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Predicting intrinsic permeability of a double porosity cellular ceramic using X-ray micro-tomography

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The microstructure of a porous cellular ceramic exhibiting two spatial characteristic lengths was characterized to determine parameters of its pore space morphology and connectivity. In order to assess the hierarchical microstructure, X-ray computed micro-tomography was employed as an imaging technique to supply three-dimensional images in two spatial scales. Two samples, at different sintering temperatures, were produced by emulsification of sunflower oil in aqueous aluminum oxide suspensions using gelatin bloom 90 as the gelling agent, dodecyl sodium sulfate as the surfactant and native cassava starch as the gelling and porogenic agent. Image processing and morphological characterization were performed on each spatial scale, individually allowing the calculation of the total porosity and pore size distribution of the ceramic sample. For the numerical simulation of the intrinsic permeability, representative images of the two spatial scales were superposed in a single super-image. This simulation was performed on the 3D pore network extracted from the super-image. The simulated permeability results were interpreted on the basis of the microstructure.

* 1. Introduction

The main purpose in materials science and engineering is to develop materials with macroscopic properties adequate to a specific application. Materials macroproperties are highly influenced by their microstructure, including intrinsic properties of the phases such as their quantity, morphology and connectivity.

A wide variety of porous ceramics are used for various applications including filtration, adsorption and catalysis, tissue engineering, and fuel cells (Gerke et al., 2015). Important physical properties of these ceramics, such as permeability, thermal and electric conductivity and mechanical resistance are closely related to their microstructure. For instance, intrinsic permeability depends on the effective (open) porosity, pore size distribution and the connectivity of the pore space.

The quantitative description of the morphology and connectivity of porous ceramics becomes important for a better understanding of the relationship between properties and microstructure. For this purpose, 3D images obtained by means of X-ray computed micro-tomography (µCT) coupled with image analysis are currently very common (Appoloni et al., 2004; Maire et al., 2007; Gerke et al., 2015; Gregorová et al., 2018; Lux et al., 2018).

Besides morphological characterization, 3D images make it possible to assess macroscopic properties by numerical simulation of physical phenomena in representative elementary volumes of the material. In addition, numerical simulation of properties is important because sometimes laboratory measurement can require a complex or time-consuming procedure, and can even cause sample brittleness (Gerke et al., 2015).

As well as porous ceramics, a wide class of materials can exhibit several spatial length scales such as carbonate rocks and cementitious materials. The microstructure of these multiscale materials is composed of multiple porosities organized hierarchically. The description and numerical simulation of properties of such a hierarchical microstructure is a challenge and can involve the combination of complementary techniques as shown in the work of Gregorová et al. (2018) using mercury porosimetry, stereology techniques on 2D images, and 3D images acquired with X-ray micro-tomography.

In this article, we present a workflow of image processing and analysis that characterizes the morphology and simulates the intrinsic permeability of a double porosity ceramic from 3D images of two spatial scales obtained using X-ray micro-tomography.

Among the various methods to numerically simulate the intrinsic or Darcy permeability, the approach based on the pore network extracted from the 3D porous space of µCT images was used (Dong and Blunt, 2009). Besides permeability, the pore network representation was also used for the determination of the coordination number and average throat size.

* 1. Materials and Methods
     1. Porous ceramic preparation via gelcasting

The development of the ceramic samples was accomplished by emulsification of sunflower oil in aqueous aluminum oxide (alumina, Al2O3) suspensions using gelatin bloom 90 as the gelling agent, dodecyl sodium sulfate as the surfactant and native cassava starch as the gelling and porogenic agent, as described elsewhere (Calado, 2017). In a muffle oven, the organic compounds were removed at a heating rate of 2° C/min up to 1100° C, followed by a 2 h isothermal plateau. Sintering was carried out in a tubular furnace at a heating rate of 2° C/min to 1400° C or 1550° C with a final 2-h plateau to produce the samples under study: S11 and E11.

* + 1. Porous ceramic characterization via 3D image processing

**Image acquisition**

An X-ray computed micro-tomograph (Zeiss Versa XRM-500) was used to observe and characterize the details of the internal structure of the samples at a spatial resolution of micrometer-order. Typically, the sample preparation is minimal, and the technique is mostly nondestructive, allowing many scans to be made at the same sample under different conditions. However, there is a trade-off between spatial resolution and sample size: high resolutions require small samples. Because of this, one single tomography acquisition image is often unable to show the entire pore size distribution of the sample. To address this problem, an original volume and a sub-volume of the ceramics were scanned under appropriate spatial resolutions, allowing each of them to show a certain range of pore sizes. Figures 1a and 1c show sample images, represent with low-resolution (LR), which is a plug of 6 mm x 10 mm (diameter and height). The high-resolution (HR) images required a sub-volume cut from the plugs (Figures 1b and 1d) with 1 mm x 3 mm of dimension. The voxel sizes of the LR and HR images are 6.8 µm and 1.2 µm, respectively.

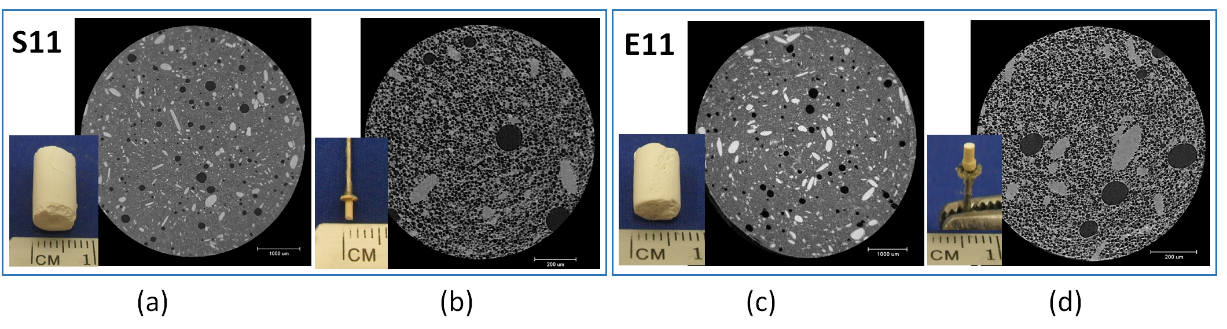


Figure 1. µCT images of S11 and E11. Figures (a) and (c) LH images (6.8 µm) acquired from samples of 6 mm x 10 mm. Figures (b) and (d) HR images (1.2 µm) acquired from sub-volume of 1 mm x 3 mm.

**Image processing**

An anisotropic diffusion filter was applied to the original low resolution µCT images (Figure 1a and 1c) after the VOI (volume of interest) delimitation (Weickert, 2001). This filtering operation was used to minimize internal variability in regions whilst preserving the contour between regions. Then, a classification was performed using Polynomial Mahalanobis (Grudic and Mulligan, 2006) to determine data grouping, denominated as pore, solid (alumina agglomerates) and non-solved (grey regions in Figure 1a and c). Applying a threshold to these regions, definitive phases inside the images of scale 1 were determined. Original high-resolution images (Figure 1b and 1d) were directly submitted for classification without filtering, resulting in two groups of data. Again, after classification, a threshold was applied confirming these regions and classifying every voxel as pore or solid. Image processing, morphological characterization and permeability simulation were performed dedicated software (Imago3D, LMPT/UFSC).

**Image characterization**

Once the pore space was segmented, the pore size distribution was measured in both LR and HR images, by opening morphological operation, i.e., erosion followed by dilation (Coster and Chermant, 1989). The opening operation was applied to the so-called background distance image using the chamfer metric d3-4-5 to generate the 3D balls (Borgefors, 1986; Thiel and Montanvert, 1992).

The 3D pore space can be represented in a pore network model where the pore space is partitioned in two basic elements: large voids (called pores) and constrictions throats that connect, at least, two pores. In general, spheres and cylinders are used to represent pores and constrictions, respectively. The computational code was constructed based on the maximal ball method (Silin and Patzek, 2006; Dong and Blunt, 2009). The original volume of pores and throats as well as the connectivity of the pore space is preserved in the pore network model. In the present work, the pore network representation for the numerical calculation of the Darcian permeability was used.

Porosity and pore size distribution were calculated in each spatial scale (individually) and afterwards placed together using a multiscale composition model developed for additive properties (Papadopoulos et al., 2009; Mantovani, 2013). The mathematical model is based on the fact that the LR image (scale 1 – s1) is composed of two well-defined regions of porosity and solids, and a third region, where it is difficult to ascertain if it is either composed of solids or by sub-micro pores under the spatial resolution. The undefined elements of this region, named as non-solved region, can be solved in the HR image (scale 2 – s2). The total porosity of the sample quantified considering the two spatial scales (images LR and HR), is given by:

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|  | (1) |

where is the volume fraction of non-solved region in scale 1, and and are the porosities of scale 1 and 2, respectively. Considering as the pore size distribution, i.e., the volumetric fractions measured for each *i* (*i* = 1, 2) scale, the total pore size distributions and can be given, respectively, by:

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|  | (2) |

In these equations, each scale covers a range of radii of pores and there is no intersection between each range.

The REV (Representative Elementary Volume) concept defines the minimum volume in a pore system for which the continuum assumption is valid; so, in this volume, macroscopic properties can be calculated. In this work, we determined the REV for porosity testing random subvolumes extracted from the super-image. The subvolumes sizes (VL) were increased in edge length (L) until the porosity converged to a certain tolerance, (±δ). With this procedure, the minimum edge length that defines REV is (Petrasch et al., 2008):

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|  | (3) |

* + 1. Super-image Permeability Numerical Simulation

Using the pore network model, it was possible to simulate the Darcian permeability of scales 1 (LR image) and 2 (HR image) individually. There are models proposed to simulate permeability of multiscale systems from the pore network extracted from each scale, such as the model of Jiang et al (2013). In this case, for the particular dual porosity microstructure analyzed, permeability was predicted using the pore network extracted directly from a so-called super-image, obtained by the combination of LR and HR images as follows. The procedure starts by cropping a representative region of scale 1 (LR image) and scale 2 (HR image). First, let´s consider R the ratio between the voxel size of LR and HR images. For the images analyzed here, R is approximated by six. Then, the size of the selected cropped section of the LR image is enlarged so that each voxel has the same size of the HR image. To do this, each voxel of the LR image is replicated R times on the three orthogonal directions of the image. The cropped section of the LR image is composed of three regions: pore space, solids and non-solved region. Voxels of the last region are shown by the HR image. These voxels are replaced by inserting the cropped section of the HR image into the regions of non-solved voxels of the enlarged LR image, keeping untouched voxels which were already labeled as pore or solid. In this operation, the cropped section of the HR image is repeatedly rotated and reinserted into the super-image until it completely fills the non-solved region. At the end of this procedure, the super-image is obtained, which is composed solely of voxels labeled as pore and solid, statistically representing the entire microstructure of the ceramics (scale 1 with scale 2). The linear size of the super-image will be the size of the sample of LR image multiplied by R. In the super-image, the pore network is extracted, and the permeability is simulated.

* 1. Results and discussion

Examples of 3D images, before and after the segmentation process, are shown in Figure 2 for the sample S11 in both spatial scales (LR and HR images). We can observe the spherical shape of pores and the agglomerates of alumina particles in scale 1. The volume fraction and geometrical configuration of phases presented by sample E11 display similar behavior to those of sample S11.

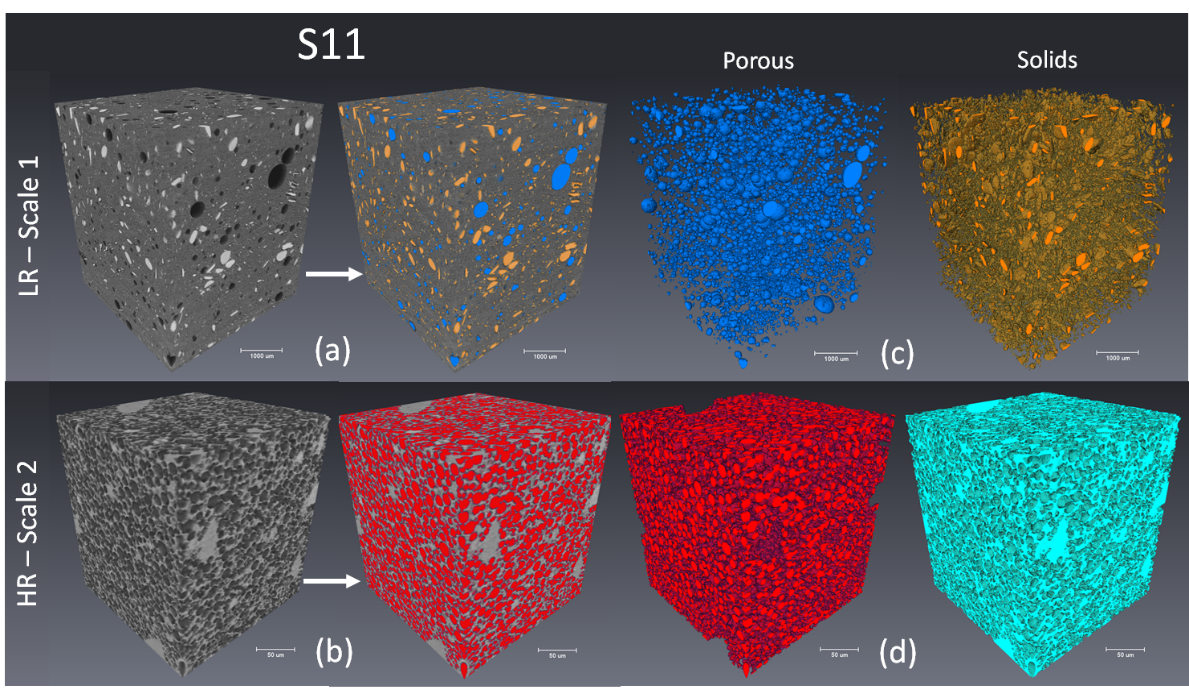


Figure 2. Segmentation image processing of sample S11. (a) LR images segmented in three-phases: porous (blue), solid (orange) and non-solved region (gray). (b) HR images segmented in porous (red) and solid (light green). (c) and (d) individual pores and solid phases from the whole sample. Scale length to LR images is 1000 µm and to HR images is 50 µm.

Image processing results in the phase segmentation of samples S11 and E11 as porous, solid and non-solved regions for scale 1, and pore and solid for scale 2 quantifying their volume fractions. The data obtained from the images in each scale was the input data for the multiscale model (Eq(1) and Eq(2)), quantifying the cellular ceramics as a whole. Table 1 shows volume fraction values as well as the average coordination number and Darcian permeability calculated by pore network modeling.

Table 1: Phase fraction (pores and solids), in each scale, and in entire sample determined by multiscale model and in the super-image.

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| --- | --- | --- | --- | --- | --- |
| Sample | Scale/  Resolution (µm) | Solid fraction (%) | Pore fraction (%) | Average coordination number | Darcian permeability (mD) |
| S11 | s1/6.82 | 8.61 | 3.87 | - | 0.00 |
|  | s2/1.17 | 61.61 | 38.39 | 3.90 | 43.90 |
|  | Multiscale | 62.53 | 37.47 | - | - |
|  | Super-Image | 67.77 | 35.23 | 4.00 | 25.10 |
|  |  |  |  |  |  |
| E11 | s1/6.82 | 7.11 | 3.35 | - | 0.00 |
|  | s2/1.17 | 55.90 | 44.10 | 4.30 | 43.50 |
|  | Multiscale | 57.17 | 42.84 | - | - |
|  | Super-Image | 57.97 | 42.03 | 4.40 | 32.80 |

Figure 3 shows the REV of samples S11 and E11 determined by porosity in relation to sub-volumes taken at random positions inside the VOI used in the image processing. These sub-volumes were then enlarged in increments of 103 voxels (203, 303, 403…) until the entire volume of the super-image was reached.

The pore size distributions measured using opening for each spatial scale for samples S11 and E11 are shown in Figure 4a. Figure 4b presents the total pore distribution measured (by opening) in the super-image compared to that calculated by multiscale Eq(2) with the data presented in Figure 4a.

Figure 3. Representative Elementary Volume for the porosity of the samples S11 and E11 determined in the super-image.

The 3D image characterization allows comparison of the morphology and the connectivity of the porous spaces of the ceramics samples E11 and S11. The pore size distribution (Figure 4) showed similarity between E11 and S11 essentially when the spatial scale 2 (HR image) was considered. Using the pore network modeling, we calculated the average throat size of scale 2 as approximately 0.9 μm and 0.8 μm for S11 and E11, respectively. Considering the super-image, it can be observed from Table 1 that the sample E11 has porosity and an average coordination number larger than the S11 sample. From super-images, the values of the simulated Darcian permeability of approximately 25 mD were obtained for S11 and ~35 mD for E11, probably due to the slightly better connectivity and larger porosity in the latter case. Individually, scale 1 from both E11 and S11 correspond to non-percolating systems; the percolation arises due the connectivity between the pores of scale 2. Therefore, the hydraulic resistance of the fluid flow in samples S11 and E11 are governed by scale 2. The results obtained for the total porosity and total pore size distribution were very close considering the calculation by the multiscale model and the measurement on the super-image.

|  |  |
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| (a) | (b) |

Figure 4. Pore size distribution for (a) each scale, and (b) composed as total (bimodal) by multiscale model and measured in the super-image. All distributions determined by mathematical morphology.

* 1. Conclusions

A morphological characterization of ceramics samples with dual porosity on 3D µCT images was carried out in this paper using a proposed image analysis pipeline. Total porosity and total pore size distribution were determined either by a multiscale model or by a constructed super-image that encompass microstructural features of both spatial scales. Results from the super-image and multiscale model are in good agreement. The permeability of the two samples was interpreted based on the morphology and connectivity of the microstructures. The analysis based on 3D images, particularly in the case of porous materials with two or more characteristic length scales, is a powerful tool for a better understanding of the relationship between microstructure and macroscopic properties.

* 1. Acknowledgments

The authors thank the Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) and PETROBRAS.

* 1. References

Appoloni C.R., Fernandes C.P., Innocentini M.D.M, Macedo A., 2004, Ceramic foams porous microstructure characterization by X-ray microtomography, Materials Research, 7(4), 557-564.

Borgefors G., 1986, Distance transformations in digital images, Computer Vision, Graphics, and Image Processing, 34, 344-371.

Calado C.M.B., 2017, Cerâmicas celulares obtidas a partir da emulsificação de suspensões de alumina com óleo de girassol, amido e gelatina, Master thesis, Federal University of Santa Catarina (in Portuguese).

Coster M., and Chermant J.L., 1989, Precis d’analyse d’images, Presses du CNRS, Paris (in French).

Dong H. and Blunt M., 2009, Pore-network extraction from micro-computerized-tomography images, Physical Review E, 80(3), 1-11.

Gerke K.M., Korost D.V., Vasilyev R.V., Karsanina M.V., Tarasovskii, V.P., 2015, Studying structure and determining permeability of materials based on X-Ray microtomography data (using porous ceramics as an example), Inorganic Materials 51(9), 951-957.

Gregorová E., Uhlířová T., Pabst W., Diblíková P., Sedlářová I., 2018, Microstructure characterization of mullite foam by image analysis, mercury porosimetry and X-ray computed microtomography, Ceramics International, 44(11), 12315-12328.

Grudic G., Mulligan J., 2006, Outdoor path labeling using polynomial mahalanobis distance, Robotics: Science and Systems II Conference, University of Pennsylvania, Philadelphia, Pennsylvania, USA.

Jiang Z., van Dijke M.I.J., Sorbie K.S., Couples G.D., 2013, Representation of multi-scale heterogeneity via multi-scale pore networks, Water Resources Research, 49, 5437–5449.

Lux J., Guzi de Moraes E., Maire E., Adrien J., Biasett, L., 2018, Gas permeability of Ti6Al4V foams prepared via gelcasting, experiments and modelling, Computational Materials Science, 152, 363-373.

Maire E., Colombo P., Adrien J., Babout L., Biasetto l., 2007, Characterization of the morphology of cellular ceramics by 3D image processing of X-ray tomography, Journal of the European Ceramic Society, 27(4), 1973-1981.

Mantovani I.F., 2013, Microtomografia e nanotomografia de raios X aplicada à caracterização multiescalar de sistemas porosos carbonáticos. PhD thesis, Federal University of Santa Catarina (in Portuguese).

Papadopoulos, A., Whitmore A.P., White R.P., Mooney S. J., and Bird N.R.A., 2009, Combining Spatial Resolutions in the Multiscale Analysis of Soil Pore-Size Distributions, Vadose Zone Journal, 8(1), 227.

Petrasch J., Wyss P., Stämpfli R., Steinfeld A., 2008, Tomography‐Based Multiscale Analyses of the 3D Geometrical Morphology of Reticulated Porous Ceramics, The American Ceramic Society, 9(8), 2659-2665.

Silin D., Patzek T., 2006, Pore space morphology analysis using maximal inscribed spheres, Physica A: Statistical Mechanics and its Applications, 371(2), 330–360.

Thiel E. and Montanvert A., 1992, Shape splitting from medial lines using the 3–4 chamfer distance, Proceedings., 11th IAPR International Conference on Pattern Recognition. Vol. III. Conference C: Image, Speech and Signal Analysis, New York: Plenum, 537–546.

Weickert J., 2001, Applications of nonlinear diffusion in image processing and computer vision, Acta Mathematica Universitatis Comenianae, 70(1), 33-50.

Dear Editor and Reviewers,

We thank you for your letter and the time you spent in reviewing our manuscript entitled “Predicting intrinsic permeability of a double porosity cellular ceramic using X-ray micro-tomography” (Paper # 99).

All comments were very helpful for improving our paper. The manuscript was revised by a native English speaker. The responses to the reviewers’ comments are provided below in red.

If you have any further queries, please do not hesitate to contact us.

Kind regards,

Dr. Iara F. Mantovani and co-authors

**Comments by reviewer #1**

This paper is aimed to predict the intrinsic permeability of cellular ceramic by an imaging technique based on X-ray computed microtomography.

The thematic is not very novel since literature already reports many papers on this topic. However the results may be interesting for researchers working in this sector.

Section 2.1. Please specify how this sentence/result "The microstructure of the resulting ceramic is constituted of spherical pores whose distribution is bimodal" has been reached.

This sentence is one conclusion from our image analysis. It is in a wrong place, so was removed from Section 2.1.

English should be revised by a native English speaking.

The paper was revised by a native English speaker

**Comments by reviewer #2**

This paper should be accepted after minor changes.

Please correct/rewrite the following spelling mistakes:

‘For the numerical simulation of the intrinsic permeability ***~~representatives~~*** images of the two spatial scales were superposed in a single super-image.’ - representative

‘***~~Main purpose~~*** in materials science and engineering (…)’ – A main purpose

‘In this sense, the quantitative description of the morphology and connectivity of porous ceramics ***~~becomes~~*** important (…)’ – become

‘The microstructure of these multiscale materials ***~~are~~*** composed of multiples porosities hierarchically organized.’ – is

‘Among the various methods to numerically simulate the intrinsic permeability (Darcy permeability), ***~~we use the approach based on the pore network extracted from the 3-D porous space of µCT images~~***.’ - the approach based on the pore network extracted from the 3-D porous space of µCT images was used.

‘The microstructure of the resulting ceramic ***~~is~~*** constituted of spherical pores whose distribution ***~~is~~*** bimodal.’ – was, was.

‘Typically, the sample preparation is minimal, and for many materials, the technique is nondestructive allowing many scans to be made ***~~of~~*** the same sample under different conditions.’ – at.

‘(…) an original volume and a sub-volume of the ceramics were scanned under appropriates spatial resolutions, allowing each of ***~~them describe~~*** a certain range of pore sizes.’ – them to describe.

‘Figures 1a and 1c ***~~shows the sample imaging~~*** with low-resolution (LR), which ***~~is~~*** a plug of 6 mm x 10 mm (diameter and height).’ – show sample images, represent

‘(…) a sub-volume cut from the plugs (Figures 1b and 1d) with ***~~of~~***1 mm x 3 mm of dimensions.’

‘(…) after the VOI (volume of interest) delimitation an anisotropic diffusion filtering ***~~is~~*** applied’ – was.

‘This filtering operation is used to minimize internal variability in regions ***~~but still~~*** preserving the contour between regions.’ – while

‘Original High Resolution images (Figure 1b and 1d) ***~~are~~*** directly submitted to classification without filtering, resulting in two groups of data.’ – were.

‘Again, after classification, a threshold ***~~is~~*** applied confirming these regions and deciding every voxel into pore or solid. ‘ – was.

‘Once the pore space was segmented the pore size distribution was measured, in both LR and HR images, by ***~~morphological operation opening~~*** .’ – opening morphological operation.

‘***~~Our~~*** computational code was constructed based on the maximal ball method.’ – The.

‘In the present work, ***~~we use the pore network representation for the numerical calculation of the Darcian Permeability.~~***’ - the pore network representation for the numerical calculation of the Darcian Permeability was used.

‘The undefined elements of this region, named as non-solved region, ***~~will be solve~~*** in the HR image (scale 2).’ – can be solved.

‘***~~Now, consider the pore size distribution, that is, the volumetric fractions measured for each i (i = 1, 2) scale. The total pore size distributions and are given, respectively, by~~***: ‘ - Considering as the pore size distribution, that is, the volumetric fractions measured for each *i* (*i* = 1, 2) scale, the total pore size distributions and can be given, respectively, by:

‘The REV (Representative Elementary Volume) concept defines the minimum volume in a pore system for which the continuum assumption is valid, so, in this volume, ***~~we can define macroscopic properties~~***.’ – macroscopic properties can be defined

‘Using the pore network model ***~~we are able~~*** to simulate the Darcian permeability on the scales 1 (LR image) and 2 (HR image) individually.’ – it was able.

In this article, for the particular dual porosity microstructure analyzed ***~~we simulate the permeability extracting the pore network directly in a constructed image~~*** (…)’ - permeability was extracted directly from the pore network of a constructed image,

‘***~~We can observe~~*** the spherical shape for pores and the agglomerates of alumina particles at scale 1.’ – It can be observed.

‘The 3D image characterization ***~~allow comparing~~*** the morphology (…)’ - allows the comparison of.

‘The pore size distribution (Figure 4) ***~~shows~~*** similarity between E11 and S11 essentially when ***~~we consider the spatial scale 2 (HR image).~~*** ‘ – showed, the spatial scale 2 (HR images) were considered.

‘Besides, using pore network ***~~we calculated the average throat size of scale 2 as about 0.9 m and about 0.8 m for S11 and E11~~***, respectively.’ – an average throat size of scale 2 as about 0.9 µm and about 0.8 µm for S11 and E11 were calculated.

‘Considering the super-image ***~~we observe~~*** from Table (…)’ – it can be observed.

‘From super-images, ***~~we obtain~~*** the values of the simulated Darcian permeability (…)’ – it was obtained.

‘(…) a constructed super-image that encompass microstructural features of ***~~the~~*** both spatial scales.’

All spelling mistakes were corrected/rewritten.

**SPECIFIC COMMENTS**

1. ‘Applying a threshold to these regions, it decides definitive phases inside of images of scale 1.’ – The sentence is not clear. Please rewrite it.

The sentence was rewritten as:

“Applying a threshold to these regions, definitive phases inside the images of scale 1 were determined.”

1. ‘In this work we determine the REV for porosity from incrementally growing subvolumes V(L) of cubes of edge length L until their calculated porosities convergences within a certain tolerance, (±δ).’ – The sentence is not clear. Please rewrite it.

The sentence was rewritten as:

“In this work, we determined the REV for porosity testing random subvolumes extracted from the super-image. The subvolumes sizes (VL) were increased in edge length (L) until the porosity converged to a certain tolerance, (±δ).”

1. ‘Different of porosity and pore distribution, permeability is not an additive property.‘ - The sentence is not clear. Please rewrite it.

The sentence was removed from paper.

1. ‘Consider R the ratio between the voxel size of LR and HR images. For the images analyzed here R is approximated by six. Now, the size of the selected sample of the LR image is enlarged in the way that each voxel have the same size of HR image. To do this, each voxel of the LR image is replicated R times on the three orthogonal directions of the image. The sample of the LR image is composed by three regions: pore space, solids and the non-solved region. Voxels of the last region are described by HR image. So, we replace these voxels inserting the sample of HR image into the regions of non-solved voxels of the enlarged LR image, keeping untouched voxels which are already labeled as pore or solid. In this operation the sample of the HR image suffer repeatedly random rotation before insertion. At the end of this procedure, we have the super-image, composed only by voxels labeled as pore and solid representing, statistically, the entire microstructure of the ceramics (scale 1 together scale 2). The linear size of the super-image will be the size of the sample of LR image multiplied by R. In the super-image, the pore network is extracted and the permeability is simulated.’ – This paragraph is not clear. Try to connect better the sentences, avoid the use of first-person pronouns (‘we’) and avoid the overuse of short and simple sentences. Please improve the English.

The sentence was rewrite as:

“First, let´s consider R the ratio between the voxel size of LR and HR images. For the images analyzed here, R is approximated by six. Then, the size of the selected cropped section of the LR image is enlarged so that each voxel has the same size of the HR image. To do this, each voxel of the LR image is replicated R times on the three orthogonal directions of the image. The cropped section of the LR image is composed of three regions: pore space, solids and non-solved region. Voxels of the last region are shown by the HR image. These voxels are replaced by inserting the cropped section of the HR image into the regions of non-solved voxels of the enlarged LR image, keeping untouched voxels which were already labeled as pore or solid. In this operation, the cropped section of the HR image is repeatedly rotated and reinserted into the super-image until it completely fills the non-solved region. At the end of this procedure, the super-image is obtained, which is composed solely of voxels labeled as pore and solid, statistically representing the entire microstructure of the ceramics (scale 1 with scale 2). The linear size of the super-image will be the size of the sample of LR image multiplied by R. In the super-image, the pore network is extracted, and the permeability is simulated.”

1. ‘Image processing, morphological characterization and permeability simulation were performed using Imago3D a software developed in the scope of a project between LMPT/UFSC (Porous Media and Thermophysical Properties Laboratory/UFSC) and PETROBRAS (Brazilian Petroleum Company).‘ - This should be in materials and methods section.

The sentence was moved to section 2.2.

1. Please check the references:

‘Calado C.M.B., 2017, Cerâmicas celulares obtidas a partir da emulsificação de suspensões de alumina com óleo de girassol, amido e gelatina, Master thesis, Federal University of Santa Catarina.’ - Please inform that it is not in English. (in Portuguese).

‘Coster M., and Chermant J.L., 1989, Precis d’analyse d’images, Presses du CNRS, Paris.’ - Please inform that it is not in English (in French).

‘Dong H. and Blunt M., 2009, Pore-network extraction from micro-computerized-tomography images, Physical Review E, 80 036307, 1-11.’ - Please check volume information.

‘Mantovani I.F., 2013, Microtomografia e nanotomografia de raios X aplicada à caracterização multiescalar de sistemas porosos carbonáticos. PhD thesis, Federal University of Santa Catarina.’ - Please inform that it is not in English. (in Portuguese).

‘Papadopoulos, A., Whitmore A.P., White R.P., Mooney S. J., and Bird N.R.A., 2009, Combining Spatial Resolutions in the Multiscale Analysis of Soil Pore-Size Distributions: Vadose Zone Journal, 8(1), 227.’ - Please check the punctuation after the title of the article.

All references were checked and eventually rewritten.