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Quantification of fluids injection in a glass-bead matrix using X-ray microtomography

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ABSTRACT

Several daily activities involve the accumulation or percolation of fluids through porous media. X-ray microtomography is a non-invasive technique capable of providing images of the internal microstructure of materials showing the different phases of fluid distribution present in the sample directly or at the pore-scale. This methodology was used to qualitatively and quantitatively assess samples consisting with glass beads of standard size, which contained fluid filling a porous region. Three samples were prepared with 0.6 mm or 0.8 mm diameter glass beads inserted into a glass tube with an inner diameter of 6.7 mm and 1.0 mm wall thickness. The fluids injected were dopant salt–water solution, industrial oil and commercial oil. The samples were scanned using a Skyscan-1172 microtomographic system. All phases present in the sample were differentiated. The values of injected fluids were determined through 2D and 3D analyses. Two types of solutions were used, one doped with KI, and the other with $BaCl_2{\cdot}2H_2O$. The percentage of KI used allowed the individualization of the solution and, therefore, the direct quantification of this phase through 2D and 3D images.

1. Introduction

Several daily activities involve the accumulation or percolation of fluids through porous media. The utilization of oil filters in industrial processes, the application of pesticides and fertilizers to different agricultural crops, oil recovery in oil wells or even the remediation of soils affected by contaminants are some of these activities. In all such cases, the behavior of the fluids dividing the porous space of the material influences the processes of percolation of the fluid of interest (Wildenschild et al., 2004). There is a general need to predict the characteristics of fluid flow in porous media. Thus, different techniques are used to study the characteristics of the porous medium and the fluid in question. Mercury porosimetry (Zeng et al., 2012) and electron microscopy (Trejo et al., 2009) are two techniques widely used in this research field.

X-ray microtomographic imaging is a non-invasive and non-destructive technique capable of providing images of the internal microstructure of numerous materials evidencing the different phases present in the sample directly at the pore-scale (Kumar et al., 2008; Moreira et al., 2012). This methodology is based on the inhomogeneous absorption of a transmitted X-ray beam in a given sample direction. This generates a map of linear attenuation coefficients computationally converted into a digital image called projection. A reconstruction algorithm uses hundreds of these projections, each one taken at a given direction of the sample, to generate 2D images transverse to the scanned volume. The 2D images can also be used for 3D reconstruction of part or all of the volume scanned (Kak and Slaney, 1999). This technique does not require any sample preparation and has accompanied the development of computational models for fluid percolation (Kohout et al., 2006; Porter et al., 2009; Andrew et al., 2014). The 3D images of the pore structure via microtomography can be used to predict fluid flow properties (Landry et al., 2011). This work aimed to identify the optimal treatment and scanning conditions so as to distinguish the different solid, liquid or gaseous phases, and quantify the volume of fluid injected into samples consisting of glass beads.

2. Materials and methods

Three laboratories provided their devices for the experiments: the Petrobrás Research Center (CENPES/PETROBRAS), Rio de Janeiro, RJ, the Laboratory for Analysis of Minerals and Rocks (LAMIR), Federal University of Paraná (UFPR), Curitiba, PR, and the Laboratory of Analysis by X-ray Techniques (LARX), State University of Londrina, Londrina, PR. The three microtomography scanners correspond to the Skyscan model 1172. This device works with

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Table 1
Individual characteristics that differentiate the three Skyscan 1172 microtomographs used.

Equipment	CCD camera (Mpixel)	Aluminum filter (mm)
CENPES	10	1.0
LAMIR	11	0.5
LARX	11	0.5

Table 2
Solutions used in the experiments.

Nomenclature	H_2O (%)	NaCl (%)	KI (%)	$BaCl_2{\cdot}2H_2O$ (%)
Solution 1	70.0	17.0	13.0	–
Solution 2	72.0	18.0	10.0	–
Solution 3	64.0	16.0	20.0	–
Solution 4	73.3	13.3	–	13.3

micro-focus X-ray tubes (5 μm spot size) operating at applied tension from 20 to 100 kV with maximum power of 10 W. Its detector is a CCD camera. This set can differentiate between details smaller than 1 μm (Skyscan 1172, 2005). Although they belong to the same general model, one of the scanners differs from the others by the model of its CCD camera and by the thickness of one of its filters, aluminum. Table 1 shows the differences between all three devices.

A sample of nylon threads was used to test the methodology. A complete description of this experiment and the achieved results can be seen in Marques et al. (2011). The samples were prepared with 0.6 mm or 0.8 mm diameter glass beads inserted in a glass tube with an inner diameter of 6.7 mm and 1 mm wall thickness. The glass beads were forced to accommodate in the interior of the tube walls by rubber gaskets that sealed both ends of the sample.

Solution and/or oil were injected with a syringe through the bottom of the tube and air was left free to exit through a needle inserted into the rubber seal from the top of the tube. The injection was discontinued when the pore space of sample was visually saturated with the solution. Table 2 shows the compositions of each type of solution tested for the experiments. In the graph shown in Fig. 1, it can be observed that solution 3 presents a linear attenuation coefficient more distinctive than all the others solutions and the main compound of the sample for all types of energy employed. The solution 2 has lower linear attenuation coefficient than the main component of the sample at energies above 90 keV. Therefore, it was not used. The solution 4 was used despite having linear attenuation coefficient very close to the main component of the sample for energies between 95 and 100 keV. The intention was to test a different dopant from that used in other three solutions.

Two types of oil were used in the experiments: commercial motor oil (LUBRAX SJ (BR) SAE 20W50 – Petrobras Distribuidora SA) and industrial oil (supplied by CENPES) with 0.97 g/cm^3 density.

The analyses of microstructural characterization entailed the segmentation of the 2D grayscale images obtained from the experiments. This method consists in selecting a threshold point for the grayscale image from the histogram. The objective of this procedure is to binarize the image to separate the phase being analyzed (Moreira et al., 2012; Sezgin and Sankur, 2004; Saetre and Tjugum, 2014). Qualitative results are shown for the EV1 and EV2 samples and qualitative and quantitative results for the EV3.

Some specifics kinds of softwares were used. The "NRecon" reconstruct the 2D images based on acquired projections. This software uses the most widely reconstruction technique called filtered backprojection (Ketcham and Carlson, 2001). For analysis two softwares were employed: the "Imago" helped in choosing the appropriate gray level for image segmentation; the gray level is applied to the "CTan" for results obtained from the 2D images and/or volume (3D). The software "CTAn" was also used to generate 3D images displayed on "CTvol" software. In addition of these, acquisition software was used. Among the mentioned software, only the "Imago" is not part of Skyscan pack. This one was developed at the Laboratory of Porous Media and Thermophysical Properties (LMPT), Department of Mechanical Engineering, Federal University of Santa Catarina in association with Engineering Simulation and Scientific Software (ESSS).

3. Results and discussions

3.1. EV1 sample

This sample was measured under three different conditions: dry, containing solution 1, i.e., the same solution used in the EV2

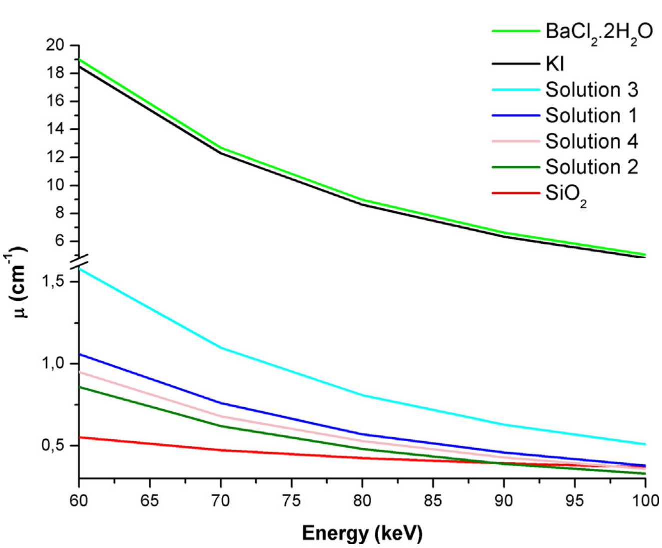

Fig. 1. Linear attenuation coefficients (μ) of the solutions, of the main compound of the sample and of the dopants used versus energy.

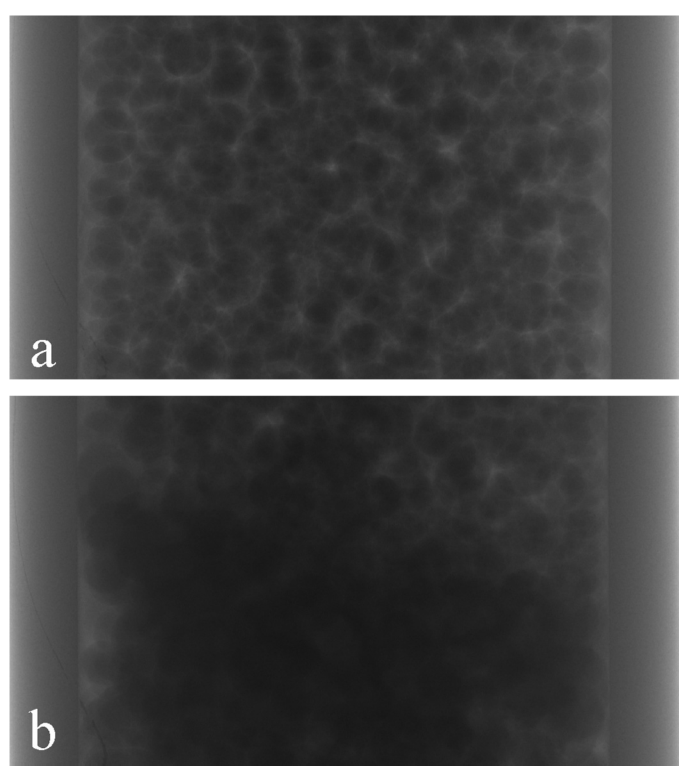

Fig. 2. Projections obtained for the EV2sample (a) dry and (b) containing fluids.