETRY IN THE COMPACTED FINE AGGREGATE MATRIX BY X-RAY MICROTOMOGRAPHY.

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1. Introduction

Fine aggregate matrix (FAM) may be used as a predictor of the behavior of asphalt mixtures [1]. The FAM consists primarily of asphalt binder, fine aggregates, fillers and air-filled pores as a major constituent of mixtures [2]. The pores in any structure may or may not be in contact with the material surface. The pores connected to the material surface are usually named as open pores and the pores that are not connected to the surface are called closed pores, even though they are internally connected within the material [3]. Pore size is not precisely defined since its shape is generally irregular and extremely variable. The quantitative pore size description is performed by models of a small number of shapes, such as cylinders, prisms, cavities and windows, slits or spheres [4]. There is great difficulty in determining and analyzing pore geometry in a structure due to its variability [5]. The particles or pores sphericity has several definitions, among them, as the relation between the diameter of the circle with an area equal to the projection of the particle and the diameter of the smallest circle circumscribed to the particle, with values ranging from 0 to 1 [6]. Briefly, these values range from 0.45 for elongated particles to 0.97 that represent very spherical particles. Research indicated that the volumetric characteristics of FAM specimens must be carefully determined to avoid undesired variability in the mechanical behavior of replicates of the same mixture. This also indicates that a certain level of variation can be tolerated around the air void content adopted on the design of FAM without affecting the stiffness of the material [7]. Traditional porosity assessment methods such as mercury porosimetry and Archimedes method are well documented and provide good results for many materials. However, they are only global average results for the entire sample [8]. In this context, X-ray microtomography (μ CT.) stands out because, besides porosity information, pore distribution and a series of other parameters related to the internal structure of the object can be evaluated [9]. The study main goal is to evaluate the open and closed pores distribution on FAM specimens extracted from different locations of SGC samples. Statistical analyses are also performed to evaluate the porosity variation and pore number with compactation within the SGC FAM samples. Furthermore, it was possible to determine the open and the closed pores geometric shape of the FAM samples.

2. Methodology

The samples were prepared at the Jacques de Medina Geotechnical and Pavement Laboratory located in the Department of Civil Engineering of the Coordination of the Graduate Program in Engineering at the Federal University of Rio de Janeiro (COPPE / UFRJ). The method used to design the FAM samples in this study was recently proposed by Amelian *et al.*[10]. This method defines the FAM volumetric characteristics based on the key parameters of the HMA (Hot Mix Asphaltic) mix design. The binder content determined for FAM was 8.49%, while the binder content for the corresponding HMA was 4.90%. The HMA and FAM gradation is shown in Figure 1. The samples FAM1, FAM2 and FAM3 were compacted in the superpave gyratory compactor (SGC) and resulted in the following densities values 2.26 g/cm³, 2.35 g/cm³ and 2.44 g/cm³, respectively, the most compacted samples presented higher density values. Then they were sawn to a final length of approximately 48 mm. Nine specimens from each FAM1,

FAM2 and FAM3 were extracted with a cylindrical drill connected to a drilling machine with a diameter of 12 mm in different positions.

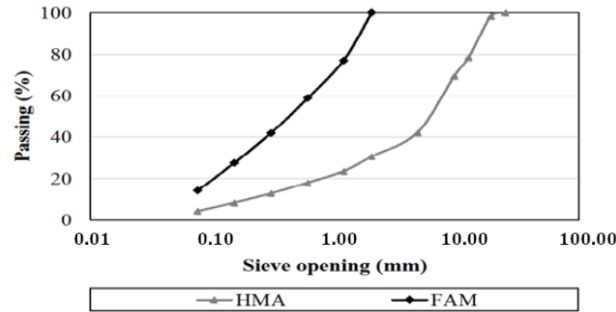


Figure 1 – Aggregate Gradation for HMA and FAM.

The image analysis of the specimens of FAMs was performed on the SkyScan model 1173 bench microtomograph (μ CT.), installed at X-rays Applied Laboratory on State University of Londrina. It is important to highlight that a threshold value (TH) that separates the two objects included in the ROI must be chosen for the quantification process. There is no default method for determining the value of TH. In this study a global TH value of 30, out of a range from 0 to 255, was selected after a rigorous analysis performed by experienced professionals. After segmentation by global thresholding, the porosity values of the 9 specimens of each FAM1, FAM2 and FAM3 were determined by μ CT. at 8 μ m resolution. This spatial resolution was chosen because it is the best system resolution to map the small pores of the specimen. Sph is a measure of how spherical of a 3D object and it was determined through the ratio between the surface area of a sphere (with the same volume as the given particle) to the surface area of the particle [11]. Equation 1 was employed to determined the sphericity.

$$Sph = \frac{\sqrt[3]{\pi \cdot (6 \cdot V)^2}}{A} \quad (1)$$

where V and A are the object volume and surface area respectively

3. Results and Discussions

The quantities closed porosity (PF), number of closed pores (NPF), total volume of closed pores (VPF) and total area of closed pores (APF) are the means of the nine specimens of FM1, FM2 and FM3 obtained from μ CT. These values are useful in determining the mean pore diameter by 3D analysis. These values are shown in table 1.

Table 1 - Quantities for determining the sphericity of closed pores.

FAM	PF (%)	NPF	VPF (mm ³)	APF (mm ²)
1	0.88	525954	22.83	3184.16
2	2.53	751084	66.37	7434.33
3	2.56	690181	73.78	8229.41

Thus, through the data presented in table 1 and by the equations for the volume and area of the sphere, we found the diameter of the volume (d_v), the diameter of the area (d_A) and then, by the sphericity equation (1), determined the closed pore shape for each sample. The results are shown in table 2.

Table 2 – Closed pore Sph.

FAM	d_v (μ m)	d_A (μ m)	Sph
1	43.54	43.86	0.99
2	55.32	56.20	0.97
3	58.90	61.66	0.91

Table 2 showed that the closed pores of the FAMs are spherical, since the Sph of the pores is close to 0.97 [6] in all cases analyzed. In addition, their Sph decreases as the sample is more density. Usually closed pores have a spherical shape. In the FAM 1, FAM 2 and FAM 3, the closed pores are spherical and

have average diameters of 43.54 μm , 55.32 μm and 58.90 μm , respectively. It is also observed that the compaction increased the volume of the closed pores. Another important fact is that closed porosity varies with compaction and this cannot be explained only by the increase in the number of closed pores. Thus establishing the ratio of the conversion factor for the closed pore (CF) we have

$$CF = \left(\frac{NPF_1}{NPF_2} \right) \cdot \left(\frac{r_1^3}{r_2^3} \right) \quad (2)$$

Table 3 presents the results of the conversion factor (CF) of the closed porosity of a pair of samples and compares the theoretically obtained value of the closed porosity (PFT) with the experimental value of the closed porosity (PFE) measured by μCT .

Table 3 - FCF result for closed spherical pores

Specimens	PF(%)	CF	PFT(%)	PFE(%)	σ (%)
FAM1 para FAM2	0.88	2.93	2.57	2.53	1.1
FAM1 para FAM3	0.88	3.25	2.85	2.56	7.6
FAM2 para FAM3	2.53	1.11	2.80	2.56	6.3

As the difference σ is small for all FAMs, then it is concluded that the theoretical and experimental results are similar and the closed pores can be considered spherical. Obtained from the average of the FAM specimens, the open porosity (PA), number of open pores (NPA), total open pore volume (VPA) and total open pore area (APA) are important values to determine the mean open pore diameter by this method of 3D analysis. These values are found in table 4.

Table 4 Quantities for determining the sphericity of open pores.

FAM	PA (%)	NPA	VPA (mm^3)	APA (mm^2)
1	10.81	102606	311.31	20457.08
2	4.31	166260	136.27	11654.09
3	0.88	126900	25.89	3642.18

Thus, through the data in table 4 and then by the sphericity equation (1), determined the pore shape open for each sample. The results are shown in table 5.

Table 5 – Open pore Sph.

FAM	Sph
1	0.48
2	0.56
3	0.33

Table 5 presents the open pore sphericity of the samples investigated in this study. The lower sphericity was 0.33 while the larger value was 0.56. The pores with sphericity values close to 0.45 should be considered as cylindrical pores [6]. Then, from the results, most of the pore shape is not spherical. As the geometry of the open pore should be elongated then using the equations of volume and area of the cylinder, to calculate the volume diameter (d_v), the area diameter (d_A). The results are shown in table 6.

Table 6 – Diameter and length of open pore.

FAM	d (μm)	l (μm)
1	64	943
2	50	417
3	30	288

Diameters d and lengths l correspond to the elongated cylindrical shaped pores. It is also observed that compaction decreased the volume of open pores (Table 6). So the conversion factor for the open pore (FCA) can be written as:

$$CF = \left(\frac{NPA_1}{NPA_2} \right) \cdot \left(\frac{r_1^2 \cdot l_1}{r_2^2 \cdot l_2} \right) \quad (3)$$

Table 7 shows the CF results for tube-shaped cylinders that relate the open porosity of a pair of samples and compares the theoretically obtained value of open porosity (PAT) with the experimental value of open porosity (PAE) measured by μ CT. As the theoretical and experimental results are similar, open pores can be considered cylindrical.

Table 7- FCA result for cylinders in cylinder shape.

Specimens	PA	CF	PAT	PAE	$\sigma(\%)$
FAM1 para FAM2	10.81	2.29	4.72	4.31	6.4
FAM1 para FAM3	10.81	12.05	0.90	0.88	1.6
FAM2 para FAM3	4.31	5.27	0.82	0.88	5.0

4. Conclusions

This study evaluated the distribution of open and closed pores of FAM specimens extracted from different locations of SGC compacted samples. From the results and analyses, it could be concluded that: The specimens extracted showed that the shape of the closed pores is spherical and the shape open pores is cylindrical for the SGC samples compacted with 2.26 g/cm³, 2.34 g/cm³ and 2.44 g/cm³.

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References

- [1] Aragão, F. T. S., Vargas, G. A. B., Hartmann, D.A., *et al.*, “Characterization of temperature-and rate-dependent fracture properties of fine aggregate bituminous mixtures using an integrated numerical-experimental approach”, *Engineering Fracture Mechanics*, vol 180, pp. 195 – 212 (2017).
- [2] Kim, Y. R., Little, D. N., Song, I., “Effect of mineral fillers on fatigue resistance and fundamental material characteristics: mechanistic evaluation”. *J. of the Transp. Resear. Board*, vol 1832, pp 1 – 8 (2003).
- [3] Klobes, P.; Meyer, K.; Munro, R. G., *Porosity and specific surface area measurements for solid materials*, National Institute of Standards and Technology, Washington & USA (2006).
- [4] Rouqueirol J., Avnir D., Fairbridge C. W., *et al.*, “Recommendations for the characterization of porous solids”, *Pure & Appl. Chern.*, vol. 66, n. 8, pp. 1739 – 1758, (1994).
- [5] Selley, R. C., Sonnenberg, S. A., *Geology of Petroleum*, Elsevier, London & United Kingdom (2015).
- [6] Rittenhouse, G., “A visual method of estimating two-dimensional sphericity”. *J Sedim Petrol*, vol. 13, pp. 79 – 81 (1943).
- [7] Osmari, P. H., Costa, R. F., Aragão, F. T. S., *et al.*, “Determination of volumetric characteristics of FAM mixtures using X-ray micro-computed tomography and their effects on the rheological behavior of the material”, *Transportation Research Record*, pp. 1 – 11 (2020).
- [8] Pessoa, J. R. C., Domiguez, J. S., Carvalho, G., *et al.*, “Concrete porosity determined by X-ray microtomography and image processing”, *ABPE*, vol. 14, pp. 20 – 26 (2014).
- [9] Gopalakrishnan, K., Ceylan, H., Inanc, F., “Using X-ray computed tomography to study paving materials”, *Construction materials*, vol. 160, pp. 15 – 23 (2007).
- [10] Amelian, S., Kim, Y. R., Osmari, P. H., *et al.*, “Development of a volumetric mix design approach for fine aggregate matrix (FAM) and validation with micro-CT. method. Transportation Research Board, Washington, D. C., USA, (2019).
- [11] CT.Analyser, The User’s Guide, Skyscan/Bruker micro-CT., Kartuizerweg, Kontich & Belgium (2012).