

NUCLEAR MAGNETIC RESONANCE (NMR) PROPERTIES OF SYNTHETIC AND SANDY SAMPLES

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ABSTRACT

Fluids can be determined on samples directly using NMR technique [1,2,3]. The amplitude of the NMR signal is related to the number of hydrogen protons, the technique can be used in geophysics to measure the water content of rocks and soils [4,5]. In this study, to identify relaxation times according to porosity values we used synthetic samples. For the NMR measurements, we used monomodal glass pearl samples (0,1-0,5-0,8-1 mm) and sand samples (0,35-0,75-1,5 mm) with additional water or petroleum. According to the relaxation times we measured porosity and compare them with ordinary lab measurement results. For the results T1(INVREC) and T2(CPMG) relaxation times are much better suited than T2*(FID) for the analysis. All measurements were carried out in the department of Geoscience, Technical University of Berlin in Germany.

Keywords: NMR, FID, CPMG, porosity

ÖZET

Akışkanlar NMR tekniği kullanılarak tespit edilebilir [1,2,3] NMR sinyalinin genliği hidrojen atom sayısı ile alakalıdır ve teknik jeofizikte kayaç ve zeminlerin su muhtevasını ölçmede kullanılabilir [4,5]. Bu çalışmada sentetik numuneler (su ve petrol eklemeli) olarak laboratuvar kaya fiziği porozite ve akışkan tayini çalışması için kullanılmıştır (monomodal glasspearl numuneleri; 0,1-0,5-0,8-1 mm çaplı ve kumlu numuneler; 0,35-0,75-1,5 mm çaplı). Relaksasyon (gevşeme) zamanına bağlı olarak numunelerin porozite ölçümleri hesaplanmıştır, buna göre T1 ve T2 gevşeme zamanları T2* gevşeme zamanına göre daha uyumlu sonuçlar vermiştir. Bu çalışmaya ait ölçümler Berlin Teknik Üniversitesi, Yerbilimleri bölümünde gerçekleştirilmiştir.

Anahtar Kelimeler: NMR, FID, CPMG, porozite

INTRODUCTION

Increased efforts have been devoted in the last two decades and apply techniques that are known to be directly sensitive to water and are in successful use already. Nuclear Magnetic Resonance (NMR) is meanwhile an established method in the fields of physics, physical chemistry, medicine, material testing and lately, also in borehole geophysics [6]. Consequently, the available investigations focus on the NMR properties of solid rocks, because of their significance as hydrocarbon reservoir rocks [7].

The NMR signal from hydrogen from nuclei is proportional to the population of hydrogen atoms in the sample, and the signal relaxation rate is proportional to the viscosity of the fluid. Therefore, it was natural to consider using the NMR property for oil exploration. However, early investigators

found that the nuclear magnetic relaxation rate of water in rocks is much faster than that in bulk phase. The increase of relaxation rate is primarily due to the surface relaxivity and is related to the surface to volume ratio of the pore space. This discovery led to the demonstration of the connection between the NMR properties of porous media and their permeability [8].

Predicting reservoir wettability and its effect on fluid distribution and hydrocarbon recovery remains one of the major challenges in reservoir evaluation and engineering. Current laboratory based techniques require the use of rock-fluid systems that are representative of in situ reservoir wettability and preferably under reservoir conditions of pressure and temperature [9].

In this study we only interested in proton (^1H) NMR, for being able to reach direct water detection and observe

hydrocarbon behavior in different types of synthetic and sandy samples. As known well that direct NMR measurements give us reliable and fastest results for wettability and porosity estimation in rock physics study.

BASICS OF NMR METHOD

The protons of the hydrogen atoms in water molecules can be described as spinning charged particles and have a magnetic moment μ . Generally, μ is aligned with the local magnetic field B_0 of the Earth. When another magnetic field H is applied, the axis of the spinning protons are deflected, owing the torque applied. When H is removed (Fig 1.), the protons generate a relaxation magnetic field as they become realigned along B_0 while precessing around B_0 with the frequency

$$\omega_L = \gamma B_0, \text{ Larmor Frequency } f_L = \omega_L / 2\pi \quad (1)$$

where $\gamma = 0,267518 \text{ Hz/nT}$, the gyromagnetic ratio for hydrogen protons.

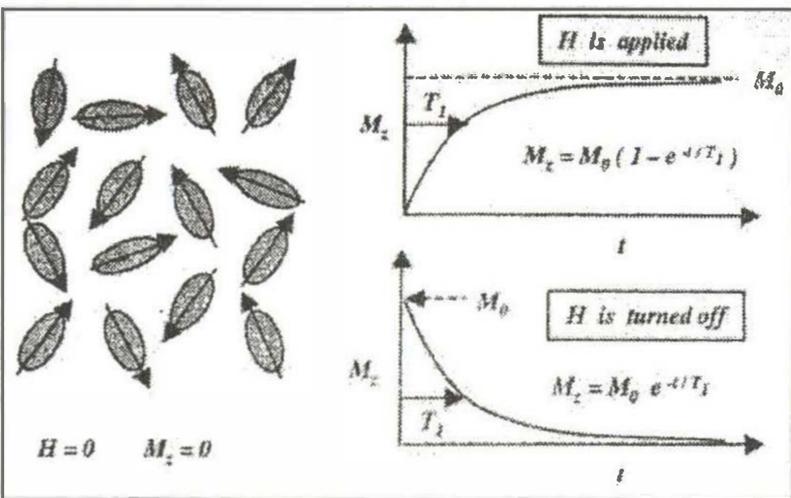


Fig.1. When there is no magnetic field, the proton spins randomly oriented, resulting in zero magnetization. As the field H is applied in z -direction, the magnetization of the system, M_z , gradually builds up until it reaches equilibrium value, M_0 . The process is reversed when H is set to zero [8].

At equilibrium, the net magnetization vector of protons is along the direction of the static magnetic field B_0 and is called the equilibrium magnetization M_z , which is referred to as the longitudinal magnetization. The time constant which describes how M_z returns to its value M_0 is the spin lattice relaxation time T_1 , with $M_z = M_0 \left(1 - \exp\left(-t/T_1\right)\right)$.

The time constant which describes the return to equilibrium of the transverse magnetization M_{xy} , is called the spin-spin relaxation time T_2 , with $M_{xy} = M_0 \exp\left(-t/T_2\right)$. The net magnetization in the

xy plane vanishes and then the longitudinal magnetization grows up to M_z along z . T_2 is always less than or equal to T_1 . In addition to T_2 the magnetization in the xy plane starts to diphas because each of the spins is experiencing a slightly different magnetic field and precesses as its own Larmor frequency. The longer the elapsed time, the

greater the phase difference. This leads to the faster decay time T_2^* . To measure T_1 or T_2 particular types of pulse echoes (sequences of the secondary magnetic field) can be applied. The most common are a single 90 degree pulse or free induction decay (FID) for T_2^* , echo trains of 180 degree pulses or CPMG for T_2 or a particular sequence of 90 and 180 degree pulses (inversion recovery) for T_1 (Fig 2.).

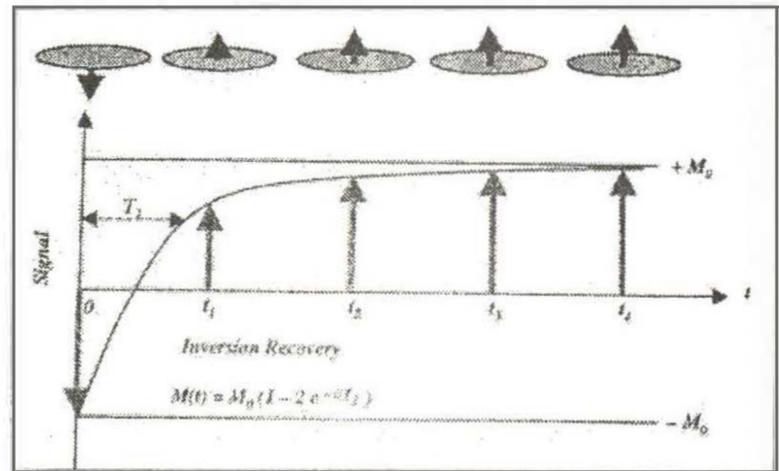


Fig 2. A typical sequence of inversion recovery for obtaining T_1 (INVREC). [10]

The volume of adhesive water can be derived from decay time spectra [10].

INSTRUMENTATION

All measurements were carried out in Berlin Technical University, NMR lab and performed with a Maran 2 from Resonance Instrument, UK (Fig 3 (a)). The instrument is equipped with a 0,047 T permanent magnet which corresponds to a Larmor frequency of 2 Mhz for protons.

In a laboratory set up, the sample is placed inside a coil between the pole pieces of a magnet. The applied field H_0 is from right to left, whereas the RF (Radiofrequency) field H_1 is pointing upward. The induced signal after a 90° pulse is shown as the Free Induction Decay [8] Shown in Fig 3 (b).

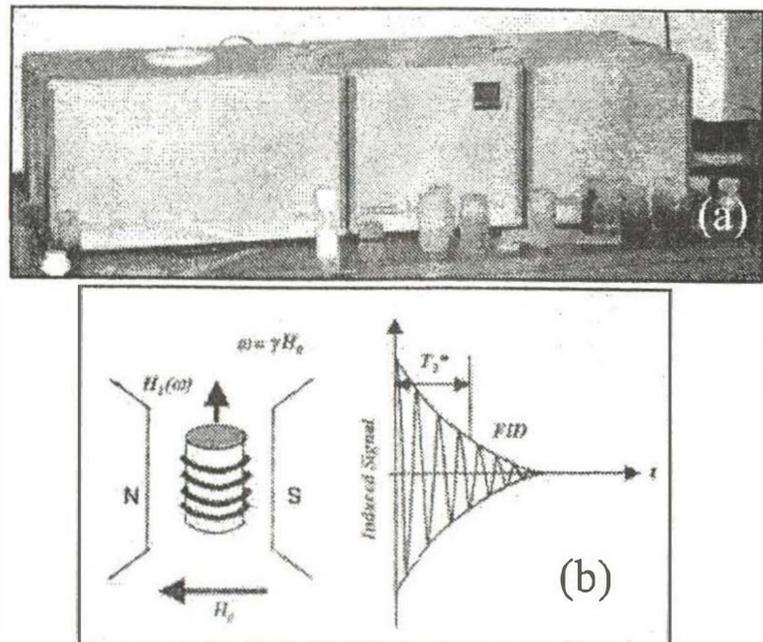


Fig.3.. (a); NMR instrument used in this study and (b); the NMR working principal [8].

NMR EXPERIMENTS

The effective NMR Porosity Φ_{NMR} can be calculated directly from water volume in the case of fully saturated samples;

$$V_{mobile} = (Amplitude_{sample} * V_{calibration}) / Amplitude_{calibration} \quad (2)$$

This leads to the effective (mobile) porosity;

$$\Phi_{NMR} = V_{mobile} / V_{sample} \quad (3)$$

1H protons in adhesive water or small pores relax much faster than protons in free water or large pores because of a higher probability of a contact with the grain surface and the associated energy release. A pore radii distribution can be derived therefore from a spectral analysis of the relaxation times [7]

An adaptation of the Kozeny-Carman equation leads to a permeability k estimation via 1H relaxation times;

$$K = c * T^2 * \Phi^2 \quad (4)$$

Where c is constant factor depending on the surface relaxivity of the material, Φ the effective NMR porosity and $T1$ the corresponding relaxation time (either $T1$ or $T2$).

The pore water close to the grains relaxes faster than water far away from the grains. The volume of adhesive water can be derived from decay-time spectra [10].

PETROPHYSICAL EXPERIMENTS

In this study, to correlate all NMR data with soil mechanics laboratory results (porosity from density), carried out basic techniques to find porosity of all samples.

For water content; Weighing the dry soil, Weighing the same soil after it has absorbed water, Calculating Wet Soil - Dry Soil = water absorbed, [11].

(Water absorbed / Original weight of soil) * 100 = Water holding porosity as a percentage of the dry soil [11].

Total Porosity (%) = Volume of air / Volume of soil [11].

SAMPLES

We analyzed two types of samples one; synthetic glass pearls with the diameter of 0,1-0,5-0,8-1 mm

and sandy samples with the diameter of 0,35-0,75-1,5 mm (Fig 4.). Samples are used in usual NMR studies during last decade [9].

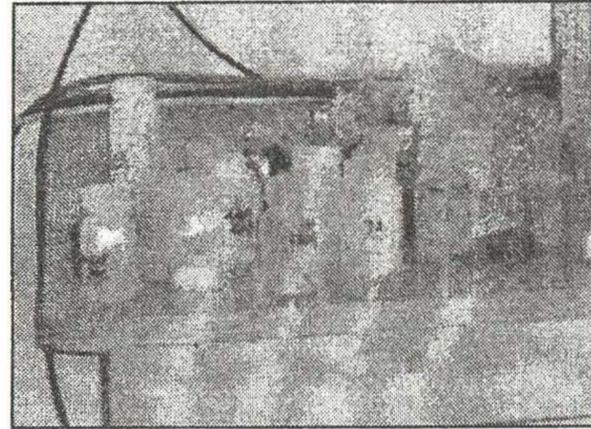


Fig 4. Samples tubes for petrophysics and NMR measurements

All samples named to such as shown in Fig 5., respectively.

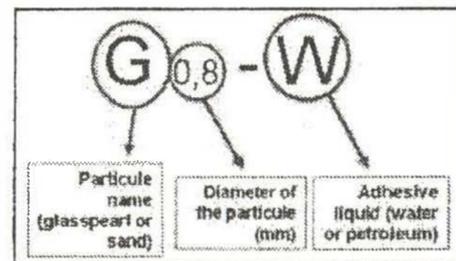


Fig 5. Definition of sample codes using for NMR measurements

LABORATORY MEASUREMENTS ON SAMPLES

The most important information derived from the laboratory measurements is that porosity, which has been determined using the bulk and grain density. Firstly, we determined the NMR porosity for three relaxation time sequences ($T2^*$, $T2$ and $T1$) and correlated with porosity from density measurements. The results are shown in Table 1. Basically, three mechanisms affect the NMR relaxation; diffusion, surface and bulk relaxivity [7] The graphics for the measurements are shown in Fig 6.

Table 1. Measurement results

SAMPLE CODE	POROSITY FROM DENSITY (%)	POROSITY NMR ($T2^*$) (%)	POROSITY NMR ($T2$) (%)	POROSITY NMR ($T1$) (%)
G _{0,1} -W	40,2	32,0	37,3	26,9
G _{0,5} -W	35,7	21,9	34,9	14,9
G ₁ -W	49,7	14,1	30,8	27,9
S _{0,35} -W	37,8	28,9	36,6	23,5
S _{0,75} -W	37,9	25,2	36,6	17,0
S _{1,5} -W	37,9	25,0	35,9	10,8
G _{0,1} -P	34,9	38,2	40,7	31,9
G _{0,5} -P	32,1	33,9	37,5	24,8
G ₁ -P	37,2	39,1	39,9	30,5
S _{0,35} -P	35,9	66,5	40,8	50,4
S _{0,75} -P	35,9	50,1	42,8	46,0
S _{1,5} -P	36,2	47,1	42,3	39,3

CONCLUSIONS

Low field NMR measurements can be obtained rapidly for T1 (inversion recovery) in about 20 minutes instead of too short time period of T2* and T2. We know from last decade papers that porosity and pore size distribution of unconsolidated rocks can be well determined by laboratory NMR. In this study we obtained more reliable results from T1. T1 (INVREC) and T2 (CPMG) relaxation times are much better suited than T2* (FID) for the analysis. This is almost due to inhomogeneous of the magnetic field circle of the instrument.

For the glasspearl samples adhesive petroleum fits the measurements (Fig. 6.). However glasspearl with water gives not exact results correlating with density NMR (Fig 7.)

For the sandy samples; adhesive water gives more reliable results instead of adhesive petroleum (Fig. 8. and Fig 9.). Extracting inhomogeneous part of magnetic coil or some misses during measurements, we would say that glasspearls make petroleum combination better than water, the converse of this phenomenon sand make better combination with water instead of petroleum. Hence the particule structure affects porosity measurements.

The NMR studies suggest that T2 and T1 distributions can provide valuable information regarding rock-fluid interactions. The development in this technique could provide faster and reliable study for rockphysics research. However further measurements and pore scale modelings are needed to establish a procedure that might be used for different type of rocks and unconsolidated rocks. Let say, for the estimation of more parameters such as permeability, pore radii etc. more investigations should be carried out with a combination of resistivity research.

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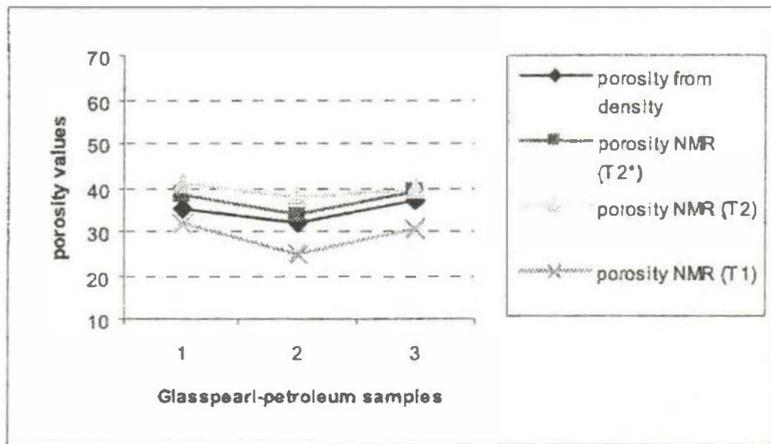


Fig 6. Porosity correlation graphic for glasspearl and adhesive petroleum samples

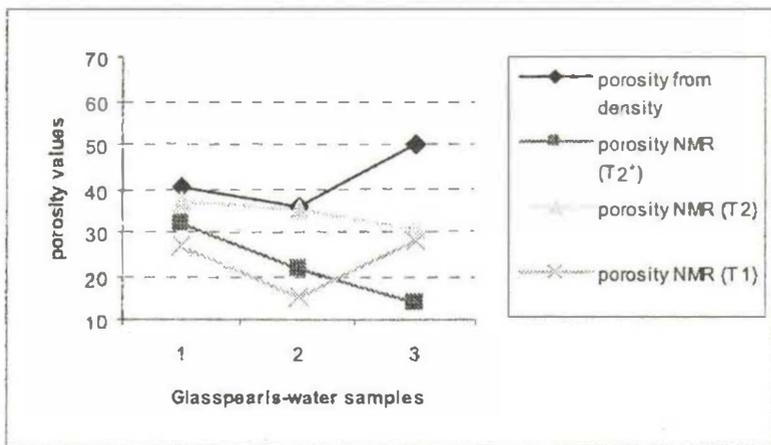


Fig. 7. Porosity correlation graphic for glasspearl and adhesive water samples

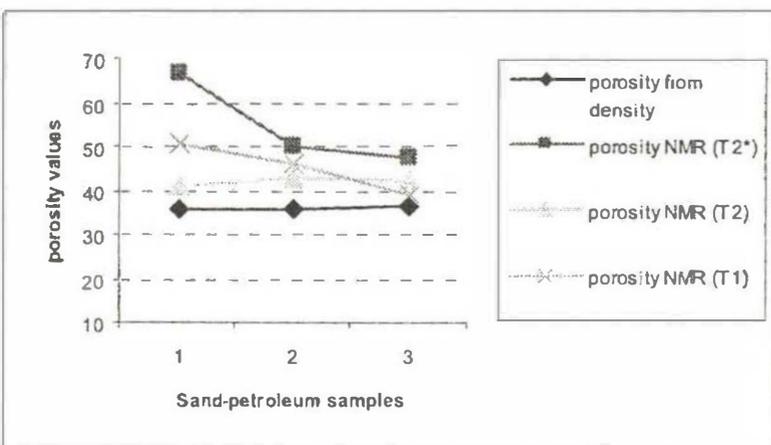


Fig. 8. Porosity correlation graphic for sand and adhesive petroleum samples

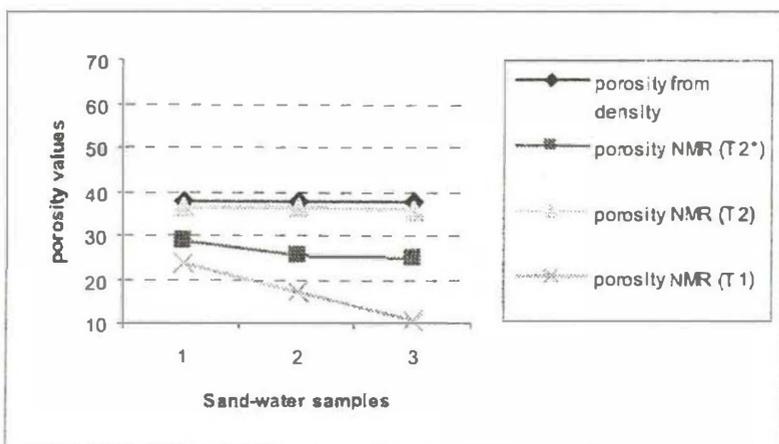


Fig. 9. Porosity correlation graphic for sand and adhesive water samples

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