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Rapid measurements of heterogeneity in sandstones using low-field nuclear magnetic resonance

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ABSTRACT

Sandstone rocks can contain microscopic variations in composition that complicate interpretation of nuclear magnetic resonance (NMR) relaxation time measurements. In this work, methods for assessing the degree of sample heterogeneity are demonstrated in three sandstones. A two-dimensional T_1–$\Delta\chi_{app}$ correlation (where $\Delta\chi_{app}$ is the apparent solid/liquid magnetic susceptibility contrast) reveals the microscopic heterogeneity in composition, whilst a spatially resolved T_1 profile reveals the macroscopic structural heterogeneity. To perform these measurements efficiently, a rapid measure of longitudinal T_1 relaxation time has been implemented on a low-field NMR spectrometer with a magnetic field strength $B_0 = 0.3$ T. The "double-shot" T_1 pulse sequence is appropriate for analysis of porous materials in general. Example relaxation time distributions are presented for doped water phantoms to validate the method. The acquisition time of the double-shot T_1 sequence is equivalent to the single-shot Carr–Purcell Meiboom–Gill (CPMG) sequence used routinely in petrophysics to measure transverse T_2 relaxation. Rapid T_1 measurements enable practical studies of core plugs at magnetic field strengths previously considered inappropriate, as T_1 is independent of molecular diffusion through pore-scale (internal) magnetic field gradients.

1. Introduction

Nuclear magnetic resonance (NMR) relaxation times are sensitive to the liquid/solid interface in porous materials. Transverse T_2 relaxation time measurements are used extensively in petrophysical rock core analysis [1,2]. The Carr–Purcell Meiboom–Gill (CPMG) pulse sequence [3,4] provides a rapid, single-shot method for measuring the exponential decay of transverse magnetization. In petrophysics, the CPMG sequence is most usefully applied at low magnetic field strengths, $B_0 \ll 0.1$ T, to minimize the effects of magnetic susceptibility contrast in these heterogeneous samples. A T_2 distribution has become the archetypal NMR well logging measurement, where analysis of the data provides estimates of porosity, permeability, and saturation state of the rock formation near the well bore [5]. When the pore structure contains only a single liquid, a relaxation time distribution is considered equivalent to a pore size distribution [6], assuming a single surface relaxivity parameter controls the scaling between relaxation time and pore size. Historically, sandstones have been studied by NMR because of the simplicity of their granular structure, although the mineralogy of sandstones will often be complicated. When the sandstone contains spatially localized mineral deposits (e.g., feldspar, clays, or metal oxides), a distribution of surface relaxivities will exist, which complicates the interpretation of relaxation time distributions [7].

NMR measurements are sensitive to variations in local sample composition through (a) surface relaxivity and/or (b) magnetic susceptibility contrast $\Delta\chi$ between the solid pore matrix and the saturating liquid. The T_2 measurement is influenced by both surface relaxation and diffusion through pore-scale "internal" magnetic field gradients caused by the susceptibility contrast [8]. Diffusive signal attenuation results in the observation of an effective relaxation time, $T_{2,\mathrm{eff}}$. The magnitude of the internal gradients is determined by $\Delta\chi B_0$, so minimizing the magnetic field strength reduces the influence of diffusion on the T_2 measurement. Laboratory-scale core analysis is routinely performed at $B_0 = 50$ mT, corresponding to $\nu_0 = 2$ MHz for ^{1}H, (a) to minimize internal gradient effects and (b) to ensure robust calibration of well logs acquired at a similar Larmor frequency.

The sensitivity of NMR experiments to microscopic variations in composition increases with magnetic field strength as both surface relaxivity [9] and internal gradients [8] are field dependent. In the present work, a $B_0 = 0.3$ T magnet is used to enhance sensitivity to structure in the NMR experiment. This field strength was previously considered inappropriate for core analysis of sandstones because of the effects of internal gradients [10].

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Notwithstanding, quantitative information is obtained through measurements of the longitudinal T_1 relaxation time. T_1 was the original metric of choice in NMR petrophysics [6,11] due to hardware constraints in well logging tools and the robustness of the T_1 measurement [10,12]. In well logging, T_1 was later rejected in favor of T_2: the single-shot CPMG measurement has sufficient time resolution to be useful on moving tools [13]. Laboratory-scale core analysis followed this trend, and now low-field measurements of T_2 are considered the industry standard.

In the laboratory context, it is usual to acquire T_1 data using the conventional inversion or saturation recovery protocols [14]. However, several rapid T_1 measurement protocols are available, as reviewed in [15] with recent method developments given in [16–22]. The double-shot T_1 measurement has an acquisition time comparable to that of the CPMG sequence [23]. In the double-shot T_1 experiment, the relaxation time is encoded as an exponential decay following two radio frequency (rf) excitation pulses. In the present work, the double-shot T_1 experiment (see Section 2) is validated for use on a low-field magnet. Quantitative T_1 distributions are presented for doped water phantoms (see Section 3) and for sandstone core plugs (see Section 4).

To determine microscopic heterogeneity in sample composition (mineralogy), a T_1–$\Delta\chi_{app}$ correlation is introduced. $\Delta\chi_{app}$ is an apparent solid/liquid magnetic susceptibility contrast, obtained from a measurement of T_2^*, the transverse relaxation rate determined by local field inhomogeneities [24,25]. An apparent susceptibility contrast is obtained because the measured T_2^* is influenced by details of the local pore geometry as well as the local magnetic field [25]. Localized variations in composition (occurring over length-scales much greater than the diffusion path-length of water) should result in observable variations in $\Delta\chi$. Furthermore, these same localized changes in composition will provide variations in surface relaxivity. $\Delta\chi_{app}$ (through T_2^*) and T_1 offer independent probes of these two composition-dependent parameters. A T_1–$\Delta\chi_{app}$ correlation therefore offers a straight-forward visual representation of heterogeneity: a strong inverse correlation between the two dimensions is indicative of compositional variation. Elsewhere, T_1 has been correlated against effective internal gradient strength g_{eff} [12], whilst $T_{2,eff}$ has been correlated against $\Delta\chi$ [26]. However, in the former experiment, g_{eff} is difficult to determine in general for sandstones [27], and in the latter experiment, both dimensions are dependent on the internal gradient distribution. The T_1–$\Delta\chi$ correlation provides direct information on the microscopic compositional heterogeneity as it unambiguously correlates magnetic susceptibility contrast with surface relaxation.

Macroscopic structural heterogeneity is explored by incorporating imaging gradients into the double-shot T_1 sequence to enable rapid acquisitions of T_1 profiles. Spatially resolved T_1 measurements have been used previously to study drilling mud filter cakes [28] and the invasion of clays and drilling mud into core plugs [28–30] where the presence of paramagnetic species (e.g., iron in barite used as a densifying agent in mud) makes conventional T_2 analysis inappropriate [1]. Rapid methods of mapping T_1 will enable time-resolved studies of such invasion processes and also permit monitoring of oil recovery [31] at magnetic field strengths previously considered inappropriate for petrophysical core analysis.

2. Experimental methods

2.1. Pulse sequences

All NMR measurements were performed on an Oxford Instruments $B_0 = 0.3$ T vertical bore, bench-top permanent magnet operating at $\nu_0 = 12.9$ MHz for ^{1}H. Imaging gradients up to $g_{x,y,z}^{(max)} = 25$ G cm^{-1} were provided by three-axis slab-format gradient coils. The gradient coils were driven by high-performance ERA audio amplifiers. A field homogeneity of $\Delta\nu_0 = 5$ ppm was achieved over the entire sample volume (cylindrical rf probe of dimensions length × diameter: 6.0 cm × 5.1 cm) by high-order shims. The experiments were controlled by an Oxford Instruments Maran DRX-HF spectrometer using an interface written in the Matlab programming environment.

The original double-shot T_1 pulse sequence, Fig. 1(a) [23], was based in part on the T_1 by multiple read-out pulses (TOMROPs) sequence [32]. However, where the TOMROPs sequence generates a relaxation curve defined by a complicated function of T_1, the total magnetization M_0, and the driven equilibrium magnetization M_∞^{eq} (following an "infinite" number of small tip angle rf pulses), the double-shot sequence generates a simple exponential decay that depends only on T_1 and M_0. In [23], a 90°–180°–90° preconditioning sequence was used to store the magnetization alternately along $\pm z$ so that after two scans, the signal amplitude was simply $b(t) \propto M_0 \exp\{-t/T_1\}$, where t is the total relaxation time.

Here, a modified version of the double-shot T_1 sequence is introduced, as shown in Fig. 1(b). The first portion of the pulse sequence consists of a series of small tip angle α pulses (such that $\sin\alpha \approx \alpha$), each pulse being separated in time by τ_1. A complete free induction decay (FID) [33] is acquired immediately after each α pulse. This portion of the pulse sequence measures M_∞^{eq} obtained during the train of n pulses. A comb of 90° pulses is then applied to fully saturate the spin ensemble in the x–y plane. A second train of α pulses is used to observe the recovery of the magnetization from zero to M_∞^{eq} on the z-axis. In a post-processing stage, the driven equilibrium signal (acquired in the first portion) is subtracted from the recovery curve (acquired in the second portion) to provide an exponential relaxation decay. The normalized signal amplitude is then given by

$$\frac{b(t)}{b(0)} = \exp\left\{-\frac{t}{T_1}\right\}(\cos\alpha)^{n-1}\sin\alpha, \tag{1}$$

with

$$t = (n-1)\tau_1 + t_{spoil}, \tag{2}$$

where t_{spoil} is the 90°-α delay required to apply a homospoil gradient pulse. To prevent the refocusing of unwanted magnetization, homospoil gradient pulses of variable amplitude are included between each α pulse. To guarantee quantitative signal amplitudes, it is only necessary to provide a recycle delay of $t_{RD} \geqslant \tau_1$ between each acquisition if the magnetization has recovered to M_∞^{eq} at the end of the acquisition. Otherwise, the condition $t_{RD} \gg \tau_1$ is required. The modified double-shot T_1 sequence has a slightly reduced acquisition time (t_{RD} is included only once every two scans) compared to the original implementation (t_{RD} is included between every scan). For practical implementation on low-field hardware,

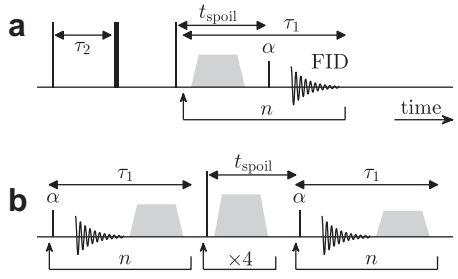

Fig. 1. Double-shot T_1 pulse sequence schematic. The original pulse sequence (a) was implemented at high field, and the modified pulse sequence (b) is used here at low field. The thin and thick vertical lines indicate 90° and 180° rf pulses, respectively, with the small tip angle (α) pulses indicated by vertical lines of reduced amplitude. The gray trapezoids indicate homospoil gradient pulses. The time between successive α pulses is τ_1, and the 90°-α delay is t_{spoil}. The half-echo time in (a) is τ_2.