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Micro-structural information of porous materials by optical coherence tomography

S.L. Campello [a,*], W.P. dos Santos [b], V.F. Machado [c], C.C.B.O. Mota [d], A.S.L. Gomes [e], R.E. de Souza [e,*]

[a] Núcleo de Formação Docente, Universidade Federal de Pernambuco, 55014-900 Recife, PE, Brazil
[b] Departamento de Engenharia Biomédica, Universidade Federal de Pernambuco, 50670-901 Recife, PE, Brazil
[c] Cenpes, Cidade Universitária, 21941-915 Rio de Janeiro, RJ, Brazil
[d] Graduate Program in Dentistry, Universidade Federal de Pernambuco, 50670-901 Recife, PE, Brazil
[e] Departamento de Física, Universidade Federal de Pernambuco, 50670-901 Recife, PE, Brazil

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ABSTRACT

In this study we describe the use of optical coherence tomography (OCT) to reveal microstructures and damages in porous media. An approach for establishing pore size and pore size distribution based upon OCT was developed. This approach was tested with oil source rock samples. Several sedimentary rock samples from Brazilian oil fields with porosity values ranging between 15% and 28% were evaluated, and the results agreed quite well with the traditional pycnometry method. In addition, pore size distribution for the samples in three dimensional planes were obtained. Finally, the experimental results proved that the OCT images with a suited digital post-processing can be used to measure pore size distribution in natural and artificial materials, with the advantage of being non-invasive, faster and much less expensive than other presently available methods.

1. Introduction

Optical coherence tomography (OCT), introduced by Huang et al [1], is a technique based on interference phenomena using broadband light sources or frequency swept optical sources. A back-reflected reference light beam and a backscattered light beam from the sample are combined to produce a low coherence interferometric pattern data, which are mathematically processed to generate the OCT image. This technique enables the generation of in situ 2D and 3D images of transparent and nontransparent samples [2] with micro-scale resolution. The main characteristics of this technique that have made OCT images successful are: low cost, contact-free, and non-invasive character.

Due to these characteristics, OCT technique has been mainly used in biomedicine. First applications of OCT were in Ophthalmology, focused on retinal disease [3]. In order to get OCT images from nontransparent tissues, the optical window was moved from 800 nm to 1300 nm, allowing *in vivo* diagnostics of other part of human body [4–6]. For a recent review on OCT and its multidisciplinary applications, see Ref [7].

Since the technique allows the visualization of microstructure within highly scattering media, several applications outside biomedical field have started to come out. First applications of OCT were in ceramic materials research, such as void structures detection as a function of depth in silicon nitride ceramics [8]. Polymer sheets and polymer based coatings were already studied using OCT techniques [9]. OCT experiments were also performed on paper, which is a highly heterogeneous system that contains fibers, fiber fragments and additives. This structure imposes depth limits that depend on paper composition. Several works presented characterization of paper surface and bulk structure [10,11].

The results from the works mentioned above suggest a new non-biomedical application of OCT for characterization of porous media. Among the most important technological porous materials are rocks from oil fields. The modern oil industry needs increasingly accurate information about the collected oil source rock samples. In order to assess the cost/benefit ratio of a given oil field before beginning the exploration, an important parameter to be considered is the available oil volume that can be extracted. An important part of information needed to estimate this volume is the pore size distribution. However, most of the techniques for pore size distribution measurements are invasive – and therefore destructive, expensive, relatively time-consuming, and/or do not have enough resolution.

In this work, it is presented the application of OCT to microstructure study in porous media. As reported in applications for paper studies [9,10], a judicious digital image processing including filtering methods is quite important to get quantitative results. The images were obtained using a commercial OCT system and 13

* Corresponding authors.
E-mail address: slcampello@df.ufpe.br (S.L. Campello).

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samples of rock from Brazilian oil fields were analyzed. The obtained results indicate that application of OCT images to obtain pore structure and pore size distribution in samples is feasible, opening up possibilities as an affordable and fast evaluation method.

2. Materials and methods

2.1. Samples

The porous media used in this investigation are rock specimens and were furnished by PETROBRAS (Petroleum Brazilian State Company), which were sampled from several different field locations. A group of 13 cylindrical samples were approximately 50 mm long and 40 mm in diameter. They were classified into 4 subgroups according to geographic location. The samples were washed several times to ensure that there was no oil or salt content. Fig. 1 shows some samples and Table 1 presents sample distribution in subgroups and their respective porosity (Φ_{pyc}) obtained by helium gas expansion (pycnometry) in a CoreLab UltraPore Porosimeter. The last column shows the $\Phi_{pyc} \pm \Delta\Phi_{pyc}$ values.

2.2. OCT instrumentation

An OCT system OCP930SR model from THORLABS was used. The system is multipurpose, although mainly designed for biological applications like *in vivo* human skin images. It operates at 930 nm and with a spectral width of 30 nm, an output power of 2 mW and axial resolution of 6 μm. This system combines a broadband light source with a high-speed spectrometer to perform a Fourier domain detection of the OCT interference fringe signals. The base unit includes the broad super luminescent diode (SLD) light source, the spectrometer, analog and digital timing circuitry, and drive electronics for the galvanometer scanner within the probe. A PC controls data measurement, collection, processing, displaying and managing OCT image files. The images were saved as numeric array matrix and composed by 2000 columns and 512 lines, providing a pixel resolution of 3 μm × 3.1 μm. Each cross-sectional image is a "B-scan" composed of several "A-scans" (columns).

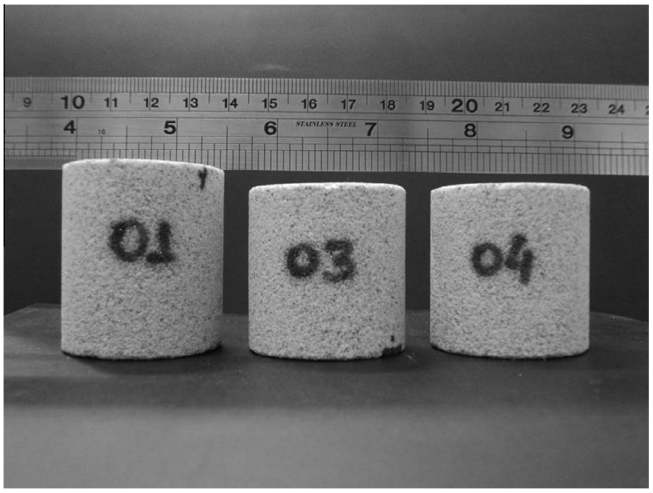

Fig. 1. Samples of the subgroup 1.

2.3. OCT method description

For each sample at least 100 B-scans were obtained from the circular base of all samples separated by 50 μm. Since samples are strongly scattering porous media, two complementary algorithms of image processing were developed in order to obtain quantitative information from OCT images. The aim of the image processing is to obtain a binary image, which contains structural information about porosity and pore size distribution. Porosity is defined as the ratio $\Phi = V_P/V$, where V_P and V are the porous volume and total volume of the sample, respectively. Void areas in OCT images of our samples are considered porous space. For a two dimensional version one can write porosity as:

$$\Phi = A_v/A \quad (1)$$

where A_V and A are the void and total area of the image, respectively. In this case, area is measured in number of pixels of the OCT image.

The image processing is divided in two parts. Initially, the first algorithm determines the optimized threshold value for a given porosity. Then, all images are binarized with the optimal threshold value and the pore size distribution is obtained in the second algorithm.

Fig. 2 shows an OCT image without any image processing. One can easily see the speckle noise effects and the signal decay through depth (lines). In general, OCT images of rocks present speckle noise. The first algorithm is started with the original image and step A is to filter out as good as possible the noise. Due to light absorption and scattering, light intensity decreases as the light penetrates into the rock sample. In order to clarify this statement, Fig. 3 shows the averaged OCT signal intensity decay for image as

Table 1
Subgroups of samples, individual porosity values Φ_{pyc} obtained by helium gas porosimeter, values with uncertainty $\Delta\Phi_{pyc}$. After image processing, sample average threshold values ($\bar{t}$) and respective standard deviation (σ_t), porosity ($\bar{\Phi}_{OCT}$) and standard deviation values ($\Delta\Phi_{OCT}$) were obtained.

Subgroup number (dominant rock color)/ Sample code		Φ_{pyc} (%)	$\Phi_{pyc} \pm \Delta\Phi_{pyc}$ (%)	$\bar{t}$	σ_t	$\bar{\Phi}_{OCT}$	σ_Φ	$\bar{\Phi}_{OCT} \pm \Delta\Phi_{OCT}$ (%)
1 (gray)	01	22.15	22 ± 1	0.3594	0.02	0.2219	0.04	22 ± 4
	03	21.08	21 ± 2	0.3556	0.03	0.2097	0.04	21 ± 4
	04	22.11	22 ± 2	0.3592	0.03	0.2242	0.05	22 ± 4
2 (gray)	4-5B	23.48	23 ± 2	0.3356	0.02	0.2380	0.04	24 ± 4
	6-2B	22.11	22 ± 2	0.3493	0.02	0.2235	0.03	22 ± 3
	10-2B	21.61	22 ± 2	0.3703	0.03	0.2189	0.05	22 ± 5
3 (red)	R266	17.46	17 ± 2	0.3994	0.02	0.1745	0.03	17 ± 3
	R267	19.21	19 ± 1	0.4209	0.03	0.1949	0.04	19 ± 4
	R268	17.64	18 ± 2	0.4120	0.02	0.1768	0.03	18 ± 3
4 (red)	L2	17.13	17 ± 1	0.3896	0.03	0.1727	0.03	17 ± 3
	L3	17.01	17 ± 1	0.4137	0.03	0.1707	0.03	17 ± 3
	L4	16.90	17 ± 1	0.4238	0.02	0.1701	0.03	17 ± 3
	L5	16.49	16 ± 1	0.3940	0.03	0.1685	0.05	17 ± 5