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Experiment and Simulation on NMR and Electrical Measurements on Liège Chalk

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Abstract

Liège Chalk is a limestone of considerable commercial interest to the petroleum industry and formation factor a quantity required in this context. In this work we compare the formation factors based on electrical conductivity and diffusional displacement in a long time limit both experimentally and numerically. Measurements are performed on Liège Chalk samples while simulations are performed on two model structures represented by randomly packed ellipsoids and utilizing a Gaussian random field approach. We ensure similarity in petrophysical sense of modelled media to Liège Chalk by matching experimental and simulated NMR relaxation response, Mercury Injection Capillary Pressure curves and electrical resistivity. Following this, the diffusional-based formation factor is estimated from a set of apparent diffusion coefficients in the tortuosity limit obtained with PGSTE NMR. All measurements have been numerically-simulated and are in good agreement with experiment. We have shown that for Liège Chalk, the NMR diffusion and electrical resistivity based formation factors do agree.

Keywords

NMR, formation factor, time dependent diffusion coefficient, tortuosity limit, resistivity

1. Introduction

Formation factor is an important petrophysical property required for the characterization of petroleum bearing reservoir and is normally obtained by electrical resistivity measurements. For nonconductive porous media saturated with brine, the formation factor F is given by Archie's equation [1]

$$F = \frac{R_o}{R_w} = \frac{\sigma_w}{\sigma} \quad , \tag{1}$$

where R_o is the resistivity of the porous medium saturated with brine, R_w is resistivity of brine, σ is the conductivity of the fluid saturated porous medium, and σ_w is the conductivity of the saturating fluid. The resistivity based derivation of formation factor or the related resistivity index can only be used for conducting fluids. In certain cases as discussed below that information may be assessed with the aid of NMR measurements.

The NMR pulse gradient stimulated echo (PGSTE) technique provides a non-destructive way of measuring time dependent diffusion coefficients [2]. The PGSTE echo attenuation is given by:

$$S(t)=\frac{1}{2}\exp\left(-\frac{T}{T_1}\right)\exp\left(-\frac{2\tau}{T_2}\right)\exp\left(-D\gamma^2g^2\delta^2\left(\Delta-\frac{\delta}{3}\right)\right), \tag{2}$$

where D is the time dependent diffusion coefficient, Δ is the diffusion time, γ is the gyromagnetic ratio (γ = 2.67×10^8 rad/Ts for ^{1}H), g and δ the magnetic field gradient pulse strength and duration respectively. The slope of the plot of echo attenuation vs. different gradient will provide the value of time dependent diffusion coefficient.

In media homogeneous beyond the PGSTE diffusion length scale [3] the spins may explore all length scales and the full tortuosity over longer diffusion times. This is similar to the measurement of electrical resistivity by two probes connecting both sides of the core plug once the diffusion time is long enough to allow the spin to diffuse through the whole system, leading to a relationship between diffusion coefficient and Formation factor [4]:

$$\lim_{t\to\infty} D(t)=\lim_{t\to\infty}\frac{\langle[r'(t)-r(0)]^2\rangle}{6t}=\frac{D_0}{\phi F}=\frac{D_0}{\alpha}. \tag{3}$$

Here $\langle[r'(t)-r(0)]^2\rangle$ denotes the average over the distance travelled by spins with initial position *r(0)* and final position *r'(t)* and α is the tortuosity.

Hürlimann et al. [5] proposed that in NMR experiment for the spin diffusing in a simple geometry with a length scale l_s and gradient g can be described by three length scales, diffusion length $l_d=\sqrt{6D\Delta}$, pore structural length $l_s=V/S$, and dephasing length, $l_g=\sqrt[3]{D/\gamma g}$. Here, *V/S* is the pore volume to surface ratio and g is the internal magnetic field gradient. These length scales form three asymptotic regimes, the free diffusion, motional averaging and localization regime. In the motional averaging regimes, pore structural length is the smallest among three length scales, particles probe all parts of the pore and the diffusing spins average all field inhomogeneities.

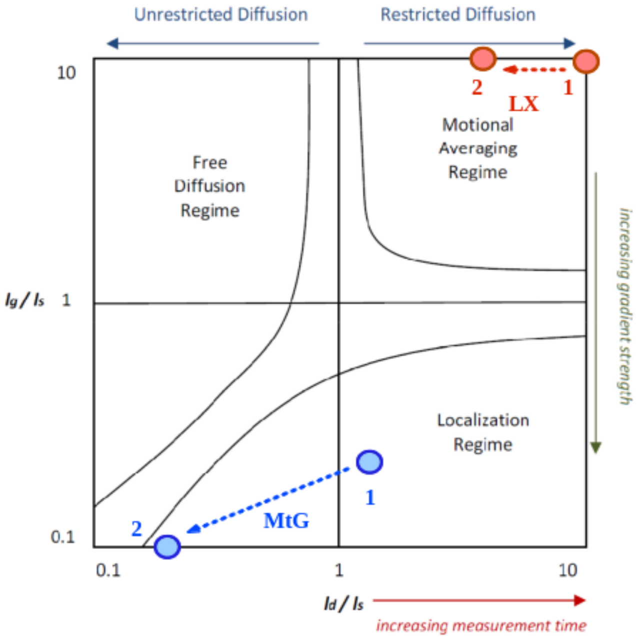

Fig. 1: Relaxation asymptotic regimes of carbonate rocks: Liège Chalk (LX) and Mount Gambier limestone (MtG). Two ultimate NMR experimental regimes are considered here: (1) long time (t_d = 20 ms) and short time (t_d = 100μs). The interval of relaxation regimes typical for Liège Chalk (LX) corresponds to two red round marks and an arrow, those corresponding to Mount Gambier carbonate – blue.

The formation factor may be derived

by diffusion only for porous media where the diffusing spins stay in the motional averaging regime with mixing time. Figure 1 illustrates the relaxation asymptotic regimes of two carbonate rocks: Liège Chalk and Mount Gambier limestone [6]. The applicability of deriving formation factor from NMR experiment depends on the characteristic pore scale of a rock. If NMR signal decays faster than spins diffuse across a sufficient number of pores (diffusion length is in the order of the pore size) the technique cannot be used (e.g. Mount Gambier limestone). On the other hand, a sufficiently homogeneous rock with pore and grain size much less than the diffusion length provides a case when spins have enough time to explore all the length scales before the loss of a signal. We expect that Liège Chalk falls into this regime.

This paper focuses on a comparison of diffusion-based formation factors derived from NMR to resistivity-based. Low field NMR diffusion-edited experiments may have technical or practical restrictions on minimum and maximum diffusion time intervals. These restrictions are known to be more pronounced for tight rocks such as chalks. In this work we intend to overcome those experimental limitations with the aid of numerical simulation. These are normally carried out using tomographic images obtained e.g. by micro-CT to capture a relevant field of view (FOV). Here Xray micro-CT imaging does not provide sufficient resolution and FOV at the same time. We treat this problem by (forward) modeling chalk as a mixture of simplified 3D geometries until key petrophysical properties of a model match those experimentally observed. Given such validated model structures we compare direct measurements of formation factor measurements by electrical and NMR techniques with numerical simulations.

2. Experiment

We acquired a large block of outcrop Liège Chalk and drilled two plugs of 25.4mm diameter and 38mm length for resistivity and NMR experiments; we also extracted one plug of 8mm diameter and 12mm length for MICP measurement. The samples were dried in the oven at 120 ˚C for 24 hours. The large plugs were placed in a vacuum for another 24 hours and then saturated with a 2% by weight NaCl brine solution in the vacuum. The porosity was calculated using the core weight difference before and after saturated with brine. Resistivity was measured by using a 2-electrode method for the plugs 100% saturated by the NaCl brine.

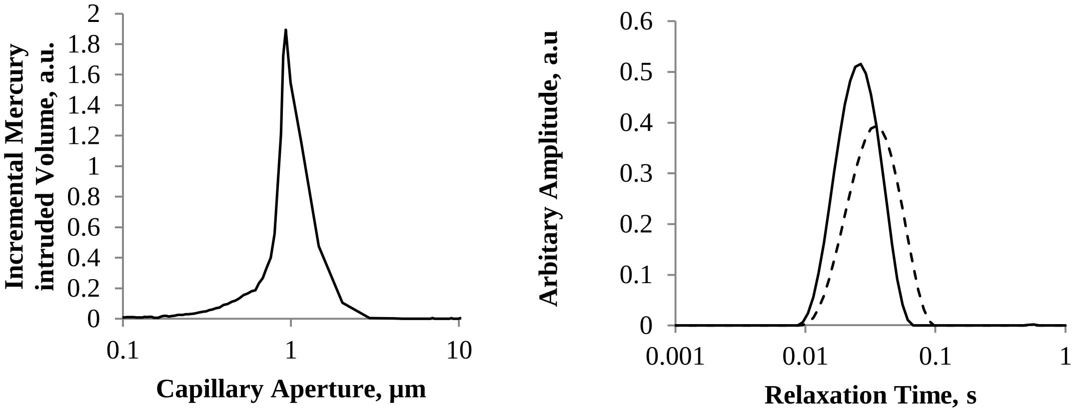

Fig. 2: *Left:* Normalized MICP experiment pore aperture size distribution; *Right:* Longitudinal (Dashed) and transverse relaxation (Solid) distribution.

MCIP measurements using a PoreMaster PM33-17 porosimeter were used to obtain the accessible volume weighted pore-throat distributions (Fig.2 left). These are compared with numerically simulated MICP experiments on digitized representations (Fig.4a). The MICP experiments indicate that the pore entry radius of Liège Chalk is 0.37 µm. The formation factor derived from electrical measurements do agree for both plugs, both 10.9 at 22.5˚C.

We used a 2 MHz Magritek Rock Core Analyzer to perform CPMG and PGSTE pulse sequence experiments to obtain longitudinal and transverse relaxation (T_1 and T_2) and diffusion attenuation measurements. For 100µs TE NMR experiments, $\mathrm{T_1}$ and $\mathrm{T_2}$ are 33.0ms and 24.5ms respectively (Fig 2 right), and the ratio T_1/T_2 is 1.34, which is relatively low. From the PGSTE measurements we estimate the time dependent self-diffusion coefficient at long times. We use a 7-interval unipolar sequence and 40 steps for the pulsed magnetic field gradient with maximum strength of 0.5 T/m and 2 ms duration. The diffusion times are ranging from 20 ms to 100 ms, which is limited by the width of the gradient pulse of PGSTE sequence, signal to noise ratio and the spin-lattice and spin-spin relaxation times of the fluid. The surface relaxivity of chalk as derived from MICP and T_2 measurements is 7.7 µm/s [7, 8].

3. Modelling

Based on morphology visualized by SEM images of Liège Chalk (Fig. 3 left), we developed two groups of models (Fig.3, middle and right) at very high resolution with various degree of heterogeneity: single porosity model systems with three different overlapping ellipsoids randomly oriented with volume fraction of either phase is $\phi = 0.32$; the other dual porosity model systems using Gaussian Random Field (GRF) [9] for volume fraction of $\phi = 0.34$ with distinct particles randomly filled into two different fields of 1-level-cut [9, 10]. Two models were determined by changing size and axis ratio of ellipsoidal particles to match V/S of the chalk sample (Table. 1). Surface relaxivity is set to be same as chalk (7.7 µm/s). Characteristic lengths and NMR-related physical properties (susceptibility contrast) were adjusted to match simulated resistivity, NMR T_2 and MICP with experiments (Fig.4).

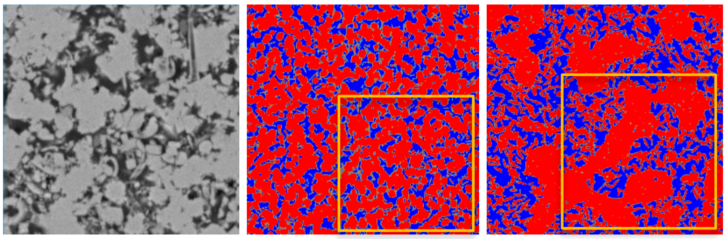

Fig. 3: *Left:* SEM image of Liège Chalk, field of view 50*50 µm; *Middle:* Slice of a 3D model structure with 3 different particles; *Right:* A slice of dual porosity GRF model structure. The size of the yellow frames is 50*50 µm.

Table 1: Model structures

Model	Dimension (Voxels)	Voxel Size (nm)	Particle sizes (Voxels)	Porosity (%)
GRF Dual Porosity	1000×1000×1000	75	10×10×6	34.2
			31×15×5	
Single porosity	1000×1000×1000	85	15×17.5×18.5	32
			20×18.5×2	
			50×10×2	

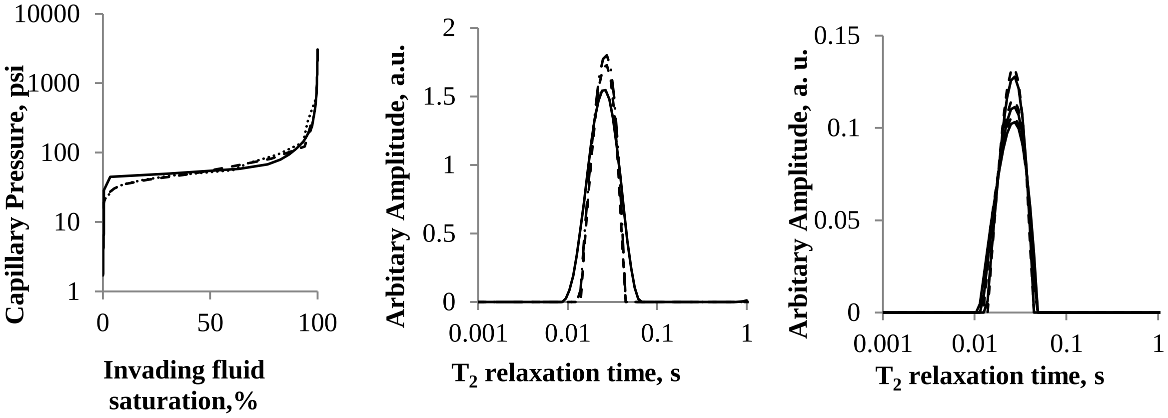

Fig. 4: *Left:* MICP curve of Liège Chalk Experiment (solid) and simulations (dashed); *Middle:* Experiment (solid) and simulations (dashed) of T_2 distribution at TE = 100 µs; *left:* T_2 distributions of single porosity model (dashed) and dual porosity model (solid) at TE = 100, 200, and 300 µs.

The conductivity is calculated by solving the Laplace equation with charge conservation boundary conditions [11, 12]. The 3D voxel microstructures are first converted into a network of resistors by connecting each pair of adjacent voxels by a resistor. A potential gradient is applied in each coordinate direction, and the system relaxed using a conjugate gradient technique to evaluate the field. We assign a conductivity of $\sigma_{grain} = 0$ to the matrix phase of the chalk and the fluid filled pore phase a conductivity of $\sigma_w = 1$. The formation factor, the ratio of the measured conductance of the fluid filled rock σ to that of the fluid itself, given by $F = \sigma_w/\sigma$, is reported.

The drainage experiments are simulated using morphological transforms on 3D voxelized representations of microstructures. Distributions of invading and displaced fluids are set by propagating through the phase labelled as a void a spherical structuring element of the size honouring local capillary pressure [11, 13]. The simulation is performed by considering all possible invading sphere radii providing the capillary drainage curve.

The NMR spin relaxation is simulated by using a lattice random walk method [14], which mimics thermal Brownian motion of spins ensembles in confined space subjected to magnetic field. Initially, the walkers are randomly placed in the 3D pore space. At each time steps of $\Delta t=\varepsilon^2/(6D_0)$, the walkers are moved from their initial position to a neighbouring site, ε is the lattice spacing and D_0 is the bulk diffusion constant of the fluid.

3. Results, Discussion and Conclusion

Table 2: Comparison table of experiments and simulations

Properties	Porosity (%)	Mean Pore Entry Radius (µm)	T_{2LM} (ms)	Susceptibility Contrast (µSI)	Formation Factor	
					Electrical	Diffusion
Experiment, Liège Chalk	32.6	0.37	25.38	10 ~ 70	10.91	12.2
Dual porosity GRF model	34.2	0.44	25.38	75	10.7	9.4
Single porosity model	32	0.43	25.40	81	8.9	10.4

We compare the computed formation factors of the fluid saturated Liège Chalk with experimentally measured values (Table 2). The predicted diffusion-based formation factors are in a good agreement (within 15%) with the resistivity-based formation factors. We see an underestimation of the electrical formation factor for the single porosity model. This may be caused by the pore structure of the model being more evenly distributed resulting in lower tortuosity. The dual-scale GRF model structure exhibits a higher tortuosity and better captures the morphology of the original chalk as the shown in the SEM image.

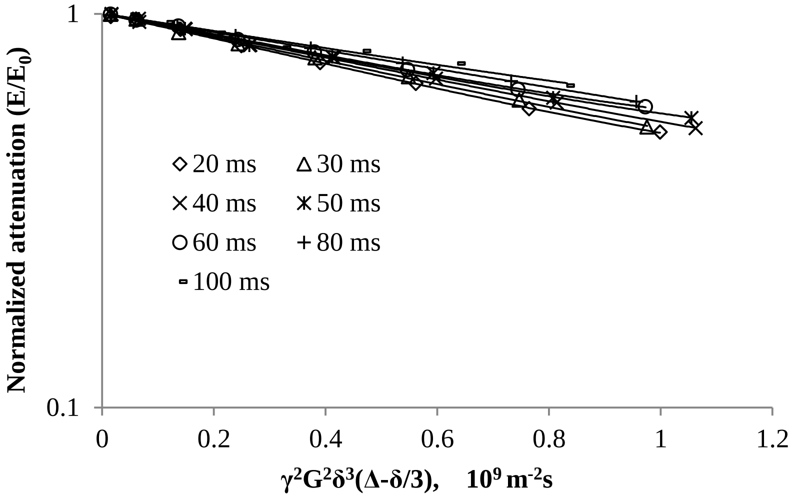

Fig. 5: Echo attenuation with respect to various diffusion times.

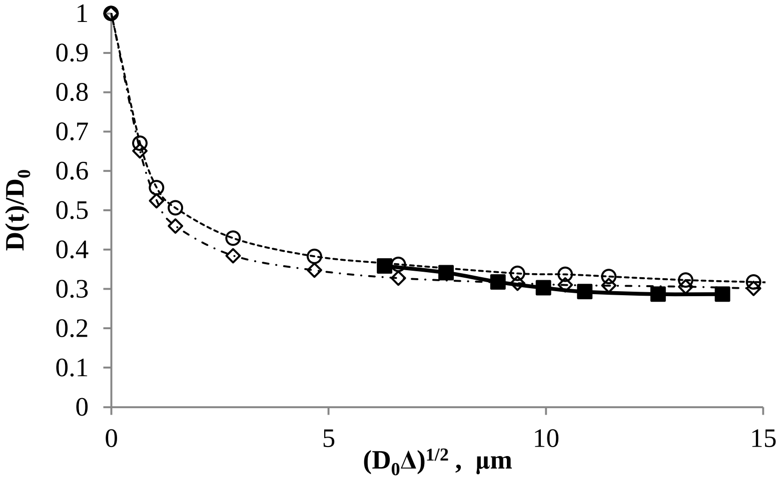

Fig. 6: Time-dependent diffusion measurements on chalk and models: PGSTE experimental data (Solid line) and simulation on single-porosity model (Dashed line), simulation on dual-porosity GRF model (Dash-dot line).

In the PFG NMR experiment the signal to noise ratio becomes smaller as diffusion time increases resulting in a maximal diffusion time limit above which the signal is not distinguishable from noise. The maximum diffusion time limit is controlled by a relaxation time of a system [15] and may be assumed as 3 times T_1. Characteristic (log-mean) T_1 of a Liège Chalk is 33 ms which limits the experimentally accessible longest diffusion time to 100 ms. The available diffusion interval Δ of our PGSTE experiments is between 20 ms and 100 ms. Figure 5 shows the experimental echo attenuation for the series of measurements in this time interval. The plot shows that the entire set of experimental data points fall into the restricted diffusion regime, i.e. near the tortuosity limit. This is an expected outcome since the diffusion length even at a shortest available Δ is about 10 μm. This displacement distance corresponds to a few pore and grain sizes.

In this work we experimentally demonstrated that for Liège Chalk the resistivity-based Formation factor may be evaluated with NMR diffusion-edited measurements. We demonstrate the possibility to design a synthetic digitized representation of a real rock wich realistically responds to a range of petrophysical simulations, including capillary drainage, conductivity and NMR relaxation. Simulations using this model structure confirm our observations about similarity between electrical and NMR-diffusion formation factors. Moreover, simulations provide access to diffusion times (short time limit) restricted for experimental measurement.

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