X-Ray Microtomography and NMR: complimentary tools for evaluation of pore structure within a core

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ABSTRACT

We propose a new scientific approach for calibration of relaxation times to the real pore sizes by combination of both NMR (Nuclear Magnetic Resonance) and X-ray microtomography (XmCT) techniques. The approach contains the following steps, (1) Application of morphometric image processing procedure to 3D XmCT image of a sample. The morphometric image processing allows studying different morphological features of pore space structure. Particularly, pore size distribution may be obtained this way. (2) Fitting of the pore size distribution with NMR T_1-T_2 spectra would provide the required calibration. The procedure based on tuning the relaxivity by minimizing a norm containing difference of the XmCT and NMR distributions in a range of pore sizes resolvable by XmCT. The capillary-lattice model also examined as method of a relaxivity calculation using experimentally measured value of absolute permeability (gas permeability with Klinkenberg correction) of the core.
The described approach was tested as on artificial and natural rock samples. Obtained values for relaxivity show good agreement with published data. The microporosity values which correspond to the volume of void structure within the samples below the XmCT resolution and sensible to NMR are confirmed by routine porosity measurements.

INTRODUCTION

Over the last decade XmCT has become well accepted in petrophysics community as a convenient and efficient technique for studying pore space structure. Nevertheless, its capabilities are limited by a spatial resolution of about 1 μm. Even though the modern nano-CT devices provide resolution down to 50 nm, the field of view for such scanners is dramatically small leading to sample representativeness loss. In contrast to XmCT, laboratory NMR machines observe hydrogen nuclei of fluids constrained by pores of any geometry. Using T_1 or T_2 relaxation spectra one could simply construct reliable pore size distribution within a rock in several minutes experiment. Unfortunately, NMR is indirect method for pore size evaluation based on proportionality between relaxation times and surface to volume ratio of each pore. The coefficient of proportionality is relaxivity or relaxation activity of mineral pore surfaces. It is common to use a single value of relaxivity for a single core assuming that pore surface properties of small sample are homogeneous enough.

METHODS AND APPARATUS

Nuclear Magnetic Resonance

All presented NMR measurements were carried out using Oxford Instruments relaxometer, which contains MARANi-Pharmasence magnet block and DRX-HF electronic control system. The resonance frequency of 20.6 MHz corresponds to hydrogen nuclei spin precession in magnetic field of ~ 0.5 Tesla. The relaxation decays of T_2 and T_1 were recorded by ordinary CPMG and inversion-recovery pulse sequences. The 2D functions T_1-T_2 was measured on cores by sectional pulse sequences consists of two parts. The first one is 180°-90° bursts pair (the inversion-recovery) for T_1 registration and the second – standard CPMG for T_2 data. We used echo time of 150 µs and numbers of echoes sufficient to complete a decay. Schlumberger edition of 1D and 2D Inverse Laplace Transform (ILT) techniques were applied computing the distributions.

It is well known that S/V ratio (S – pore surface and V – pore volume) is proportional to inverse value of pore size and approximately to $1/T_{1,2}$ [1]. As follows from the above, the $T_{1,2}$ is proportional to pore size for a single fluid saturated reservoir, e.g. by brine model. The statement is based on the assumption that proton diffusion from one pore to another is negligible during a relaxation period and consequently relaxation in individual pore is not dependant on the processes in neighboring ones. In other words, relaxation time averaging does not occur between pores. Therefore, spin-lattice and spin-spin relaxation times spectrum of fully saturated rock could characterize the pore size distribution.

The transformation from relaxation times to geometrical sizes requires a calibration by direct methods and a pore model (spheres, cylinders, tubes, inscribed sphere, capillary lattice and etc.). The suggested procedure is based on XmCT study of cores.

X-ray Microtomography

We used laboratory table-top micro-CT scanner in this study. The radiation source was an X-ray tube (10 W) with a voltage of 100 kV, current 100µA and focal spot diameter less than 5 µm. Small size of the source allows using of cone beam geometry for projection image magnification. Reconstructed dataset had the size ~4000x4000x2000 with effective pixel size ~2.2 µm. For image processing and analysis we used either commercial or developed in our center software.

The techniques of pore size evaluation by X-ray micro-CT

In contrast to NMR, XmCT allows "direct" measuring of pore space geometry in 3D. For this purpose, the reconstructed 3D image of a core sample should be segmented into two classes of objects: pores and minerals. Further calculation of the pore size distribution may be performed in various ways.

Individual pore analysis

The most intuitively obvious approach consists in separation of a whole pore space on a set of individual pores with further quantification analysis of each. General algorithms for separation are based on watershed technique. Equivalent spherical diameter is one of the most commonly used quantities for estimating the effective size of the object body. Finally, the histogram of the size values may be constructed.

Sphere fitting technique

Alternative method for calculation the object size distribution was proposed in [2]. This technique is based on sphere fitting inside the 3D structure. In that case, the local

thickness of the object in the point *A* is defined as the diameter of the largest possible sphere that fully inscribes into the structure and covers point *A*. Thus, point A may not be a center of the covering sphere. The final distribution of the pore sizes may be obtained by calculating the histogram of the 3D local thickness distribution.

In our view this algorithm more adequate describes the pore space and more correctly fits with physical phenomena underlying in NMR method.

Relaxation time to pore radius transformation

The most simple and widely-distributed ways to present the ratio *S/V* are theoretical estimations using an idealized pore structure models for spherical, cylindrical or slit-like pores. In these cases, the ratios *S/V* correspond to 3/*r*, 2/*r* and 2/*d* values accordingly. We applied the spherical pore model assumption $r = 3\rho T_{1,2}$ as the inscribed sphere method used to calculate pore sizes by XmCT data. However, regardless of applied model the value of relaxation activity mathematically represents the proportionality coefficient between pore sizes and relaxation times. Therefore, the pore surface relaxivity used as a tuning parameter of spectra coincidence during the construction of pore sizes distributions by NMR data. It is essential that the introduced coefficient of proportionality between relaxation times and pore diameters is complicated value contributed by a pore surface relaxivity and wettability, as well as pore shape. Therefore, it is more correct to name the coefficient ρ as pseudo relaxation activity or cumulative parameter responsible for surface relaxation properties defined by mineralogical composition of rock and a value of specific surface.

Along with the direct comparison of two distributions, we also applied the approach based on multilateral capillary lattice model of reservoir proposed by V.A. Murtsovkin [3]. This model of the porous medium consists of several different-scale three-dimensional cubic capillary lattices. Each lattice can be represented by a large number of identical cubic cells with identical structures of pore space and preset pore size distributions over the cells. There is one node per each cell in which three perpendicular capillaries intersect. Cells only differ in the sizes of corresponding capillaries, which are assumed constant within one cell. To simplify calculations, it is supposed that all capillaries in the *i*th cell are characterized by a square cross section with side δ_i.

Lattices differ in size *a* of the corresponding cells. The number of lattices is determined by the peculiarities of porosity distribution over pore sizes. Pore-size distribution over cells is described by the *f(δ)* function that present the differential probability distribution function for random magnitude δ. The *f(δ)* function can be determined directly by the spectrum of the times of transversal or longitudinal relaxation [3].

Finally, given measured values of the absolute permeability (Klinkenberg gas permeability) we can estimate the relaxivity coefficient using the multilateral capillary lattice model and the NMR data.

EXPERIMENTAL EXAMPLES

The study of void space structure was performed on cylindrical samples 8 mm in diameter and 10 mm length. This size of core allows experiments on the same sample using the NMR and XmCT techniques. Experimental tests of the proposed approaches were conducted on several objects with different structure and mineralogical

composition: sintered glass beads sample, well-sorted sandstone and the dolomite with complicated structure. Therefore, the wide range of pore structures classes from simple packing of spheres to complicated system with dual porosity and mixed wettability type was analyzed.

Sintered glass beads

For a simplest object to study we chose a sintered glass beads sample (Φ = 25.5%, Permeability = 1266 mD). The sample was composed from rather big solid grains and so the pores should be visible. For that reason we assume that XmCT had successfully resolved almost the whole pore space structure. The results are shown on Fig.1. One can notice the broadening of NMR T_1 spectrum peaks, as well as distinction between shapes of both spectra. The first issue is related to mathematical processing drawback (ILT), while the shape the distinction originates from nonlinearity of relaxivity dependence on pore size or $\rho_{T1,2}(T_{1,2})$. Presented on the right of Fig.1 T_1/T_2 2D map allows to see this effects clearer. After the calibration of NMR by XmCT the following values of relaxivity have been obtained: $\rho_{T2}(mCT)$ = 53µm/s; $\rho_{T1}(mCT)$ = 28µm/s. The theoretical estimations for capillary-lattice model according to the laboratory measured gas permeability are rather close $\rho_{T2}(cap.lattice)$ = 73µm/s, $\rho_{T1}(cap.lattice)$ = 31µm/s. It shows the consistency of achieved data, as well as validity of the capillary lattice model for this particular case.
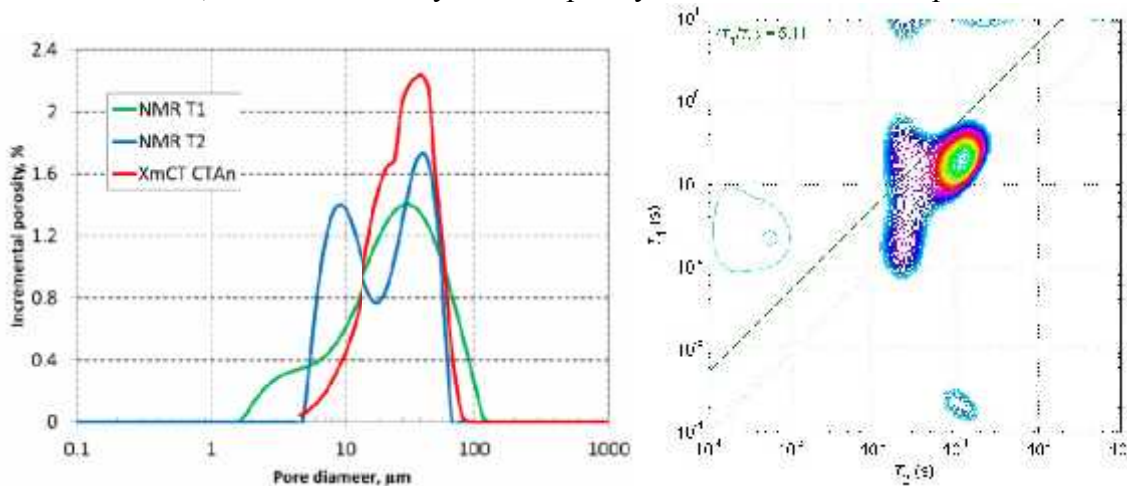

Fig.1. Pore size distributions by NMR and micro-CT and T1-T2 2D-map for the glass beads artificial sample (Probably pure silica, Φ = 25.5 p.u., Permeability = 1266 mD)

Sandstone

A similar series of experiments and calculations were carried out for granular quartz sandstone. The results of fitting T_1 and T_2 distributions with XmCT data, as well as T_1-T_2 map are presented on Fig. 2. Combination of T_2 spectra with XmCT data leads to relaxivity value $\rho_{T2}(mCT)$ = 24µm/s. Capillary-lattice model provides rather close value $\rho_{T2}(cap.lattice)$ = 30.2µm/s. Obtained results allow estimation of microporosity amount which is below the XmCT scanner resolution (~5µm). Thus, for studied sample considered approach gives the volume value of microporosity "invisible" by XmCT equals to 45.6% of the total capacity, i.e. 14.6 p.u.

Analogous procedure was applied to the spectra of the longitudinal relaxation time T_1 and provided the following results: $\rho_{T1}(mCT)$ = 7.4µm/s, $\rho_{T1}(cap.lattice)$=6.7µm/s and microporosity=9.2 p.u. (29.7%).
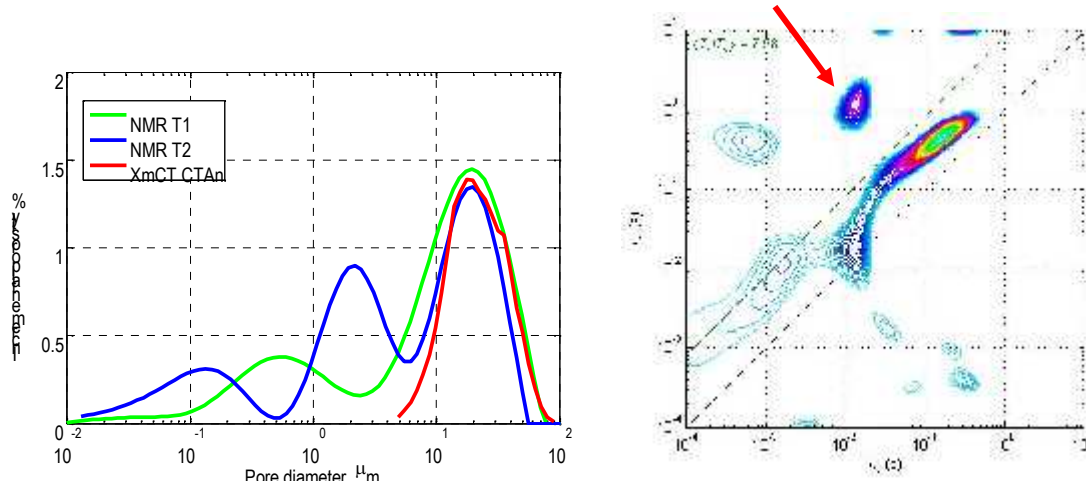

Fig.2. Pore size distributions by NMR and micro-CT and T1-T2 2D-map for the sandstone rock sample (Silica=62%, Feldspar=20%, Clays=18%, Φ=27.8%, Permeability=407mD)

Received values of relaxivity are in good agreement with the literature data for the same type of terrigenous rocks [4]. Lower values of the relaxation activity of the pore surface in the second case caused by the various natures of the physical mechanisms of longitudinal and transverse relaxation. Comparison of T_1 and T_2 values measured on a set of water-saturated rock samples of different types was performed in [5]. It was shown that T_1/T_2 ratio ranges in 0.8 – 2.3. Generally, average value of this ratio is taken as 1.5 – 1.7. Overestimation of ρ_{T2} values is caused by the diffusion relaxation mechanism originated from significant internal magnetic gradients. These gradients exert tremendous influence on NMR studies with high values of the magnetic field or the resonance frequency (20.6MHz).

Differences in T_1 and T_2 spectra are clearly observed on 2D T_1-T_2 map (Fig.2). One can notice the area with inflated T_1/T_2 ratio (shown by arrow on Fig.2). This separate region may be associated with the presence of the hydrocarbons remaining after extraction and/or contrasting pore surface areas with high content of paramagnetic centers enhancing local diffusion processes, and hence drastically reduce T_2

Dolomite

The results of the same study of test dolomite sample are presented below on Fig.3. Achieved relaxivity values are $\rho_{T2}(mCT)$=75µm/s, $\rho_{T1}(mCT)$=13µm/s, $\rho_{T2}(cap.lattice)$=2.1µm/s, $\rho_{T1}(cap.lattice)$=0.3µm/s and microporosity=7.9 p.u. (45.5%). Significant differences in ρ values obtained by different methods reflect the complexity of the rocks pore space. We assume that the filtration system (determining the permeability) sufficiently differs from the capacitive structure, i.e. pore throat and pore body size distributions have different size ranges and dependencies shapes. Therefore, the

accuracy of using the measured permeability as an indicator of the capacitive structure is very low for complex carbonate rocks.
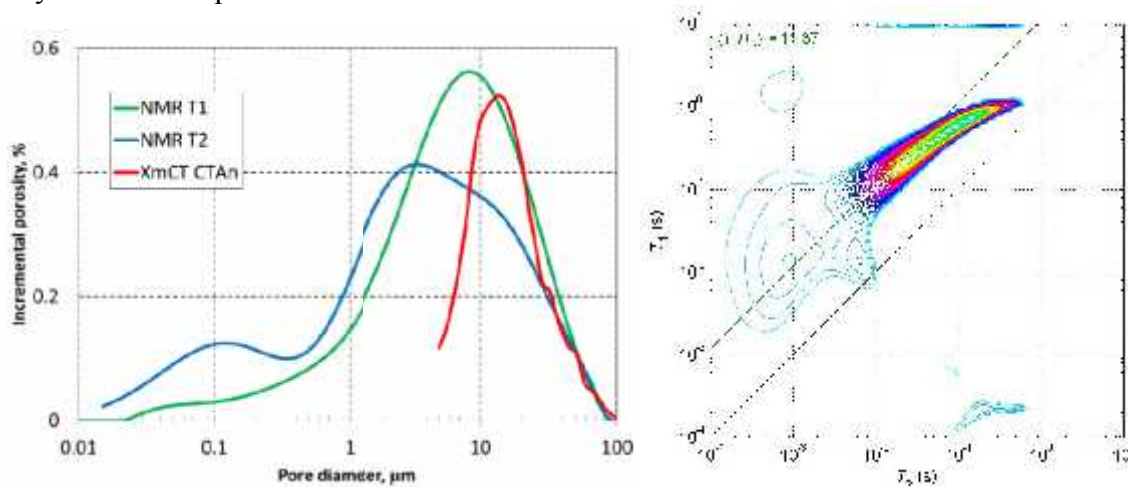

Fig.3. Pore size distributions by NMR and micro-CT and T1-T2 2D-map for the dolomite rock sample (Dolomite=85%, Anhyd.+Hal.=15%, Feldspar=1%, Φ=13.4%, Permeability=0.21mD)

CONCLUSIONS

Achieved results confirm the applicability of the proposed technique. Moreover, they point out the necessity of integrated study of the pore space structure of rocks by the methods investigating pore bodies, throats and surface properties. Detailed investigation with several methods allows to avoid uncertainties in the interpretation of NMR spectra in range of a long relaxivity times (responsible for the effective porosity), as well as the short (microporosity).

ACKNOWLEDGEMENTS

REFERENCES

[1] W. E. Kenyon, "Nuclear Magnetic Resonance as a Petrophysical Measurement," *Nuclear Geophysics,* vol. 6, no. 2, pp. 153-171, 1992.

[2] T. Hildebrand and P. Ruegsegger, "A new method for the model independent assessment of thickness in three dimensional images," *J. Microsc.,* vol. 185, pp. 67-75, 1997a.

[3] V. A. Murtsovkin, "The Use of Multilattice Capillary Model for Calculating Permeability by NMR Data," *Colloid Journal,* vol. 71, no. 5, p. 699–705, 2009.

[4] W. E. Kenyon, "Petrophysical Principles of Application of NMR Logging," *The Log Analyst,* vol. 38, no. 2, pp. 21 - 43, 1997.

[5] R. L. Kleinberg, "Pore Size Distribution, Pore Coupling, and Transverse Relaxation Spectra of Porous Rocks," *Mag.Res.Imag.,* vol. 12, no. 2, pp. 271 - 274, 1994.