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Pore size distribution characterization in carbonates by correlating NMR, MICP and micro-CT data

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Abstract. From laboratory tests on pre-salt plugs, it is possible to obtain information such as pore body and pore throat size distributions, that may indicate the storage capacity and potential for extraction of fluids from a reservoir. This paper correlates data from NMR, MICP and micro-CT tests in order to characterize the pore body to pore throat ratios (BTR) for coquinas from Morro do Chaves Formation, in the Sergipe-Alagoas Basin. MICP data was converted to pore throat size distributions using Washburn equation and compared with pore body sizes computed from micro-CT data analyzed with image processing software. The comparison between MICP and micro-CT data yields the pore body to throat ratios, which are known to vary considerably in carbonates due to the heterogeneity of the pore structure. Next, the NMR T2 distributions were scaled to pore size distributions using the surface relaxivity parameter (ρ_e) and compared to both MICP pore throat size distributions and micro-CT pore body size distributions. The results indicate a good correlation between MICP pore throat distributions and T2 distributions, but when comparing T2 distributions with micro-CT distributions, allowance must be made for the low resolution limit of the micro-CT (20 microns) and the effect of bulk relaxation on larger pores.

Keywords: Carbonates, micro-CT, Pore distribution.

1 Introduction

Since pre-salt reservoirs were discovered, several studies have been developed in order to comprehend and characterize the porous structure of the carbonate rocks that compose them. Due to the high complexity and heterogeneity of carbonates, information such as pore size, pore shape and connectivity are fundamental for the petrophysical characterization, reservoir simulation and understanding of the fluid flow dynamics in the porous media.

This information can be obtained through laboratory tests performed on reservoir rock plugs. Data from tests such as Nuclear Magnetic Resonance (NMR) and Mercury Injection Capillary Pressure (MICP) helps to understand how pores are distributed and connected inside porous medium. In addition to these tests, the images and data from X-ray Computed Microtomography (micro-CT) give us an indication of heterogeneity inside plugs.

This work aims to correlate NMR, MICP and micro-CT data obtained in tests performed in coquinas from Morro do Chaves formation, in the Sergipe-Alagoas basin, in order to characterize the pore body to pore throat ratio (BTR) and pore distribution of these plugs to understand how pores are connected and their fluid flow capability. All laboratory data used in this study were made available by the Sedimentary Geology Laboratory (Lagesed), located in the Federal University of Rio de Janeiro (UFRJ).

In this paper, we will present a brief theoretical review about the tests involved in the data estimation, and a brief discussion about pore body to pore throat ratio. In addition, we present the methodology used and the discussion about the results obtained.

2 Theoretical review

In this section, we will present some concepts about carbonate reservoir, MICP, NMR, micro-CT, and pore body to pore throat ratio.

2.1 Carbonate reservoirs

Carbonate reservoirs are mostly constituted by limestones and dolomites. According to Rosa et. al. [1], pores of carbonated rocks are larger than in sandstones, with secondary porosity being dominant in a reservoir of this type, mainly due to the dolomitization process – which is the replacement of calcium ion, in calcium carbonate, by magnesium ion, creating calcium magnesium carbonate (dolomite). As stated by Peters [2], this new component has a smaller size due to ion replacement, resulting in a shrinkage of 12 to 13% in the grain volume and, consequently, increased porosity.

Carbonates are more heterogeneous due to the wide variation in their porous structure, resulting from the complex nature of their components and the diagenetic process. In these reservoirs, porosity and permeability are mainly controlled by fines and diagenesis.

Carbonate rocks are the main constituents of pre-salt reservoirs. The high degree of heterogeneity of these rocks increases the complexity during petrophysical analysis carried out in laboratory to characterize the reservoir.

2.2 Mercury Injection Capillary Pressure - MICP

MICP measures the volume accessed through a specific pore throat. According to Torsaeter and Abtahi [3], this procedure consists of injecting mercury in increments into a small rock fragment, placed in a holder cell, at increasing pressure levels. As the pressure increase, the injected fluid invades pores with increasingly smaller radii. The mercury injected is estimated as a percentage of pore volume and related to pressure.

According to Washburn [4], if a porous medium behaves as an assemblage of very small cylindrical capillaries, it is possible to calculate the pore throat radii that the fluid was able to access correlating with capillary pressure, as in eq. (1):

$$P_C = \frac{0.29\sigma cos\theta}{r_t}. \tag{1}$$

where P_c is capillary pressure (psi), σ is the mercury superficial tension (480 dyne/cm), Θ is the Hg-air contact angle (140º) and r_t is the pore throat radii (μm).

After calculating r_t, it is possible to obtain a pore-throat size distribution by incremental mercury saturation curve for the rock, in order to understand its heterogeneity and connectivity.

2.3 Nuclear Magnetic Resonance – NMR

NMR is applied in petrophysics to investigate the porous space in reservoir rock plugs saturated with fluids and consists of registering magnetic responses from atomic nuclei exposed to external magnetic fields.

Considering a rock plug filled with water, a magnetic field is applied and the spins axis of the protons in the hydrogen atoms align. When a second magnetic field is applied, the spins tip away from this equilibrium position, and once this field is removed, they begin tipping back to their original position. This process is called relaxing, and the longitudinal (T_1) and transverse relaxation time (T_2) measure how fast the spins go back to their initial positions, in msec.

According to Lowden and Porter [5], once NMR measures the amount of hydrogen contained in the rock, the amplitude of NMR signal is proportional to the fluid occupying the pores. The surface relaxivity (ρ_2) can be dominated by relaxation at all sites of the surface or by relaxation associated with paramagnetic ions in the surface.

Brownstein & Tarr [6] demonstrated that NMR relaxation time is proportional to pore size, as in eq. (2):

$$T_2 = \rho_2 \left(\frac{S}{V}\right)_{pore}. \quad (2)$$

where *S* is the pore surface and *V* is the pore volume.

According to Marschall [7], for a same sample, the pore-throat distribution from MICP could be related with the pore-body distribution from NMR through a "scaling factor", which he called the effective relaxivity, ρe (μm/s). It can be obtained adjusting both curves using eq. (3) and considering the pore space as an assemblage of very small cylindrical capillaries:

$$T_2 = \frac{1000r}{3\rho_e}. \quad (3)$$

The NMR-MICP correlation provides an opportunity to identify which part of the pore system is constituted by pore body and which is by pore throat [8].

2.4 X-ray Computed Microtomography – micro-CT

In petrophysics, the micro-CT is a non-destructive technique widely used to visualize the internal structure of rocks. According to Machado [9] and Silva [10], the images are continuously and sequentially taken, then are compiled to create 3D illustrations that are digitally processed in order to obtain morphologic and geometrical parameters. All process is divided in three steps: acquisition, image reconstruction and visualization.

Micro-CT equipment can take images with resolutions in the order of microns. Generally, they are constituted by a fixed X-ray tube and a radiation detector. The sample is rotated during the acquisition prosses [9]. The reconstruction process consists of uniting all projections obtained during acquisition to produce the 3D object which is then divided in slices that are used in the investigation of the porous space.

2.5 Pore-body to pore-throat ratio

Pore-body to pore-throat ratio can indicate the connectivity in a porous medium. According to Andersson [11][12], a large difference in size between the radius of a pore-body and a connected pore-throat can result in capillary trapping, which is the immobilization of non-wetting phase clusters into the pore space [13]. Thus, the study of pore-body to pore-throat ratio can give us an indication of how much fluid can be left in the reservoir during its production.

For Muller-Huber [14], in carbonate rocks, pore body to pore throat correlation often fails mainly when in the presence of large pores associated with secondary porosity, where connectivity among pores is not always related with pore size. Due to their high heterogeneity, where vugs, faults or even fractures can constitute the porous medium, por-body to pore-throat in carbonates can achieve high values, leading to a miss-interpretation about the porous system and fluid flow.

This work investigates the pore distribution and pore-body to pore-throat ratio in coquinas with similar characteristics to pre-salt carbonates, in order to understand their connectivity and fluid flow capability.

3 Methodology

In this study, data from MCIP, NMR and micro-CT tests were used to characterize pore distribution and pore-body to pore-throat ratio (BTR) of coquinas from Morro do Chaves Formation, of the Sergipe-Alagoas Basin. The tests were performed in five plugs generated from outcrop samples and belonging to the Sedimentary Geology Laboratory (Lagesed), of the UFRJ, which also made the results available so that this study could be carried out. The plugs and test data were also used by Silva [10] in her master's dissertation in which geological and petrophysical interpretation were also presented. In this paper will be presented the results for three plugs, shown in Fig. 1.

Gas Porosity and permeability were obtained using the equipment *AP-608 automated Porosimeter-Permeamer, Coretest Systems.* For MICP tests, rock fragments were removed from the samples and placed in the holder cell of the equipment. The pixels size used for the micro-CT image acquisition of each plug is presented in

Tab. 1. For NMR tests, all plugs were saturated with distilled water with an applied isostatic pressure of 1,000 psi.

For characterization, MICP data was converted to pore throat size distributions using eq. (1) and pore body sizes were computed from micro-CT data analyzed with image processing software. Next, the NMR T2 distributions were scaled to pore size distributions using the surface relaxivity parameter (eq. 3) and an assumed BTR value of 10. The pore size distributions from NMR were then compared to both the MICP pore size distributions and the micro-CT pore body size distributions. Also, BTR's values were calculated through NMR-micro-CT and MICP-micro-CT pore distribution correlation.

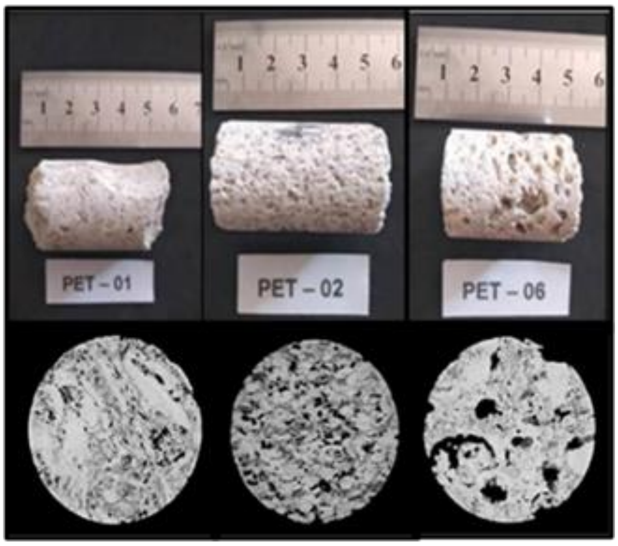

Figure 1. Picture of the plugs PET-01, PET-02 and PET-06 [10].

Table 1. Micro-CT pixel size for each plug [10].

Plug name	Diameter (in)	Length (in)	Pixel size (μm)
PET-01	1.02	1.73	19.0
PET-02	0.98	1.77	20.4
PET-06	0.99	1.59	19.2

4 Results

In this paper, we present the results for plugs: PET-01, PET-02 and PET-06. In general, Silva [10] classified the plugs as grainstones with fragmentation and cementation. The porosity types identified were mainly intra and inter particle. The visible pores in PET-06 appear larger than in PET-01 and PET-02 as seen in Figure 1. Effective porosity, absolute permeability (k_{abs}) and grain density are introduced in Tab. 2.

Table 2. Effective porosity and absolute permeability for each plug [10].

Plug name	Por (%)	k_{abs} (mD)	Grain Density (g/cm^3)
PET-01	12	18.6	2.70
PET-02	15	27.9	2.69
PET-06	19.5	282	2.70

Marzouk et. al. (1995) [15] pore size classification is adopted in this study: pores with diameter less than 0.6 μm are classified as micro pores, diameters between 0.8 and 8 μm are considered as meso pores and pore diameters bigger than 8 μm are classified as macro pores.

Figure 2 presents the pore throat size distribution from MICP according to this classification. From these results, it is possible to notice that all plugs are mainly constituted by micro and meso pores. As Silva [10] identified intra-particle porosity in theses plugs, according to Schuab [16] the micropores can be related to it. For the meso porosity, the author considers that it can be related with the connections between micro and macro pores of the bimodal grainstones. The comparison between intrusion and extrusion data can give an idea about the pore-body to pore-throat ratio for MICP once the extrusion curve indicates the micro and meso pores.

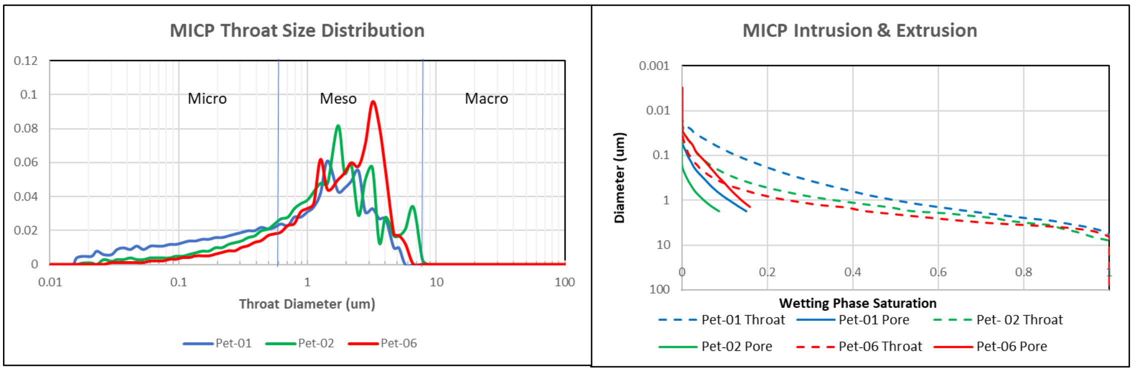

Figure 2. Pore throat size distribution from MICP on left and Intrusion and Extrusion curves on right.

A pore size distribution from NMR was estimated using eq. 2 and ρ_2 = 3 μm/s and assuming a BTR of 10 based on the comparisons of the MICP intrusion and extrusion curves in Fig. 2, which is consistent with the findings of Song [8] for typical BTR values in carbonates and Allen [17] for typical surface relaxivity values in carbonates.

Figure 3 presents the NMR T_2 distribution and pore size distribution for the three samples analyzed in this paper. From the T_2 relaxation time is possible to notice a dispersed distribution for the three samples. This can be explained by the fact that carbonate rocks are constituted by different rock types, pore size and pore interconnection types [14]. In addition to that, compared to MICP, NMR identified mainly meso and macro pores. This can be explained because the mercury injection tests used small rock fragments that do not contain the large macro pores, while the NMR measures the core plugs with the large macro pores shown in Fig. 1.

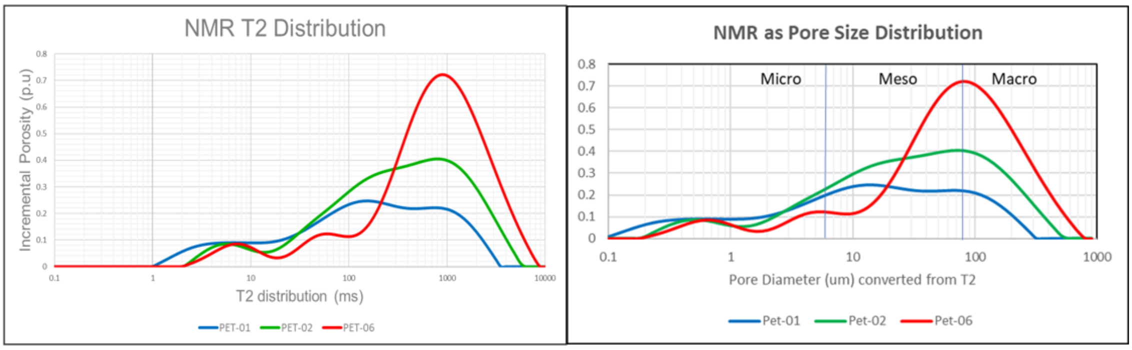

Figure 3. Pore size distribution from converted NMR data.

An NMR-MICP-micro-CT correlation was then performed in order to obtain the effective relaxivity and understand the relationship between pore-throat and pore-body distribution. Figure 4 presents in green the NMR pore size distribution, in blue the MICP pore size distribution which assumed a BTR of 10 and in red the pore size distribution from the micro-CT analysis. Table 3 introduces the ρe (μm/s) estimated by moving one curve over the other until their peaks match. From these results it is possible to notice a good relationship between NMR, MICP and uCT for PET-01 and PET-02, while for PET-06 this proportionality is poorer in the macro region. This indicates the limits for using NMR for identifying the largest pore sizes accurately as the effect of bulk relaxation and diffusive pore coupling becomes more significant, as noted by Lowden and Porter [5]. New enhancements in micro-CT acquisition which will improve the resolution of the micro-CT images to 3 microns which will allow the resolution of not only macro pores but also the meso porosity and a significant amount of micro porosity. This will enable greater overlap with the MICP results and lead to a more accurate comparison with NMR data for determining BTR in carbonates and how it may vary according to rock types.

Comparing NMR and MICP distribution, it is possible to notice that the peak in MICP curve is narrower than the NMR spectrum for the same sample. According to Song [8], this indicates that there are distinct pore throats in the pore space. The MICP pore throat distribution correlates with the micro to the meso pores indicated by NMR while the micro-CT pore distribution region indicates that the macro-pore body portion of the pore network (large pores) are connected via the pore throats associated with the meso pores [8].

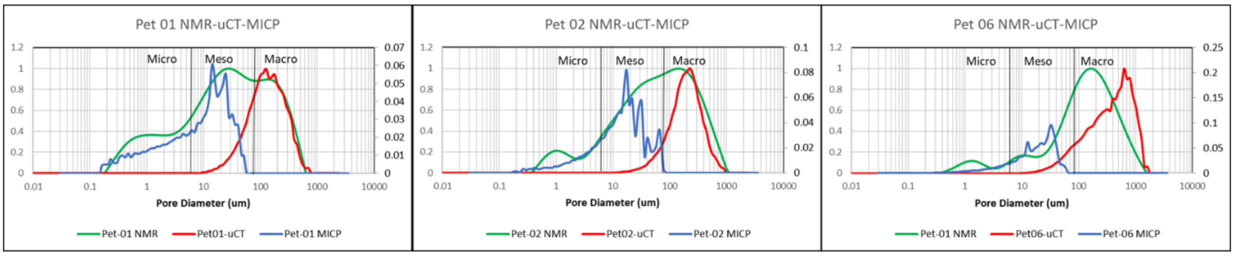

Figure 4. NMR-MICP- correlation.

Table 3. Effective relaxivity from NMR-MICP correlation

Plug name	*ρe* (μm/s)
PET-01	3
PET-02	3
PET-06	3

5 Conclusions

We have demonstrated an NMR-MICP-micro-CT correlation for complex carbonate samples from Morro do Chaves Formation, in Sergipe-Alagoas Basin, which are considered analogous to the pre-salt carbonates rocks, in attempt to quantify the pore sizes, the pore throats and the relationship between pore body and pore throats. The NMR T2 distribution is assumed to primarily represent pore size, MICP is assumed to primarily respond to pore throats and the micro-CT analysis presented here with a 20 micron resolution is mainly quantifying the macro pores.

The MICP pore throat distributions indicated that the three plugs are constituted by micro and meso porosity, while the NMR pore size distributions indicated the presence of mainly meso and macro pores. This can be explained because the mercury injection tests used small rock fragments that do not contain the large macro pores, while the NMR measures the core plugs with the large macro pores. Analyzing the NMR-MICP-micro-CT correlation identified a good relationship for PET-01 and PET-02 curves, while for PET-06 this proportionality is poorer in the macro region. This indicates the limits for using NMR for accurately identifying the largest pore sizes accurately as the effect of bulk relaxation and diffusive coupling becomes more significant. In addition, the correlation of the three distribution imply that the macro-pore body portion of the pore network (large pores) are connected via the throats associated with the meso pores.

New capabilities with micro-CT equipment can now lower the resolution limit to 3 microns which will be sufficient to evaluate the meso pores and even some of the micro porosity which will lead to greater accuracy for the methodology presented here. Additional studies of coquinas and other carbonates are being undertaken at LRAP to further develop this analysis for improved estimation of pore body to pore throat ratios and their impact on oil recovery in carbonate formations.

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MICP, NMR and micro-CT results were also used in her master's dissertation "Characterization of porous system of coquinas from Morro do Chaves Formation, Sergipe-Alagoas Basin using MICP, NMR and μCT".

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