Improved Methods for Determination of Petrophysical Properties of Unconventional Tight Rocks Using Particulate Samples

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ABSTRACT: Drill cuttings are available continuously over the entire depth of any drilled wells. The use of drill cuttings to obtain petrophysical data can add a significant value in formation evaluation. An update to the previous study is presented using NMR and Archimedes principle to determine petrophysical properties including porosity, bulk density, and matrix density from drill cuttings of organic-rich mudrock formations. In the original published method, sonication was used to clean and saturate the drill cutting samples. In this improved method, particulate samples were saturated using both sonication and pressure injection methods. The obtained results were compared with accepted lab measurement techniques. Results show that pressure saturation of particulate samples can provide accurate results of petrophysical properties. Obtaining reliable petrophysical data from drill cuttings can provide continuous and quasi-real-time data for the formation evaluation of reservoirs and can effectively reduce costs by reducing or eliminating expensive formation evaluation methods.

INTRODUCTION

Conventional methods for evaluating petrophysical parameters of a reservoir include well-log measurements or laboratory core measurements. However, acquiring logs and/or cores can be expensive, and therefore, the data are obtained in only a few wells at selected sections. On the other hand, drill cuttings are available for all of the wells and can be continuous over the entire interval of the well. Therefore, the use of drill cuttings for petrophysical evaluation can provide a detailed representation of the formation, which can be critical for thinly laminated organic-rich mudrock formations.

Furthermore, real-time drilling and reservoir monitoring require a fast and continuous input of petrophysical data. Therefore, a reliable method to extract the required parameters from drill cuttings at the well site is essential. The use of drill cuttings in reservoirs for formation evaluation has been widely investigated. Drill cuttings have been used to determine properties of the rock such as mineralogy,1 wave velocity,2 density/porosity,3 and permeability estimates.4 Cutting samples have also been imaged using scanning electron microscopy (SEM) and properties explored using digital rock physics.5,6

Routine core analysis measurements, such as mineralogy from X-ray diffraction (XRD) or X-ray fluorescence (XRF) and organic content from pyrolysis, have been performed on cuttings from organic-rich mudrocks.7 However, the presence of nanopores and organic matter in source rock reservoirs can make measurements of bulk density and porosity challenging. The accurate estimation of properties from drill cuttings is also dependent on the removal of contamination from drilling mud and other heavy elements present in the drilling mud. Moreover, for the reliability of the cutting analysis, it is essential to consider the drilling fluid used for drilling the wells.

A variety of NMR techniques have been used to investigate cutting samples,8–12 including methods to mask solvents using deuterium or fluorine and multidimensional methods. In a recent study, we used a combination of NMR measurement techniques and Archimedes principle to obtain petrophysical parameters, including porosity, bulk density, and matrix density, of irregularly shaped drill cuttings. In the same paper, we used sonication to remove the drilling mud and other contaminants from the drill cutting surface13 and as a method for saturating the samples. Although this method for sample saturation gives reliable estimates of petrophysical properties for many samples, there was an underestimation of...
bulk density in some samples, potentially due to incomplete saturation of the drill cuttings.

Source rocks contain nanosized pore throats with high capillary entry pressures. Therefore, to completely saturate the samples, the pressure applied should overcome the capillary entry pressures. The capillary entry pressure of various nonwetting fluids for some shales has been estimated to range from 150 to 950 psi. Saturation pressures of up to 7000 psi have been used to saturate shale cuttings to estimate porosity based on capillary pressure estimates for pore throats of around 1 nm. In another study, complete resaturation of the shale core samples was achieved at 2000 psi pressure saturation.

In this paper, we improve the accuracy of our previous method of estimating petrophysical properties from drill cuttings using pressure saturation. We compared the results from both the original and improved methods to industry-standard practices for crushed rock using the Gas Research Institute (GRI) techniques. We found that the results of our improved method agree well with those obtained from GRI.

**METHODS**

**Samples.** For the purpose of validating the method, we used crushed particulate samples instead of drill cuttings. We chose crushed samples because part of the crushed material was also used for GRI measurement, therefore allowing ready access to data for comparison that would not be available for drill cuttings. In addition, we did not have to worry about deformation of the drill cuttings from the PDC drilling bit that generates additional porosity. As a result, we can focus solely on the effect of the saturation mechanism on the measured results.

Analysis was performed on 10 samples obtained from a source rock reservoir. The samples were crushed and sieved to a size between 0.5 and 3 mm to mimic the size of drill cuttings. First, an approximately 20 g portion of each sample was dried at 110 °C for 96 h. The samples were then saturated with diesel using the sonication method, as described in Althaus et al. for 24 h. NMR was then acquired on the saturated samples, followed by mass measurement of the samples performed in air and submerged in diesel. Next, we saturated the samples with diesel using a pressure cell. The NMR and mass measurements were then repeated.

**Saturation.** The samples were first saturated using sonication by placing the samples in a mesh container with a mesh size less than 0.5 mm to ensure that samples are contained. The mesh containers are placed in a beaker filled with diesel, which were placed in a sonicator (Bandelin Sonorex Digitec with sonoshake). The samples are sonicated in an ultrasonic bath with an ultrasonic frequency of 35 kHz in a continuous mode for 24 h.

For pressure saturation, the samples were placed in a Teflon bottle, with perforations on top of the bottle. The bottles were placed in an autoclave pressure cell filled with diesel, and the pressure was ramped to 3500 psi over the course of 3 h using an Isco pump. The pressure was then held at a constant pressure of 3500 psi for 7 days. The pressure was then released back to 0 psi slowly over 3 h. The samples were then removed from the pressure vessel, and the excess fluid was decanted. A nonperforated Teflon lid was then placed on the bottle to seal the sample for NMR measurement. A diagram of the setup is shown in (Figure 2).

**NMR Measurements.** NMR measurements were performed to determine the fluid volume within the pore and surrounding the sample. A 12 MHz Oxford NMR spectrometer with a resonant frequency of 13.07 MHz equipped with Green Imaging Technology software was used to measure the fluid contents in the samples. The samples were measured in Teflon bottles using a CPMG pulse sequence with parameters: 106 μs echo-time, 47170 data points, 12.2 μs 90 pulse, 24.2 μs 180 pulse, 7.5 s recycle delay, and 16 accumulation of scans. The NMR relaxation decay curves were processed using a Laplace transform in Green Imaging

![Figure 1. Workflow used to test the saturation methods and obtain petrophysical properties from cuttings.](https://doi.org/10.1021/acsomega.1c07034)
Technology software. A cutoff of 80 ms was used to separate bulk diesel between particles from pore diesel.

Mass Measurement. The mass of the Teflon bottle used for containing the samples was first measured in and out of the diesel using a Mettler-Toledo balance. The mass of each sample was measured in Teflon bottles in air after removal from the NMR. Then, the bottle containing the sample was submerged in diesel and measured in the fluid. The sample mass was obtained by subtracting the mass of the Teflon bottle from the total mass.

Densities and Porosity from the Measurements. The results from NMR and mass measurement were then used to determine the bulk density, matrix density, and porosity. A detailed description of the method is in ref13, specifically.

The bulk density of the samples is calculated as

\[ \rho_b \approx \frac{m_{\text{air}} - V \rho_t}{V_c} \]  

where \( m_{\text{air}} \) is the mass of the sample in air, \( V \) is the volume of the surrounding diesel obtained from the NMR long relaxation peak, \( \rho_t \) is the density of diesel, and \( V_c \) is the volume of cuttings defined as

\[ V_c = (m_{\text{air}} - m_t - V \rho_t)/\rho_t \]  

where \( m_t \) is the mass of the sample in the fluid. Matrix density is calculated as

\[ \rho_m = \frac{m_{\text{air}} - (V \phi + V_t)\rho_t}{V_m} \]  

where \( V \phi \) is the pore volume obtained from the NMR short relaxation peak and \( V_m \) is the volume of the solid matrix that can be obtained from

\[ V_m = (m_{\text{air}} - m_t - V \phi)/\rho_t - V \phi \]  

Porosity is calculated as

\[ \phi = \frac{V \phi}{V_c} \]  

Industry-Standard Measurements. The bulk density, grain density, and porosity measurements of the samples were also analyzed by an external vendor using the GRI method.17,21 The results obtained from the workflow described above are compared with the vendor results, reported as Vendor GRI. The petrophysical properties obtained from the GRI measurement of the samples are shown in Table 1.

Relative Error Calculation. The effectiveness of pressure and sonication saturation is determined by a comparison to GRI measurements of the same samples. The relative error from the GRI is used to assess the results. The relative error in the results from NMR–Archimedes method compared to the GRI results is calculated as follows

\[ \text{error (\%)} = \left| \frac{\rho_{b,NMR} - \rho_{b,GRI}}{\rho_{b,GRI}} \right| \]  

where \( \rho_{b,NMR} \) is the density/porosity measured using the NMR–Archimedes method, and \( \rho_{b,GRI} \) is the density/porosity obtained from the GRI method.

RESULTS AND DISCUSSION

This method requires that the amount of fluid outside the cuttings is sufficiently large so that the \( T_2 \) is long enough to be separated from the short \( T_2 \) peak of the pore fluid. Figure 3 shows the NMR \( T_2 \) spectra of a representative sample saturated using sonication and pressure. An 80 ms cutoff was used to separate the pore fluid inside the cuttings and bulk fluid outside the cuttings. The volume of pore fluid is then the integration of the \( T_2 \) spectrum between 0 and 80 ms. From Figure 3 (inset), we see an increase in the pore fluids when using pressure saturation.
Results from the Two Saturation Methods. The NMR-measured pore volume and bulk volume are then combined with the weight to determine the bulk density, matrix density, and porosity (Table 2). The bulk density is calculated using eqs 1 and 2. The pressure saturation leads to an increase in the bulk density that is more in line with typical values for unconventional source rocks.

The matrix density is calculated using eqs 3 and 4 with NMR and weight values. We again see an increase in the measured matrix density of the pressure-saturated samples that aligns better with typical values for these types of rocks.

The porosity is calculated using eq 5. The difference in the porosity between the two saturation methods is much smaller than the density changes. The porosity appears to be much less sensitive to the saturation method.

Validation of Measured Results. Figure 4 plots the comparison of bulk density results obtained from the workflow of the current study with vendor GRI bulk density results. As previously reported, the bulk density results are underestimated when compared to GRI results for samples that were saturated with sonication only. However, the results show that the measured bulk density for samples saturated with pressure is in good agreement with vendor-reported GRI results.

The samples saturated with sonication showed an average relative error of 8.0% when compared to vendor GRI bulk density. The relative error between NMR bulk density and vendor GRI bulk density decreases significantly to 1.6% when the samples are saturated with pressure. This indicates that the samples are not completely saturated when using sonication, and pressure saturation is required to saturate the samples to obtain accurate estimates of the bulk density of samples.

Figure 5 shows the comparison of matrix density results obtained from the workflow with vendor GRI grain density.

![Figure 4](https://doi.org/10.1021/acsomega.1c07034)

**Figure 4.** Cross plot of the bulk density of the crushed rock samples from different methods. The line represents the 1:1 density result measured by a vendor. Orange dots and blue triangles represent the NMR-derived bulk density for samples saturated with pressure and sonication, respectively, plotted on the y versus the vendor data on the x axis.

![Figure 5](https://doi.org/10.1021/acsomega.1c07034)

**Figure 5.** Cross plot of the matrix density of the crushed rock samples from different methods. The line represents the 1:1 density result measured by a vendor. Orange dots and blue triangles represent the NMR-derived matrix density for samples saturated with pressure and sonication, respectively, plotted on the y versus the vendor data on the x axis.

The results show that the matrix density obtained for samples saturated with pressure is in better agreement with the industry-standard GRI grain density than the matrix density obtained when the samples are saturated by sonication. The samples saturated via sonication show a significant underestimation of matrix density as compared to that of the vendor GRI matrix density.

The relative error between the matrix density of samples obtained from NMR–Archimedes technique and industry-standard technique decreases from 9.9 to 1.7% when the samples are saturated with sonication and pressure, respectively. A close agreement of matrix densities between pressure-saturated samples and vendor results indicates that pressure...
saturation should be used for the reliable estimation of matrix densities using the workflow.

Figure 6 shows the comparison of porosity results obtained from the workflow with vendor GRI porosity. The results show that the porosity of pressure-saturated samples is in better agreement with the industry-standard GRI porosity when compared to the sample porosity obtained when the samples are saturated with sonications.

The relative error between sample porosity obtained from the NMR–Archimedes technique and industry-standard technique decreases from 22 to 13% when the samples are saturated with sonications and pressure, respectively. The better agreement between pressure-saturated samples and vendor GRI porosity indicates that the proposed method should be applicable to the measurement of drill cuttings.

It is important to note that the pressure-saturated porosity in this study is slightly underestimated compared to the GRI results. There may be multiple causes for this. One could be the disparity in peak sizes between the slow and fast relaxing peaks. The slow, or bulk diesel, dominates the signal, and therefore, the fast relaxing peaks may be prone to inversion error due to the low signal to noise. This is more relevant in low porosity systems where the signal of the fast relaxing peaks. The slow, or bulk diesel, dominates the signal, and the disparity in peak sizes between the slow and fast relaxing results. There may be multiple causes for this. One could be that the GRI samples are cleaned using a Dean–Stark technique that may have introduced an additional pore space.

**CONCLUSIONS**

An improved method to measure the source rock petrophysical properties of bulk density, matrix density, and porosity from drill cuttings/crushed rocks was presented. The results showed that pressure saturation of the cuttings improved the measurement accuracy of samples when compared to saturation using sonication. The average error when compared to that of the industry-standard method showed an improvement of 68, 76, and 126% in the bulk density, matrix density, and porosity, respectively, in pressure-saturated samples over sonicated samples. This new methodology can be applied to drill cuttings to assist in the determination of sweet spots and to evaluate reservoir quality. It could also be used for real-time drilling decisions and modeling.

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