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Structural Characterization of Rocks Using the X-ray Microtomography Technique

Caracterização Estrutural de Rochas Utilizando a Técnica de Microtomografia de Raios-X

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Abstract

In this article are presented the results obtained for the determination of mineral composition and petrophysical properties of eight sedimentary rock samples through a proposed new method, which is supported by X-ray microtomography image analysis (microCT). The results are compared with their corresponding ones obtained by traditional techniques in order to assess the efficiency of the new method. Three samples of carbonate rocks and five of sandstone were used in this study. Two sandstone samples come from the Rio do Peixe basin (BRP, at northeastern Brazil) while the rest were extracted from USA basins. The best results were obtained in the quantification of the mineral composition of the rocks and, consequently, in the estimation of grain density. Due to its insufficient image resolution, the microtomography was sometimes unable to quantify very small pores, giving low porosity values. The correlation between bulk densities measured by the two methods shows an intermediate efficiency. Although the results presented here indicate that the microCT image analysis is appropriate for quantification of mineral phases, this technique has potential for determination of many other physical properties, such as mechanical, electrical and fluid flow rock properties.

Keywords: *Rock density; Porosity; Mineralogy*

Resumo

Neste artigo são apresentados os resultados alcançados para a determinação da composição mineral e de propriedades petrofísicas de oito amostras de rochas sedimentares a partir de um novo método proposto, o qual é suportado pela análise de imagens de microtomografia de raios-X (microCT). Os resultados são comparados com seus correspondentes obtidos por técnicas tradicionais a fim de avaliar a eficiência do novo método. Foram utilizadas neste estudo amostras secas, sendo três de rochas carbonáticas e cinco de arenitos. Duas amostras de arenito são oriundas da Bacia do Rio do Peixe (BRP, no Nordeste Brasileiro) enquanto as demais foram extraídas de bacias dos EUA. Os melhores resultados foram obtidos na quantificação da composição mineral das rochas e, consequentemente, na estimativa da densidade de grãos. Devido à resolução insuficiente das imagens, a microtomografia foi algumas vezes incapaz de quantificar adequadamente poros muito pequenos, fornecendo valores baixos demais para a porosidade. A correlação entre a densidade total medida pelos dois métodos mostra uma eficiência mediana. Embora os resultados apresentados aqui indiquem que a análise de imagens de microCT é apropriada para a quantificação das fases minerais, essa técnica apresenta potencial para a determinação de muitas outras propriedades físicas, tais como propriedades mecânicas, elétricas e de fluxo de fluidos.

Palavras-chave: *Densidade de rochas; Porosidade; Composição mineral*

1 Introduction

Determining the mineral composition of rocks is important because this controls the majority of physical and chemical properties of the lithotypes, determining the own recognition of that rock and affecting every stage of your economic exploitation, as the mining method and mineral processing strategies. Several techniques are conventionally applied for recognition and quantification of minerals, especially the X-ray diffraction (XRD), X-ray fluorescence (XRF), scanning electron microscopy (SEM) and energy dispersive microscopy (EDS) (Chalmers *et al.*, 2012; Kubala-Kukus *et al.*, 2015; Dias *et al.*, 2017). Each of these techniques has advantages and limitations, and none of them presents final results to any situation, but they are complimentary. As an example of incompleteness and complementarity, the technique EDS offers good performance for the recognition of accessory elements while XRD does not quantify amorphous elements.

A more recent technique, which reconstitutes the three-dimensional architecture of the pore system and mineral phases that constitute a rock, is the X-ray microtomography (microCT). This technique gives a new approach to rock characterization. Several authors have presented many applications for the rock digital models generated from microCT data (Schmitt *et al.*, 2015; Archilla *et al.*, 2016; Soares *et al.*, 2018; Oliveira & Soares, 2018).

Reis-Neto *et al.* (2011) quantify the porosity, the pore connectivity degree and the mineral composition of four samples of rocks from X-ray microtomography data and digital photographs of thin sections. The samples analyzed were of marble, quartzite, sandstone and dolomitic breccia. The achieved results demonstrate the ability of micro-CT for characterization of porous distinct frameworks, enabling the qualitative and quantitative analysis of the shape, size, volume, distribution and connectivity of rock pores. Moreover, it is a tool that integrates in a 3D way petrophysical aspects to petrographic analysis. The possibility of detecting mineral phases with the technique allows a better understanding of the porous framework and three-dimensional observation of phase or sample region of most of the pores, aiding in the understanding of the rock genesis process.

Lopes *et al.* (2012) analyzed rock samples of stone pumice, granodiorite, protomilonite and a sandstone with deformation bands. They analyzed the porosity of each sample, quantifying the proportion of connected and isolated pores. In addition, quantify mineral phases that make up each sample with a 3D visualization of pore spatial distribution, assisting in the petrogenetical interpretation of the rock samples. The ductile and brittle microstructural analysis by micro-CT integrated with petrographic analysis provides significant advances in rocks that present contrasting X-ray attenuation minerals. Microstructures can be analyzed three-dimensionally by isolating mineral phases for individual analysis.

Palombo *et al.* (2015) estimated the porosity of four samples of rocks, being two of limestone and two of sandstone, from the analysis of digital images of X-ray microtomography. Three-dimensional visualization of pore network led the authors to conclusions concerning the form and spatial distribution of the pores, its degree of interconnection and the possible occurrence of a preferred direction for the pores network.

Kubala-Kukus *et al.* (2015) used several analytical techniques to solve the elemental composition of soils and geological samples. Among them, the X-ray microtomography was applied to estimate the mean value of grains size.

Samples of carbonate rocks (stromatolites and travertines) were analyzed by Coelho (2016) using digital images of X-ray microtomography. In that study were analyzed the porosity, pore size and pore roundness. Although the samples exhibit similar porosities, the structure, the size and shape of the pores are very different due to the genesis and the diagenesis processes of such rocks are also very different.

Dal Col *et al.* (2016) investigated the porosity and the representative elementary volume (REV) of a Rio do Rasto Formation sandstone sample through digital images of X-ray microtomography. They quantify the proportion of interconnected and isolated pores and defined the REV as 2.9 mm edge cube.

Gruia (2017) used the X-ray microtomography besides other related analytical techniques to investigate the mineral composition of three rock samples of a meteorite. It was determined that the X-ray micro tomography is a powerful technique to observe on extended volumes the morphology, the alignment, and the local bulk density in samples. In addition, it is proved that the tomography analysis provides a substantial new information about pore connectivity.

Sena & Soares (2017) demonstrate the potential of the X-ray microtomography technique to quantify mineral content of fifteen carbonate rock samples as well compare the achieved contents with those from XRD analysis and to evaluate the effect of the mineral composition on the petrophysical properties of those rock samples. The results show the content of major minerals controls the petrophysical properties, especially grain density and elastic velocities. However, other factors which were assessed through the μCT analysis also significantly affect the elastic velocities: porosity, type of porosity (if intergranular or vugular) and the proportion of microporosity present in the rock.

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The purpose of this article is to demonstrate the feasibility of X-ray microtomography images obtained in millimetric sedimentary rock samples for the quantification of their mineral components, as well as determine basic physical rock properties as grain density and porosity. The results obtained by the proposed method are calibrated by traditional methods such as X-ray diffractometric analysis (XRD), for mineral composition, and gas pycnometer test, for rock porosity and grain density measurements.

2 Methodology

Eight sedimentary rock samples are analyzed in this work, being five of sandstones and three of carbonate rocks. Two sandstone samples were extracted from an outcrop of the Rio do Peixe Basin, located in the extreme west of the Paraíba State, Brazil. The other three samples of sandstone, as well as all samples of carbonate rocks are from outcrops of sedimentary basins of the United States of America. Table 1 lists the samples analyzed with country of origin, basic petrophysical properties and their mineral composition as determined by XRD analysis.

The digital imaging of rock samples by X-ray microtomography was carried out in a Versa XRM-500 device with approximately 1,000 slices per sample. The resolution (pixel size), as well as the subsample volume analyzed of each sample are listed in Table 2.

Figure 1 presents a mosaic with a slice of each analyzed rock sample. It can show the level of heterogeneity, the texture and the possible presence of structures in each sample. The first three slices belong to samples of carbonate rocks, while others are of sandstone.

The AC12 sample consists of an oolitic/oncolitic limestone with a micritic matrix and presents predominance of intergranular/moldic macroporosity and a minor content of microporosity in the matrix. The IL320 sample is formed by an aggregate of fossil shells of larger dimensions compared to the microfossils present in the sample AC12. It shows growth of calcite crystals around and into the shells and a secondary presence of micrite. Macroporosity is predominant. The sample SD12 is composed basically by dolomite grains (note the diamond-shaped grains), with little micrite and the predominant presence of macropores. The grain size is on the same order of the grains of the AC12 sample.

Rock sample	Country of origin	Porosity (%)	Grain density (g/cc³)	Bulk density (g/cc³)	CAL (%)	DOL (%)	QTZ (%)	PLG (%)	C+M (%)	KFD (%)	LIM (%)	PYR (%)
AC12	USA	27.23	2.700	1.977	100	–	–	–	–	–	–	–
IL320	USA	17.67	2.690	2.210	100	–	–	–	–	–	–	–
SD12	USA	16.88	2.820	2.327	–	100	–	–	–	–	–	–
CGS15	USA	18.61	2.660	2.107	7	4	87	2	–	–	–	–
PSS2	USA	18.36	2.650	2.133	–	–	86	7	7	–	–	–
SCS1	USA	22.70	2.670	2.235	–	–	79	9	10	2	–	–
AM14V1	Brazil	17.40	2.596	2.145	–	–	28	–	71.4	–	0.4	0.2
AM14V2	Brazil	17.40	2.596	2.145	–	–	28	–	71.4	–	0.4	0.2

Caption: CAL = calcite, DOL = dolomite, QTZ = quartz, PLG = plagioclase, C+M = clay minerals and mica, KFD = K-feldspar, LIM = limonite, PYR = pyrite.

Table 1 Country of origin, porosity, grain density, bulk density and XRD mineral content of the analyzed rock samples.

Rock sample	Resolution (μm)	Subsample volume (μm³)
AC12	2.20	500^3
IL320	2.40	500^3
SD12	2.17	500^3
CGS15	2.65	500^3
PSS2	2.40	500^3
SCS1	2.40	500^3
AM14V1	4.00	500^3
AM14V2	3.00	600^3

Table 2 Resolution (pixel size) and subsample volume analyzed for each rock sample.

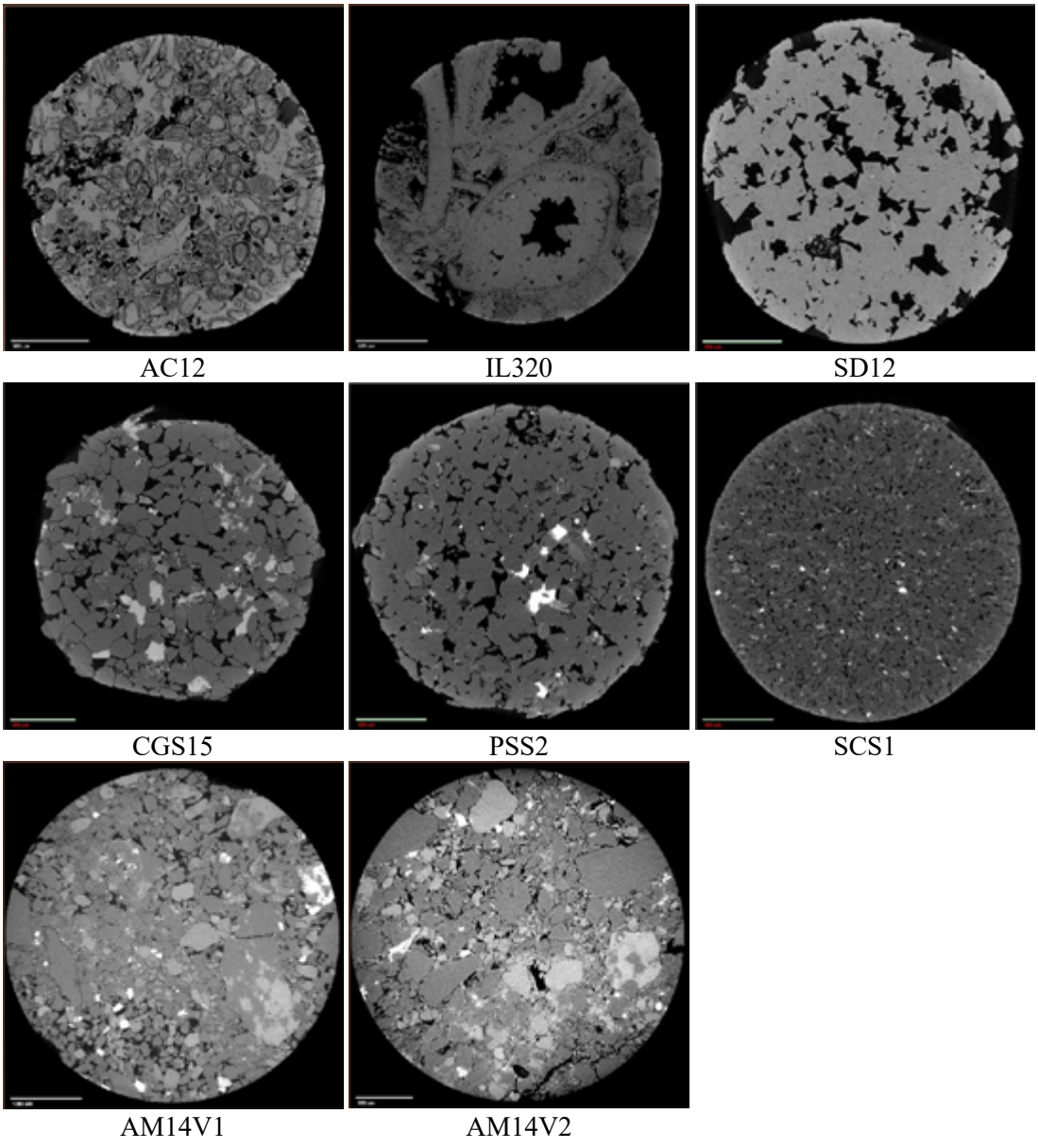

Figure 1 Mosaic with a central microCT slice of each analyzed rock sample.

Sandstones rock samples CGS15 and PSS2 are quite similar in terms of size and shape of grains and both presents predominance of intergranular macroporosity. The average grain size is on the order of 250 μm, which allows classify them as fine to medium sandstones according to the Wentworth scale. The SCS1 rock sample represents a sandstone with very fine grain size, with an average grain diameter on the order of 100 μm. The three sandstone samples from North American basins also appear to present a similar mineralogical composition. This can be stated by the predominance of a same gray level in them all.

The siliciclastic rock samples from the Rio do Peixe basin are very poorly selected, *i.e.*, there is a great variation in grain sizes. In general, one can classify these samples as sandstones of fine granulometry. It should be emphasized that, due to the variation in image resolution, the scale presented in slices of these samples also varies: to the AM14V1 sample the scale bar corresponds to 1000 μm, and to the AM14V2 sample it corresponds 500 μm. These samples were extracted from deformation bands of the Antenor Navarro Formation. Due to their reduced dimensions, it is not possible to identify the presence of

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structures (micro-cracks, grain alignment and laminates) in the samples AM14V1 and AM14V2. Note that these sandstone samples are composed of grains of different sizes immersed in a matrix of fine granulometry.

Avizo Fire software was used in the recognition of pores and mineral phases that constitute the samples analyzed from microCT images. Figure 2 shows the workflow applied for recognition, three-dimensional visualization and quantification of all constituent phases.

Initially a subvolume is extracted from the original images. This step aims to obtain a cubic subvolume centralized to prevent edge artifacts, and with dimensions suitable for the data processing. Then the distribution of gray tones is analyzed in all the slices of the subvolume aiming to identify areas with different shades of grey (constituent phases). For each phase extracts a region of a representative slice of that phase and builds a histogram to identify the range of gray tones characteristic of that phase.

After the definition of the grayscale intervals characteristic of all phases the Multi-Thresholding function is applied in order to turn grayscale in labels, which are quantified in terms of the number of pixels through the Material Statistics function. This processing step provides the volumetric fractions of each mineral phase and pores. Table 3 presents, as an example, the result of the application of Material Statistics function in the PSS2 rock sample. It contains the number of pixels, voxels, and the X, Y and Z coordinates of the center of the set of mineral grains or pores of each constituent phase.

Three-dimensional visualization of each phase can be obtained by applying the Interactive Thresholding, Generate Surface and Surface View functions. The function Interactive Thresholding generates binarized images in which a given phase is isolated, while the function Generate Surface generates a three-dimensional surface of that phase, and finally, the function Surface View allows its viewing. Figure 3 shows, as an example, the three-dimensional visualization of pores (green) and the white phase (in red, the densest mineral phase) for the PSS2 sample.

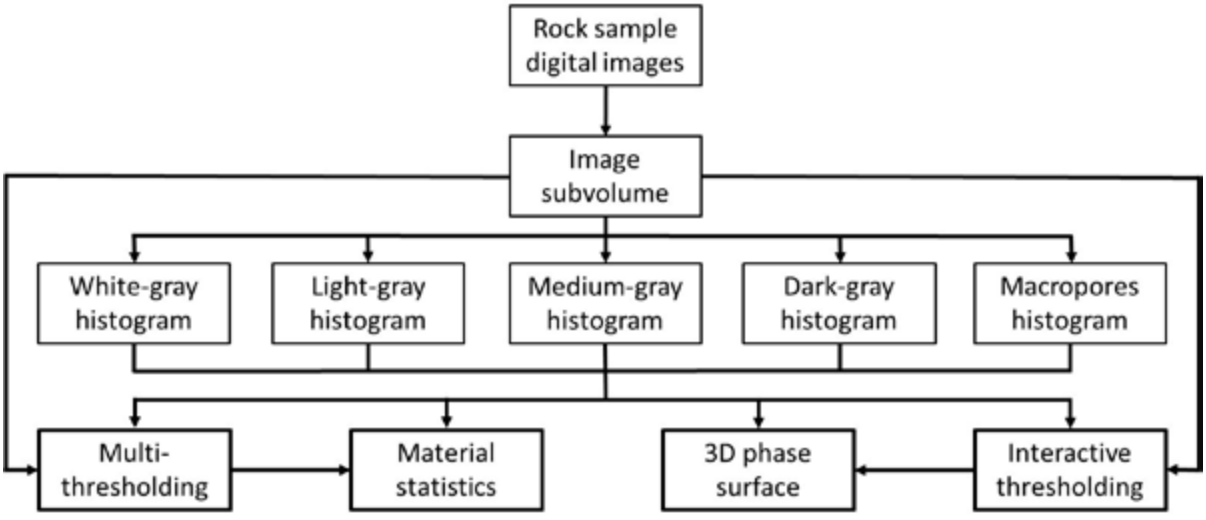

Figure 2 Workflow used in Avizo Fire software for mineral phase quantification and 3D visualization.

Ref. #	Material	Count	Volume	Center X	Center Y	Center Z
1	Pores	15712598	217210944	1153.340088	1163.078857	723.769897
2	Dark	20754516	286910432	1183.827759	1155.956421	737.50061
3	Medium	81334040	1124361728	1180.635376	1154.488525	709.635559
4	Light	6237862	86232200	1244.845947	1159.277466	756.248291
5	white	960982	13284615	1318.695923	1095.827515	766.219971

Table 3 Mineral and pore contents as quantified by the Material Statistics function.

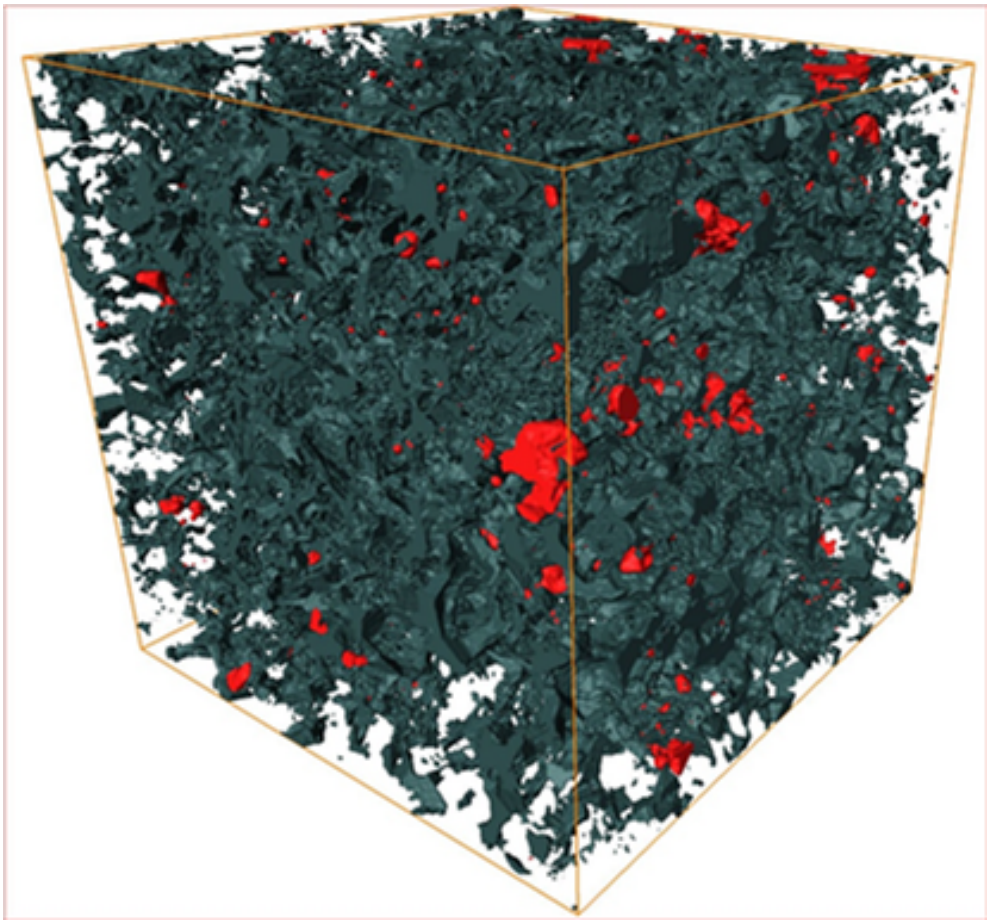

Figure 3 Visualization of the pores (green) and the densest mineral phase (pyrite, in red) for the PSS2 sample.

3 Results and Discussions

Following the microCT digital image analysis methodology, as shown in the workflow of Figure 2, the petrophysical properties (porosity, grain density and bulk density) and the mineral composition of the samples were quantified as presented in Table 4.

Figure 4 presents a comparative analysis between the levels of the mineral phases, for each sample, determined by XRD and microCT. For the three carbonate rock samples the mineral composition as determined by both methods match perfectly, both for limestone samples as for the dolomite one. Similarly the three sandstone samples from USA feature approximately equal mineral contents both when these are determined by XRD and by microCT. On the other hand, for both BRP sandstone samples the phase recognized by XRD as "clay + mica" seems it was recognized by microCT as feldspar. Note from the last two graphics on Figure 4 that the quartz content as analyzed through both techniques are reasonable alike, while the unique complimentary phase, which is labeled as "clay minerals and mica" by XRD, was recognized as feldspar by microCT analysis. Since the feldspar alteration results in kaolinite, that it is a clay mineral, it is acceptable that both are the same material.

Figure 5 shows a comparison between the values of porosity measured by gas pycnometer method and their corresponding values estimated by microCT. Should be noted the smaller porosity values estimated by microCT in comparison with the values measured by laboratory petrophysical tests, with exception mainly of the SD12 sample. This probably can be explained by insufficient digital image resolution for the recognition of micropores. A reason for the SD12 sample having greater porosity may be the fact that it has many closed pores. It is well known that laboratory petrophysical tests measure the effective porosity, that is, the gas must enter and out of the sample, so the pores must be opened and connected so that the gas percolate through it. MicroCT, on the other hand, measures total porosity regardless of whether the pores are open or closed.

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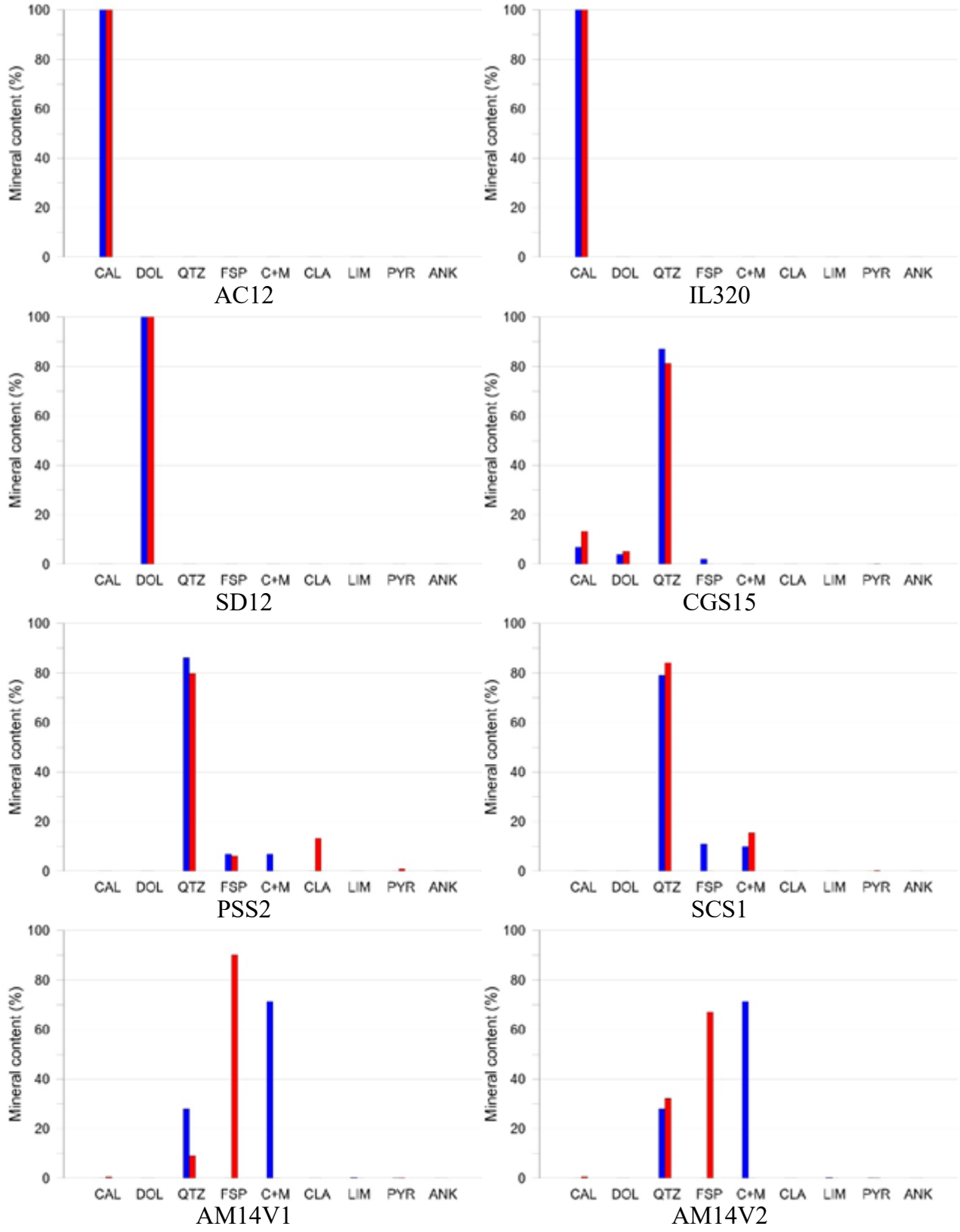

Figure 4 Comparison between the mineral phases contents as determined by XRD (blue bars) and by microCT (red bars).

Rock sample	Porosity (%)	Grain density (g/cc³)	Bulk density (g/cc³)	QTZ (%)	CAL (%)	FSP (%)	DOL (%)	CLA (%)	C+M (%)	PYR (%)	ANK (%)
AC12	19.2	2.710	1.972	-	100.0	-	-	-	-	-	-
IL320	17.5	2.710	2.231	-	100.0	-	-	-	-	-	-
SD12	25.2	2.850	2.369	-	-	-	99.9	-	-	-	0.1
CGS15	20.2	2.672	2.174	81.3	13.3	-	5.2	-	-	0.2	-
PSS2	12.6	2.672	2.182	79.7	-	6.1	-	13.2	-	1.0	-
SCS1	8.2	2.676	2.068	84.2	-	-	-	-	15.4	0.4	-
AM14V1	5.6	2.576	2.128	9.0	0.6	90.1	-	-	-	0.3	-
AM14V2	9.3	2.593	2.142	32.1	0.7	67.0	-	-	-	0.2	-

Caption: CAL = calcite, DOL = dolomite, QTZ = quartz, FSP = feldspar, C+M = clay minerals and mica, CLA = clay minerals, ANK = ankerite, PYR = pyrite.

Table 4 Porosity, grain density, bulk density and mineral content as analyzed through microCT.

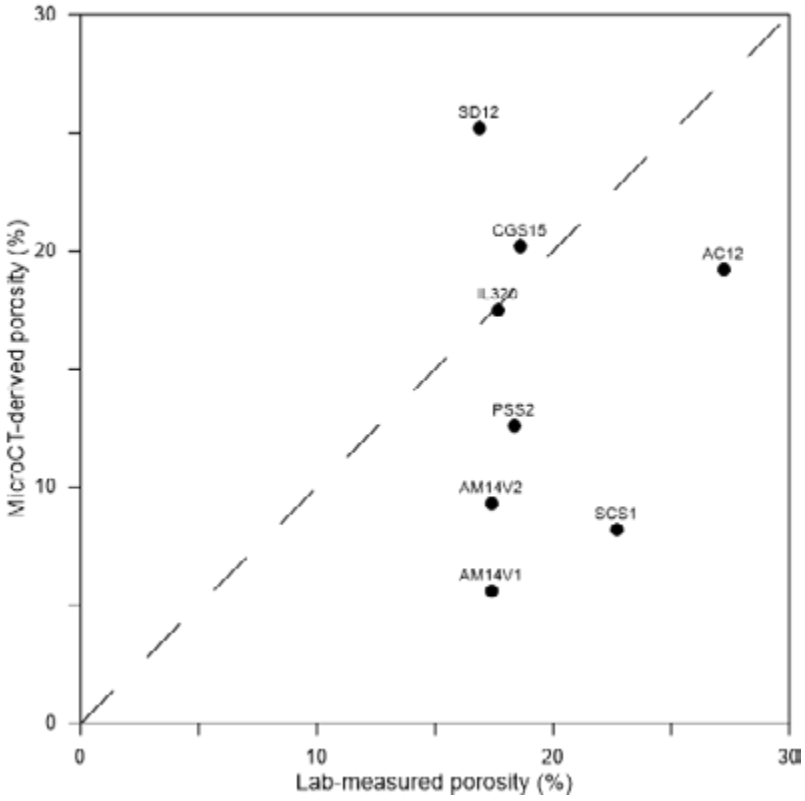

Figure 5 Comparison between the porosity values measured in the laboratory and those ones estimated by microCT.

Figure 6 confronts the grain density values measured in petrophysical tests with those estimated by microCT. There is an excellent correspondence between the values obtained by both methods. In the case of bulk density (Figure 7) there is also a very good match between the values provided by two methods, except for the SCS1 sample. It should be noted again that microCT porosity and grain density measurements are calibrated by gas pycnometer tests.

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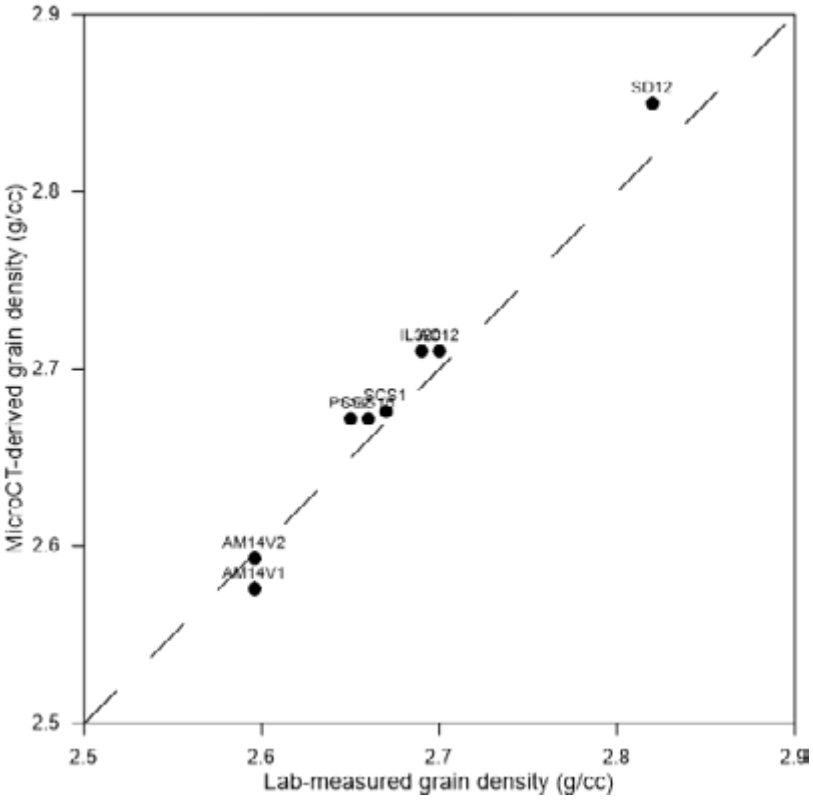

Figure 6 Comparison between the grain density values measured in the laboratory and those estimated by microCT.

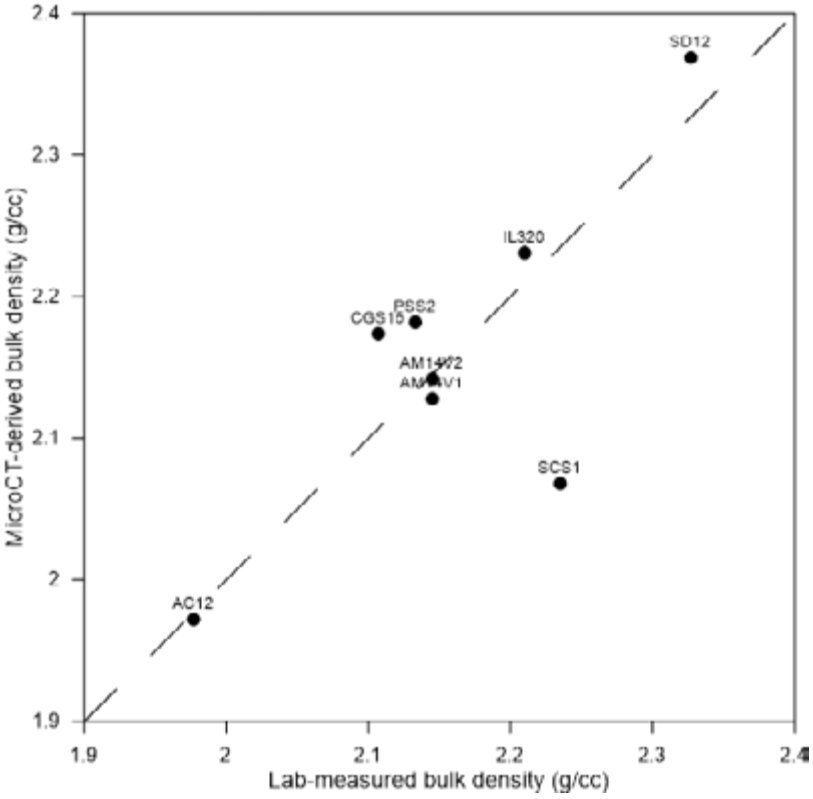

Figure 7 Comparison between the bulk density values measured in the laboratory and those estimated by microCT.

4 Conclusions

This work demonstrates the efficiency of the method for determination of mineral composition and petrophysical properties of carbonate and sandstone rocks through X-ray microtomography digital image analysis. The results obtained by the proposed method are compared with their corresponding obtained by methods traditionally established for these purposes. The best results were obtained in the quantification of the mineral composition of the rocks and, consequently, in the estimation of grain density. The porosity values obtained by microtomography were in most cases lower than the values obtained by gas pycnometer method. This might be caused by insufficient image resolution to quantify very small pores. On the other hand, rocks with substantial content of isolated pores may present higher microCT porosity in comparison with gas-measured one. Heterogeneity of the samples can be responsible for a grain density - microCT derived - greater than that measured in petrophysical test, once the prior is estimated on a sample of millimetric dimensions, while the last is measured in a centimeter-sized plug. The bulk density of a dry sample depends on the rock mineral composition and on its porosity. For this reason the correlation between bulk density measured by the two methods shows an intermediate efficiency, something between those of grain density and of porosity. Although the results presented here, as well those offered by the specialized literature, indicate that the microCT image analysis is appropriate for quantification of mineral phases, this technique has potential for determination of many other physical properties, such as mechanical, electrical and fluid flow rock properties.

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