NMR Core Analysis On Whole Core Samples

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Abstract. Nuclear Magnetic Resonance (NMR) has proven to be an important tool in the core analysis laboratory. Traditional NMR core analysis is restricted to core plug sizes (1" to 1.5" in diameter and ~2" in length). NMR analysis of whole core has been restricted by the size of typical NMR instruments and, more importantly, signal interference from portions of the core sample that are outside the probe's field of view. This overestimates NMR porosity and creates unusable images and profiles with 2-D and 3-D measurements.

In this paper, new methods for performing quantitative NMR core analysis on whole core samples are presented. For bulk measurements, such as T_2 distributions or T_1-T_2 maps, one method accounts for the signal outside the field of view through calibration using a sample of known volume that is larger than the probe field of view. The NMR volume of the sample is then compared with the known volume of the sample. Any difference between the two volumes is due to signal being folded in from outside the field of view. The ratio of the observed NMR volume to the known volume of the calibration sample is a correction factor, this can be applied to all whole core measurements done with the NMR probe. This technique has been successfully applied to measurements of T_2 and T_1-T_2 maps on different whole core samples.

This calibration method works well for bulk measurements but is ineffective for imaging because the signal from outside the field of view spatially interferes with the signal within the field of view. This spatial interference leads to image distortion and erroneous results. NMR pulse sequences supressing signal from outside the field of view improve profile measurements and maintain quantitative capabilities while eliminating sample blurring.

1 Introduction

NMR rock core analysis is an important tool in reservoir evaluation. Coring oil and gas deposits extract long sections (normally 4" diameter) of core from the well to the surface. Care is taken to preserve the core in its native state by sealing it with wax and storing it for core studies in a core analysis lab. Most core analysis is performed on plug samples extracted from the larger core. Typically, these plugs are taken at strategic locations obtaining standard 1" or 1.5" diameter samples for testing. Depending on rock type, plugging a core can be a challenge due to cracking, fracturing, or even pulverization by the plugging drill bit; it can be difficult to obtain proper cylindrical core plugs of sufficient length. Additionally, friction between the bit and the core generates considerable heat causing fluids in the preserved rock to evaporate and problems with saturation measurements. Also, obtaining plugs from whole core can involve drilling fluids that can change the original saturation. Whole core NMR analysis is appreciated for its ability to quantify fluids in an undisturbed state.

Depending on the plugging interval, core plugs can be affected by heterogeneity, skewing the description of the core and upscaling efforts. The ability to measure porosity and saturations continuously along whole core is advantageous compared to discrete plug samples, especially when trying to match core to logs.

Addressing issues arising from plugging whole core, we propose using long sections of whole core for NMR analysis. Using the whole core comes with its own set of unique challenges: a larger NMR spectrometer is required, increasing complexity with moving the bulky core through the NMR field of view, and sample preparation is more difficult because of its increased size.

Our approach to NMR long core experiments consists of two parts, bulk measurement – to measure porosity of a section; and an image measurement – to measure saturations along the section. Bulk measurements are obtained by using an NMR T_2 [1] test determining the signal in the maximum field of view. Details on how this is achieved and calibrated is explained in the following section. Image measurements can be obtained by using one-dimensional saturation profile tests [2, 3]. For imaging long core samples, an out of field of view signal suppression technique is required because the sample exceeds the length of the NMR probe. A specialized pulse sequence [4] suppresses signal folding in from outside the field of view. This specialized sequence is outlined in the following section.

2 Experiment

All NMR data for this experiment is recorded with an Oxford Instruments 8 MHz NMR spectrometer [5] using a 100 mm diameter probe. The spectrometer has magnetic gradients in all three directions (X, Y and Z) allowing full

profile measurements. Gradients allow for localized measurements of small volumes by dephasing protons outside the volume of interest; this allows for "scanning" data acquisition where volumes or "slices" are acquired sequentially. Green Imaging Technologies software was employed for data acquisition, inversion, reconstruction, and analysis [6].

Two pieces of whole core (from the same core) were used for this study, one long (40 cm) and one short (10 cm). The core was from a well from the Sweetwater region of Wyoming at a depth range of 7140-7145 feet. Table 1 summarizes the physical properties of each sample and Figure 1 shows an image of the longer core. Prior to the NMR scans, each piece of core was saturated with 2% KCl brine. 2% KCl was chosen to prevent dissolution of the sample by pure water. Saturation was performed in a high-pressure vessel with an overburden pressure of 10,000 PSI. The porosity data summarized in Table 1 was derived from the NMR data. This is the most accurate measure of porosity recorded on these samples. Porosity derived from mass data is prone to error such as grain loss which can affect the porosity derived. A hydraulic lift was employed to control the movement of the longer core through the spectrometer.

Core Sample	Short	Long
Core Diameter (cm)	6.70	6.66
Core Length (cm)	10.18	40.0
Bulk Volume (mL)	358.9	1393.4
Porosity (p.u.)	11.94	11.52

Table 1: Sample Information

Figure 1: Long Sample

3 Results

3.1. Calibration of field of view

The first step of bulk measurements on whole core is mapping the probe field of view. A 10 cm diameter and 1 cm long glass vial is filled with a calibration fluid containing a mixture of $CuSO_4$ in water. $CuSO_4$ is paramagnetic and reduces the bulk relaxation time of the fluid. The pancake shaped calibration sample is laid flat in the magnet's field and is moved along its vertical axis in 1 cm steps. At each step, a T_2 test is used to measure the observed volume. The change in observed volume vs. displacement along the vertical axis (Figure 2 – Red Trace)) shows that the probe does not have a field of view with sharp edges. Instead, the observed signal rolls off more gradually along the vertical axis of the magnet. This makes assigning a fixed field of view more difficult. Based on the map plotted in Figure 2, the probe field of view is estimated to be 14 cm (±7 cm) (Figure 2 – Blue Box). Clearly, this is just an estimate of the field of view because samples > 14 cm will have some fraction of their total observed signal from contributions beyond the field of view estimated as ±7 cm. The derivative also shows that samples within the field of view do not uniformly contribute to the total observed signal. For example, material at a displacement of -5 cm contributes only 75% of the equivalent amount of material at 0 cm of displacement.

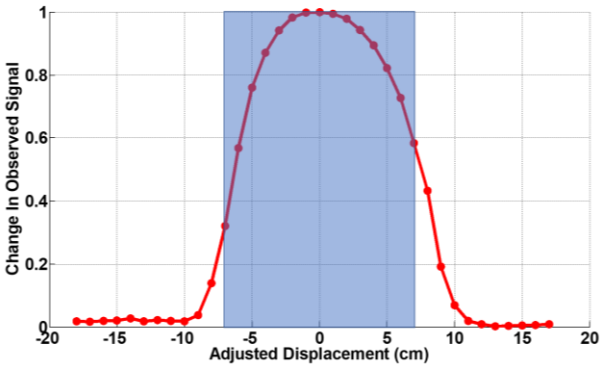

Figure 2: Probe field of view measurement profile.

To complete a bulk NMR measurement on a long piece of core, varying contributions to overall signal intensity along the vertical axis of the magnet need to be calibrated. To do this, a Teflon tube (62 cm in length and 3.71 cm in diameter) is filled with calibration fluid ($CuSO_4$) and centred within the field of view. A T_2 acquisition then measures the observed volume of the fluid in the tube within the field of view. As shown in Equation 1, the observed NMR volume was compared to the geometric cylinder volume whose diameter is the same as the Teflon vial and length is in the field of view of the NMR probe. This is the effective volume the NMR measurement should observe.

$$\frac{NMR\ Volume}{Geometric\ Vol} = \frac{NMR\ Volume}{\pi r^2 l} = \frac{156.44\ ml}{151.3\ ml} = 1.03367 \quad (1)$$

The ratio of the observed NMR volume to the geometric cylinder volume is the correction factor applied to any NMR measurements taken of core longer than the field of view of the magnet. In other words, the observed NMR volume overestimates the true geometric volume by approximately 3%. As a result, the observed NMR volume should be divided by this calibration factor to compensate the true volume of core within the field of view. Because the calibration constant was determined using a long uniform liquid calibration source, this method assumes that the core sample is also homogeneous along its length and is longer than the field of view of the magnet.. However, this method can be applied to any rock type (conventional or unconventional) if it is homogeneous. In addition, experimental conditions such pressure or temperature will have no effect on the calibration.

3.2. Porosity measurement of long core using calibration factor

To test the validity of employing a correction factor to determine the porosity of long core samples, validation measurements are performed on the two samples described in Table 1. First, the short sample is placed in the magnet and its T_2 distribution is measured (Figure 3 – red trace). The derived pore volume is then divided by the samples bulk volume to give a porosity of 11.94 p.u. The length of the short sample is completely within the field of view of the NMR probe, so no correction factor is needed to account for signal contributions outside the field of view.

Next, the long sample is placed in the magnet and its T_2-distribution is measured (Figure 3 – Blue Trace). The measured pore volume is divided by the geometric volume of the field of view (Geometric Volume = $\pi r^2 l$ = $\pi(6.66\text{ cm})^2(14\text{ cm}) = 487.7$ ml). Note that we are dividing by the geometric volume of the field of view here and not the bulk volume of the sample. This is necessary because the sample extends beyond the field of view of the magnet. Because the sample length is beyond the NMR probe field of view, the pore volume is also divided by the correction factor. The porosity of the long core sample is determined to be 11.52 p.u. The good agreement between the porosity derived for the long and short samples validates employing the calibration method for bulk measurements on samples longer than the probe's field of view.

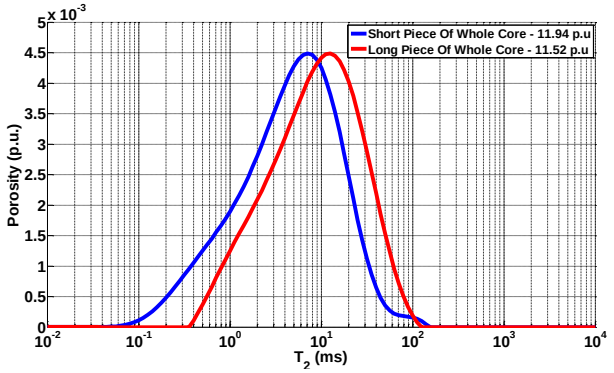

Figure 3: T_2 relaxation time distribution of long and short core samples

Figure 4 shows the T_1-T_2 distributions of the long and short samples. T_1-T_2 maps are also bulk measurements so the same calibration factor employed to correct the T_2 distributions can be employed to rectify the long core data. Employing the correction factor the porosity determined for the long sample from the T_1-T_2 data was 12.07 p.u. which was in good agreement with the porosity derived from the T_1-T_2 data of the short sample (12.53 p.u.) More importantly, the two maps plotted in Figure 4 are almost identical. This means T_1-T_2 maps can be effectively employed for core analysis such as fluid typing.

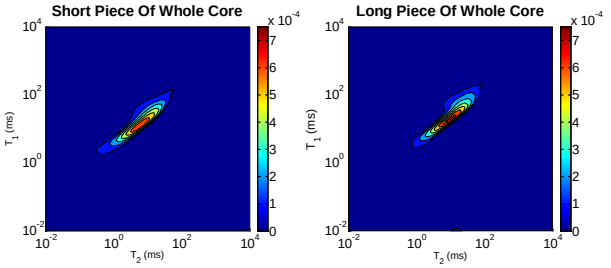

Figure 4: T_1-T_2 maps of long and short core samples. The similarity between the plots validates the normalization techniques for samples exceeding the NMR's probe field of view.

While employing a correction factor to NMR relaxation measurements on whole core samples is effective and accurate, it is far from ideal. As mentioned earlier, it assumes homogenous core samples and any heterogeneity will lead to inaccurate results. For example, the small differences observed in the T_2 distributions (Figure 3) and the T_1-T_2 maps (Figure 4) for the long vs. short core samples can be attributed to inhomogeneity of the sample. Figure 1 shows that our sample was not perfectly homogeneous. Additionally, correction factors cannot be applied to NMR images, i.e. it's not effective for regions close to the ends of the core. The correction method assumes that there is sufficient material on either side of the field of view. If not, then the assumptions made in determining the correction factor are invalidated.

3.3. Region of interest selection for whole core measurement

Because of the shortcomings of the calibration method just outlined, an exploration of a method for a region of interest selection of long core samples was examined based on insights developed by Vashee et al. [4]. These techniques can be applied to any NMR/MRI pulse sequence allowing any bulk or imaging technique to be adapted for long core samples. In our case, validation began by applying it to one-dimensional saturation profiles. Saturation profile measurements produce a 1D image of a rock core by collapsing (or summing) the signal in the 2nd and 3rd dimension. . There are several different methods to acquire a one-dimensional profile [2,3] and an out of volume suppression technique can be applied to most of them. Without this suppression, samples that extend beyond the useable volume of the probe and/or linear region of the magnetic field gradients will create profiles that are unusable, as signal from outside the probe folds back into the measured profile, making the profile useless. The black trace in Figure 5 shows a saturation profile acquired on the long core sample without the out of volume suppression turned on.

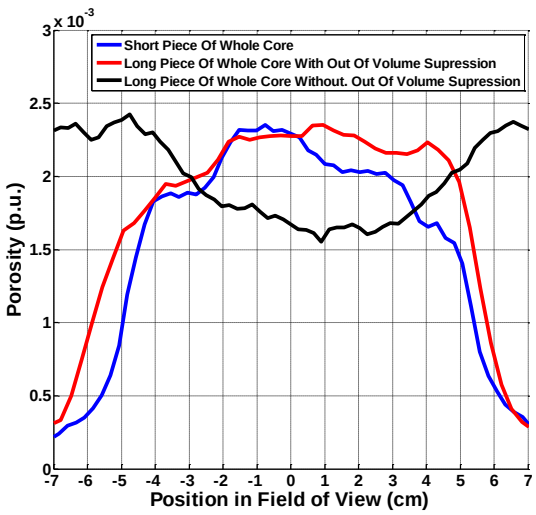

Figure 5: Saturation profiles of long and short core samples. Also shown is the saturation profile recorded for the long core sample without the use out of volume suppression.

Out of volume suppression mitigates signal from outside the volume of interest. This works by taking two profiles, one normally and one with a spatially selective inversion pulse on the front end [4] The spatial inversion pulse is performed in the presence of a magnetic field gradient to excite the desired region of the profile. The two profiles are then subtracted (in the time domain) and only signals from within the selected inversion slice are added, all other signals are subtracted. The selective inversion pulse is a slice selective adiabatic inversion pulse (hyperbolic secant pulse [4]). The adiabatic nature of the pulse ensures correct inversion even in the presence of radio-frequency (RF) inhomogeneity due to limitations of the RF coil. The duration between the normal profile sequence and the inversion pulse is about 1 ms. Therefore, if the T_1 of the sample is less than about 3 ms, this technique will fail to fully suppress out of volume signal. The technique also doubles the scan time, although the extra scan increases signal to noise by a factor of the square root of two. This is because in the volume of interest the signal is measured twice (once with the normal saturation profile and once with the spatially selective inversion pulse). When the two profiles are then subtracted, the profile has an increase in signal to noise of the square root of two as any signal does when you measure it twice.

A saturation profile of a long sample with out of field suppression is shown in Figure 5 (red trace). In this profile example, the region of interest is chosen as 11.2 cm or eighty percent (80%) of the field of view (14 cm). Also shown in Figure 5 (blue trace) is a saturation profile recorded on the short core sample without the suppression technique applied. Out of volume suppression was not necessary with the shorter sample as it did not extend beyond the field of view of the probe. The good agreement between the porosity per cm of both core sample lengths support that out of volume suppression techniques give accurate results. The length of the short sample as determined by the saturation profile (Figure 5 – blue trace) is also accurate. The length of the short core sample is known to be 10.18 cm and should fall within the region of interest (11.2 cm) probed by the out of volume suppression measurement of the long core sample (Figure 5 – red trace).

4 Conclusion

We have successfully measured the T_2 relaxation time distribution and T_1-T_2 maps of long core samples by employing a calibration method for handling signal from outside the field of view. The T_2 relaxation time and T_1-T_2 map measured from a long core sample were identical to those measured with a short sample (shorter than the probe's field of view) taken from the same core validating the method.

The bulk calibration method has some shortcomings; it requires a homogeneous sample and cannot be applied to NMR images. To circumvent these problems, an NMR pulse sequence that suppresses contributions to the observed signal from portions of the sample outside the probe field of view is used. We have successfully employed this out of volume suppression technique to measure a saturation profile on a long core sample. Next steps include expanding the out of volume suppression for use beyond one dimensional saturation profiles to two- and three-dimensional images. Additionally, volume suppression methods to T_2 distributions and T_1-T_2 maps to create slice selective version of these bulk measurements are being implemented.

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