Microstructural Characterization of Siltstone and Sandstone Pore Space by X-Ray Microtomography

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Abstract. Microstructural parameter assessment of reservoir rocks is extremely important for oil companies. In this regard, computerized X-ray microtomography (μ-CT) has proven to be an exceptionally useful methodology for the analysis of these rocks, since it provides significant microstructural parameters, such as porosity, permeability, and pore distribution. X-ray microtomography is a relatively novel non-destructive technique in the petroleum area which, besides enabling the reuse of already measured samples, also provides 2-D and 3-D images, as well as a 3-D mathematical model of the sample. This technique has the great advantage that it does not require sample preparation, allowing the reuse of the sample and also reducing the measurement time. A Skyscan model 1172 microtomograph was used for the acquisition of microtomographic data from the reservoir rocks. This work presents results of porosity and pore size distribution of six sandstones and two siltstones. Most of the analyzed samples from the Tibagi River basin had porosity below 10 %, except for the PG6 and PG8 samples, which presented 12 % and 13 %, respectively. Maximum pore radius ranged from 8 to 59 μm for sandstones and from 5 to 6 μm for siltstones.

Keywords: pore size distribution, porosity, sandstone, siltstone, X-ray microtomography.

1. Introduction

X-ray computerized microtomography (μ-CT) is a non-destructive methodology that measures density variations of the material. This technique uses a set of two-dimensional projections of an object to reconstruct its three-dimensional structure (Landis & Keane, 2010), using a mathematical algorithm (Cormack, 1963, 1964; Wellington and Vinegar, 1987; Flannery et al., 1987). It requires the employment of adequate software for the treatment and analysis of images that enables the determination of important microstructure parameters, thus providing a better knowledge of the analyzed material. A schematic illustration of the acquisition, reconstruction, and generation of 3-D images and models is shown in Fig. 1 (Fernandes et al., 2016).

The application of X-ray microtomography and image analysis enabled the determination of some microstructural parameters for analyzing five samples of sandstone and two of siltstone from various geological formations in Brazil, which are or may become reservoir rocks for some type of fluid. Additionally, a sample of Australian sandstone was also analyzed. Microcomputed tomography (μ-CT) provides two-dimensional (2-D) and three-dimensional (3-D) images, from which various characteristics, such as porosity, permeability, and phase size distribution (pores or solids) can be obtained. Furthermore, it permits the creation of a 3-D volume of the sample, allowing the visualization of the actual structure of the porous network present in the scanned volume and also for mathematical simulations. As X-ray beams pass through the object being scanned, the signal is attenuated by scattering and absorption. The basic equation for attenuation of a monoenergetic beam by a homogeneous material is given by the law of radiation absorption (Siegbahn, 1979):

![Figure 1 - Representation of the acquisition, reconstruction, and generation of three-dimensional images and models (Fernandes et al., 2016).](image-url)
where \( I_0 \) and \( I \) are the intensity of incident and transmitted X-ray beams, respectively, \( \mu \) is the linear attenuation coefficient for the material being scanned, and \( x \) is the thickness of the sample through which the beam will traverse. As in most cases the scanned object is composed of a different number of materials and the energy of the X-ray beam tube is polyenergetic, this equation is rewritten as (Kalender, 2011):

\[
I = \int_{0}^{E_{\text{max}}} I_0(E) \exp \left( -\int_{0}^{x} \mu(E,x) \, dx \right) \tag{2}
\]

The petroleum industry commonly uses other methods to determine porosity and permeability, among which the most widespread is mercury intrusion porosimetry. The principle of this technique is based on the fact that mercury does not wet most substances and, therefore, will not penetrate pores by capillary action, unless it is forced to do so. Liquid mercury has a high surface tension and also exhibits a high contact angle against most solids. Entry into pore spaces requires applying pressure in inverse proportion to the pore diameter (Lowell & Shields, 1991; Webb & Orr, 1997; Johnson et al., 1999). However, due to their destructive nature, this method often prevents the use of the samples for future analysis. Besides enabling future analyses of the sample already measured, the \( \mu \)-CT also provides internal visualization of the sample through three-dimensional images (Fernandes et al., 2016).

Through the most widespread methods such as mercury intrusion porosimetry, a sample may exhibit porosity lower than that it actually possesses, due to the lack of connection between the pores of the rock, which through the 3-D images obtained by microtomography, is verified and quantified.

The importance of not destroying the sample is justified when there is a need for future analysis, or by evolution of measurement techniques, or archiving of samples in their natural state.

The determination of the porosity of reservoir rocks through X-ray microtomography is quite widespread (Ap poloni et al., 2007; Flukiger and Bernard, 2009; Tsaki rogloú et al., 2009; Izgec et al., 2010), however, for the rocks of the Tíbagi River basin such analyzes are pioneering.

After being microphotographed, the samples were submitted to mercury porosimetry analyses to obtain the total porosity for comparison of the achieved microCT results.

2. Materials and Methods

2.1. Rocks analyzed

Except for the sample from the Tumblagooda Formation, which was collected from an outcrop of the Kalbarri National Park in Australia and provided by CENPES / PETROBRAS, all other rock specimens were taken from the Tíbagi River Basin located in the central region of the Paraná State, Brazil. Table 1 shows a description summary of the rock samples analyzed, indicating their collection site, group, and formation. Such samples were selected to represent rocks of easy analysis (with larger pores and without clay) and rocks of difficult analysis (smaller pores and often with clay).

Figure 2 shows scale images of the analyzed samples with parallelepipedic shapes with diameters ranging from 5 to 8 mm and height from 10 to 20 mm to facilitate visualization of their dimensions.

Table 2 shows information about some of the most significant acquisition parameters. The number of projec-
tions for each sample can be obtained dividing the total rotation by the angular step.

For the acquisition of the above data, a 1 mm thick aluminum filter was used, aiming to reduce beam hardening effects on the sample images (Ketcham and Carlson, 2001).

2.2. Equipment and software used

A microtomograph Skyscan model 1172, installed at the Research and Development Center (CENPES) of PETROBRAS, Rio de Janeiro, RJ, Brazil, was used. The microtomographic images (projections) were reconstructed by the NRecon software (Skyscan, 2018). The porosities of the samples were obtained through the Imago software (Imago, 2018) developed in the Laboratory of Porous Media and Thermophysical Properties of Materials (LMPT) of the Department of Mechanical Engineering of the Federal University of Santa Catarina, Florianópolis, SC, Brazil, in association with the Brazilian software company ESSS (Engineering Simulation and Scientific Software). Other software used was the CTan (Skyscan, 2018), with which the 3-D reconstructions of the samples were performed. Figure 3 (Fernandes et al., 2016) shows a photo of the Skyscan 1172 microtomograph installed at CENPES.

This microtomograph is provided with an X-ray tube with tungsten anode (W), 10 W maximum power, 20-100 kV voltage, and 0-250 μA current. A CCD camera of 10 Mp (megapixel) was used to detect the X-rays. The maximum spatial resolution obtained by this equipment is 1 μm for samples with 5 mm maximum diameter and 80 mm height (Fernandes et al., 2016). To determine sample porosity for comparison purposes, a commercial porosimeter, PoreSizer model 9320 of Micromeritics was applied. However, it was not possible to perform the intrusion into one of the samples, since it had pores of very small dimensions, thus precluding the measurements.

Table 2 - Key parameters used for the µ-CT image acquisition.

| Sample            | Tension (kV) | Total rotation (°) | Angular step (°) | Spatial resolution (μm) | Exposure time for each projection (ms) | No. of frames | Total acquisition time |
|-------------------|--------------|--------------------|------------------|-------------------------|---------------------------------------|---------------|------------------------|
| Sandstone 107     | 70           | 0 to 360           | 0.25             | 2.9                     | 3245                                  | 5             | 6 h 28 min             |
| Sandstone 108     | 70           | 0 to 180           | 0.25             | 2.9                     | 9735                                  | 4             | 8 h 5 min              |
| Sandstone 403     | 70           | 0 to 360           | 0.25             | 1.4                     | 2655                                  | 3             | 3 h                    |
| Siltstone MC16    | 70           | 0 to 180           | 0.25             | 2.5                     | 632                                   | 5             | 59 min                 |
| Siltstone PG6     | 70           | 0 to 180           | 0.25             | 2.9                     | 2655                                  | 5             | 2 h 45 min             |
| Sandstone PG8     | 80           | 0 to 180           | 0.25             | 5.0                     | 1180                                  | 5             | 1 h 30 min             |
| Sandstone PG19    | 70           | 0 to 180           | 0.25             | 1.4                     | 8260                                  | 2             | 3 h 30 min             |
| Sandstone Tumblagooda | 50           | 0 to 180           | 0.5              | 2.9                     | 4425                                  | 5             | 2 h 18 min             |

Figure 2 - Images of rock samples analyzed in this work. (A) Sandstone 107; (B) Sandstone 108; (C) Sandstone 403; (D) Siltstone MC16; (E) Siltstone PG6; (F) Sandstone PG8; (G) Sandstone PG19; (H) Sandstone Tumblagooda.

Figure 3 - Microtomograph Skyscan 1172, 20 - 100 kV X-ray source, 10 W maximum power (Fernandes et al., 2016).
2.3. Image processing

Figure 4 shows two projections, 0° and 180°, acquired by the CCD chamber of sandstone sample 107. The projections were reconstructed by the NRecon software using a cone-beam reconstruction algorithm (Feldkamp et al., 1984), resulting in 2-D grayscale images of a sandstone and a siltstone, as shown in Figs. 5a and 6a, respectively. After generation, the 2-D images are analyzed by the Imago software. In this step a region of interest (ROI) is defined and binarization (segmentation) (Imago, 2002) is performed, transforming the grayscale image into black and white, where black is the solid phase, and white, the porous phase, as shown in Figs. 5b and 6b (Fernandes et al., 2016).

From the projection of 2-D images, it was reconstructed 3-D images using the CTan software, to reconstruct the actual volume of the rocks analyzed. As the 3-D reconstruction process is very limited and requires a large computational capacity, it was necessary to investigate the representative elementary volume (REV) of these samples, which must be large enough to represent the characteristics of the sample and the smallest possible when compared to its total volume. This scaling was analyzed, and it was concluded that the REV should be $1400 \times 1400 \times 1400 \, \mu m$ (Fernandes et al., 2012). For a better view of the internal structure of sandstone 107, a subvolume of $1500 \times 1001 \times 300 \, \mu m$ was reconstructed, as shown in Fig. 7.

Figure 8 (Fernandes et al., 2012) shows the 3-D image of the PG8 sandstone sample: in yellow, the porous networks with relatively large diameters and few isolated pores; in gray, the solid phase.

3. Results

Figure 7 clearly shows that the pores of sandstone sample 107 are isolated, having no connection among them, thereby preventing the flow of any fluid through the rock, thus providing virtually no permeability. This same figure reveals that there are relatively large, but few pores, reflecting the low porosity of the sample. The porosity ($\phi$) for this subvolume is $5.4 \pm 0.2 \%$, and even with a relatively small subvolume, this value is very close to the mean value of the porosity determined from all analyzed 2-D sections ($\phi = 6.1 \pm 0.2 \%$).

Table 3 shows the results of the average porosity of all 2-D sections, the porosity of the 3-D images based on the representative elementary volume, and the porosity obtained with the mercury porosimetry for all samples analyzed. In addition, the pore radius with the highest frequency in the pore size distribution histogram is also presented.
The results of Table 3 show that the porosity found by X-ray microtomography is systematically lower than that achieved by mercury porosimetry, except for sample 107. Regarding this sample, it is conceivable that mercury did not completely fill its pores. In fact, the outcomes of the microtomographic image analysis reveal that the pores of sample 107 are isolated. In this connection, these findings may explain the impossibility of mercury infiltration into some of its pores, thus impairing a good performance of this methodology to determine the absolute porosity of the sample.

For the other samples, the porosity obtained by the µ-CT was smaller. Therefore, it was concluded that there are pores smaller than the resolution used by the microtomograph. Since the porosimeter can measure pores up to 6 nm in diameter, the microtomograph did not detect a portion of the pores with smaller diameters and indicated lower porosity values for those samples.

The porosity value for all 2-D images compared to the porosity of the 3-D images obtained using the representative elementary volume can also be observed from Table 3. These values are statistically the same for most of the samples. This demonstrates the great advantage in determining REV for the generation of representative 3-D images.

Figures 9 to 16 show the pore size distributions for the analyzed samples. In these figures, it can also be observed that the histogram for sandstone 107 presents two distributions: one around the pores with a radius of 5.8 µm and another for pores with a radius of 58.7 µm, and that 95% of the pore of this sample are comprised between 2.9 µm and 108 µm.

For sandstone 108, approximately 92% of the porous phase refers to pores with radii between 2.9 and 20.6 µm and there is a distribution around a value of 11.7 µm.

From the sandstone 403 histogram, it can be observed that approximately 97% of the porous phase refers to pores with radii between 3.9 and 11.9 µm. The frequency of 37.5% for pores with a radius of 3.9 µm (spatial resolution used) is considered very high, indicating that there are pores with smaller radii.

The histogram for siltstone MC16 reveals that nearly 95% of the porous phase refers to pores with radii between 2.5 and 10.2 µm. Likewise, for sample 403, the 25% frequency for pores with a radius of 2.5 µm (spatial resolution used) is considered very high, revealing that there are pores with smaller radii. Notwithstanding, when analyzing the 2-D and 3-D images of this sample, we observed the pres-

![Figure 8 - 3-D image reconstructed from the sample PG8 (1400 × 1400 × 1400 µm): gray represents the solid phase, and yellow, the porous phase (Fernandes et al., 2012).](image)

### Table 3 - Summary of the microstructural properties of the analyzed samples.

| Sample          | ϕ sections 2-D (%) | ϕ image 3-D (%) | ϕ porosimetry Hg (%) | Pore<sup>3</sup> radius (µm) |
|-----------------|--------------------|-----------------|----------------------|-----------------------------|
| Sandstone 107   | 6.1 ± 0.2          | 5.4 ± 0.2       | 3.1                  | 5.8-58.7                    |
| Sandstone 108   | 4.2 ± 0.1          | 4.0 ± 0.1       | -                    | 11.7                        |
| Sandstone 403   | 9.3 ± 0.2          | 7.6 ± 0.2       | 12.9                 | 7.9                         |
| Siltstone MC16  | 7.0 ± 0.2          | 7.0 ± 0.2       | 13.4                 | 5.1                         |
| Siltstone PG6   | 12.0 ± 0.3         | 11.5 ± 0.2      | 18.5                 | 2.4-5.9                     |
| Sandstone PG8   | 13.0 ± 0.2         | 12.6 ± 0.2      | 15.8                 | 20                          |
| Sandstone PG19  | 4.9 ± 0.1          | 5.1 ± 0.1       | 8.4                  | 10                          |
| Sandstone Tumblagooda | 15.3 ± 0.5     | 13.4 ± 0.4       | -                    | 11.7-23.5                   |

<sup>1</sup>Average total porosity with 95% confidence.
<sup>2</sup>REV porosity with 95% confidence.
<sup>3</sup>Most frequent value.
Figure 9 - Distribution of pore sizes for sandstone 107.

Figure 10 - Distribution of pore sizes for sandstone 108.

Figure 11 - Average pore size distribution for sandstone 403.

Figure 12 - Average pore size distribution for siltstone MC16.

Figure 13 - Average pore size distribution for siltstone PG6.

Figure 14 - Average pore size distribution for sandstone PG8.

Figure 15 - Average pore size distribution for sandstone PG19.

Figure 16 - Average pore size distribution for sandstone Tumblagooda.
ence of cracks with a diameter of approximately 5-10 μm caused by the laminated part of the sample. Those fissures can be seen in Fig. 17 and are repeatedly found throughout the sample, justifying, in part, the high frequency at pores with smaller radii.

The histogram of siltstone PG6 shows that approximately 90 % of the porous phase refers to pores with radii between 2.9 and 11.7 μm. As for samples 403 and MC16, the 27 % frequency for pores with a radius of 2.9 μm (spatial resolution used) is considered very high, providing indications that there are pores with smaller radii.

Regarding sandstone PG8, it can be observed that approximately 91 % of the porous phase refers to pores with radii between 5 and 60 μm. The highest frequency refers to pores with radii equal to 20 μm.

Sandstone PG19 presented approximately 95 % of the porous phase with pores with radii between 5 and 15 μm. The 38 % frequency for pores with a radius of 5 μm (spatial resolution used) is considered very high, indicating that there are pores with smaller radii.

From the histogram of the Tumblagooda sandstone, it can be observed that approximately 90 % of the porous phase refers to pores with radii between 2.9 and 76.4 μm. This sandstone has a very heterogeneous distribution and does not present pores with high frequencies.

4. Conclusions

We observed that most of the analyzed samples from the Tibagi River basin had porosity below 10 %, except for the PG6 and PG8 samples, which presented 12 % and 13 %, respectively. If we consider the size of the Tibagi River ba-

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