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ScienceDirect

Russian Geology and Geophysics 57 (2016) 1509–1514

RUSSIAN GEOLOGY AND GEOPHYSICS

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Relation of NMR parameters with specific surface and resistivity of shaly sandstone and siltstone samples: experimental study

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Received 23 October 2015; accepted 29 January 2016

Abstract

Using an MST-05 NMR relaxometer, we determined the dependence of NMR parameters on the specific surface and resistivity of water-saturated shaly sandstone and siltstone core samples. The influence of the type and quantity of clay minerals was evaluated at residual water saturation, because the main contribution to the measured NMR signal in this case is made by clay content. Based on NMR relaxometry data, we obtained quantitative estimates of the specific surface with clay- and capillary-bound fluids. The surface relaxivity was estimated from thermal-desorption and NMR relaxometry data. The high degree of reliability of its values was confirmed by the agreement between the pore size distributions determined by NMR relaxometry and by capillarimetry and granulometry. We have established that the parameters of NMR spectra depend on the specific surface and resistivity, which are, in turn, a function of the surface properties of both clay and sandstone/siltstone particles.

Keywords: nuclear magnetic resonance; relaxometry; clay minerals; specific surface; surface relaxivity; resistivity

Introduction

Pulsed NMR relaxometry is one of the effective methods for laboratory studies of petrophysical characteristics of reservoir rocks. This method permits one to obtain qualitative and quantitative information about the structure of the pore space of geologic environment rapidly and without mechanical impact. Therefore, in the recent two decades, laboratory NMR methods, such as spectroscopy, relaxometry, and X-ray tomography, have been widely used in petrophysical studies of petroleum field cores (Galkin et al., 2015).

Effective porosity and portions of free and bound fluids are among the major parameters determined by NMR relaxometry. The content and type of clay minerals exert a significant effect on the NMR signal related to clay-bound water. Therefore, interpretation of results obtained for core samples with a high portion of clay fraction leads to errors in the determination of major petrophysical parameters (Aksel'rod and Neretin, 1990; Coates et al., 1999; Dzhafarov et al., 2002). Some researchers ignore the influence of clay component, considering its contribution close to a noise level (Kenyon, 1992). To reduce the error, an interpretational model of samples is commonly used, which takes into account their lithological and petrophysical characteristics (Chen et al., 2012; Peveraro and Thomas, 2010). At the same time, there are almost no works on quantitative estimation of the clay effect and on analysis of methods of NMR signal processing in studies of shaly core samples.

An NMR experiment in study of the clay effect

To elucidate the dependence of parameters of NMR spectra on the specific surface and resistivity, we performed experiments on a collection of samples with different petrophysical characteristics and different types and contents of clay component. The parameters of NMR spectra (hereinafter, NMR parameters) were determined on an MST-05 NMR relaxometer; the specific surface was measured by applying the thermal argon desorption method and was calculated via the Brunauer–Emmett–Teller (BET) method; resistivity was measured with the two-electrode method. The experiments were performed at 100% and residual water saturation achieved by centrifugation in a PC-6 centrifuge at 6000 rev/min for 45 min. The

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http://dx.doi.org/10.1016/j.rgg.2016.01.019

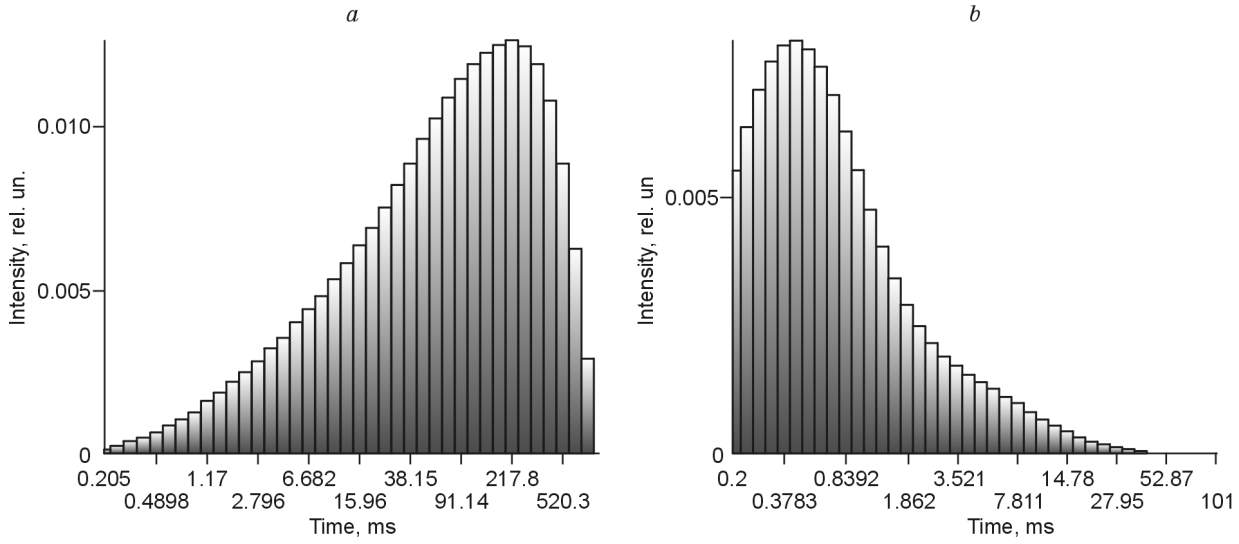

Fig. 1. Distribution of transverse relaxation time at 100% (*a*) and residual (*b*) water saturation.

NMR parameters were measured in two stages: (1) recording of an NMR signal as a relaxation curve and (2) its processing and interpretation.

Stage 1. Magnetization of a sample, or polarization of the magnetic moments of hydrogen nuclei, when the sample is placed in a magnetostatic field, which induces macroscopic magnetization directed along the applied field. Rotation of the macroscopic-magnetization vector into the transverse plane occurs under the impact of a 90º Carr–Purcell–Meiboom–Gill (CPMG) pulse sequence. A subsequent series of 180º pulses results in spin echoes; the curve enveloping them is the relaxation curve to be studied. Its initial amplitude corresponds to EMF in the receiver coil after the first 90º pulse and is proportional to the number of hydrogen nuclei in the sample; this number is then converted to the total NMR porosity (Coates et al., 1999; Venkataramanan et al., 2014).

Stage 2. Processing and interpretation of the relaxation curve consist in reverse transformation of an NMR signal into the distribution of transverse relaxation time (T_2), which characterizes the pore size distribution in fully fluid-saturated rock (Denisenko, 2012). The transformation is reduced to the solution of the Fredholm integral equation of the first kind:

$$\int_{T_{2\min}}^{T_{2\max}} e^{-t/T_2} z(T_2)\, dT_2 = f(t),$$

where T_2, $T_{2\min}$, and $T_{2\max}$ are the transverse relaxation time and its minimum and maximum values, respectively, t is the time of exposure to the static magnetic field, $z(T_2)$ is a function of the differential distribution of signal amplitudes by transverse relaxation time, and $f(t)$ is an NMR signal. The inverse problem is commonly solved by the Tikhonov regularization method. Subsequent interpretation consists in obtaining the information about the pore space structure, including the capacity and porosity parameters of the core samples.

To obtain a relaxation curve ensuring highly reliable results of its processing, it is important to choose the optimal parameters of the pulse sequence, such as the distance between echo pulses (*TE*), number of echo pulses (N_b), polarization time (T_w), and number of echo sequences (N). The *TE* value is specified as low as possible for recording signals from the smallest pores and fractions with short transverse relaxation time (clay-bound fluid). It was experimentally established that an optimal *TE* value for an MST-05 NMR relaxometer is 0.2 ms (Dolomanskii and Murav'ev, 2010). It is reasonable to increase it only if the signal described by the relaxation curve at the maximum N_b damps out incompletely. To obtain data on the large pores and fractions with large transverse relaxation time, the T_w value should be 3–5 times greater than the maximum transverse relaxation time for all hydrogen nuclei to be polarized. The N_b value is chosen in such a way as to ensure the damping required to complete the relaxation process at the specified *TE* value (Ivanov and Soshin, 2013).

As an example, let us consider the values of experimental NMR parameters at 100% and residual water saturation of a typical terrigenous sample, Lower Cretaceous fine-grained sandstone with porous shaly cement evenly distributed throughout the sample and consisting of chlorite-kaolinite and hydromica clay. Residual water saturation was obtained by centrifugation of the core sample at 6000 rev/min for 45 min. To compare the results of NMR measurements at 100% and residual water saturation, it is necessary to specify the same *TE* value in both cases, which will permit recording of signals from the same pores; $TE = 0.2$ ms. Other parameters are specified separately for each measurement so as to obtain a relaxation curve ensuring reliable results of its processing. For the above-discussed sandstone sample, $N_b = 400$, $T_w = 200$ ms, and $N = 500$ at 100% saturation, and $N_b = 150$, $T_w = 200$ ms, and $N = 400$ at residual saturation. The value of N is chosen in such a way that the signal/noise ratio is maximum at the optimal time of the experiment. The time of recording of a