

# DIELECTRIC MEASUREMENTS ON ARTIFICIAL COMPACTED CLAY-QUARTZ MIXTURES

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*This paper was prepared for presentation at the International Symposium of the Society of Core Analysts held in Aberdeen, Scotland, UK, 27-30 August, 2012*

## ABSTRACT

Determination of the hydrocarbon content from resistivity measurements can become challenging if the formation water resistivity is unknown or if the water is fresh. Moreover, in fresh-water shaly sands the formation resistivity is significantly influenced by the clay content. An alternative method, based on the dielectric contrast between formation water and other rock constituents, is provided by dielectric measurements.

The multitude of factors influencing dielectric response includes porosity, lithology, texture, brine salinity, and clay type and content. Understanding of the physical mechanisms controlling dielectric dispersion in shaly sands requires laboratory studies in which key petrophysical parameters can be varied systematically from sample to sample. It can be difficult or impossible to obtain such a collection from oilfield reservoirs. The ability to prepare artificial compacted samples with prescribed properties allows systematic variation of the sample lithology, clay content, and brine salinity and also eliminates many of the uncertainties in the sample characterization associated with reservoir cores.

A new laboratory setup was constructed for the compaction and saturation of clay-quartz mixtures in a dielectric measurement cell that eliminates the need for sample transfer between the compaction and measurement cells. An extensive study was undertaken over a wide range of saturated artificial clay-quartz samples to investigate the dielectric properties of shaly sands. Dielectric spectroscopy experiments were performed on these samples in the range of 10 MHz to 1 GHz at various saturating brine salinities. This paper describes the experimental setup and protocols for compaction and saturation of clay-quartz mixtures and presents measurement results.

## INTRODUCTION

Approximately one-half of the world's oil is currently produced from shaly sand deposits.<sup>1</sup> One of the primary goals in evaluation of these reservoirs is to determine their hydrocarbon content. Determination of the hydrocarbon content from deep resistivity measurements can become challenging if the formation water resistivity is unknown or if the water is fresh. Moreover, in fresh-water shaly sands the formation resistivity is significantly influenced by the presence of clays.

These issues make it difficult to accurately estimate hydrocarbon content with a conventional resistivity approach. An alternative method, based on dielectric contrast between formation water and other rock constituents, is provided by dielectric measurements<sup>2</sup>. However, to use this method for the quantitative determination of water content, the relationship (the so-called mixing law) between the properties of the constituents and their mixture needs to be established. It has previously been demonstrated that dielectric dispersion in carbonates is strongly influenced by the rock texture.<sup>3</sup> In addition to texture, clays have a significant effect on dielectric properties of shaly sands that has to be elucidated. In the present paper we describe the experimental apparatus and procedures developed for the systematic study of the clay effects on artificial samples. A similar investigation of the NMR relaxation have previously been carried out on brine-saturated clays<sup>4</sup>. In the present study we utilize saturated and compacted clay/quartz mixtures that better represent lithology and, consequently, physical properties of shaly sand formations.

## **EXPERIMENTAL APPARATUS AND PROCEDURES**

### *Compaction Setup:*

Several challenges have to be addressed to prepare and conduct dielectric measurements on artificial compacted samples. A straightforward approach to prepare artificial samples is to first press the sample in a compaction cell and then conduct required measurements on the compacted sample. However, if dielectric measurements are required, the sample has to be transferred from a compaction cell to a dielectric measurement cell. It can be difficult or impossible to transfer samples with low clay content owing to potential disaggregation. Moreover, the dielectric measurements are significantly influenced by the gaps between the sample and the measurement cell electrodes.<sup>5</sup> The movement of the sample between the compaction and measurement cells increases a chance of introducing a gap and reducing the quality of the dielectric measurements.

To avoid these issues we opted to compact samples directly in the measurement cell, which eliminates both the sample transfer and potential gap problems. The experimental setup for the preparation of artificial compacted clay-quartz mixtures is shown in Figure 1. It consists of a compaction press, press control box, data acquisition computer, brine de-aerator, and pumps. In Figure 1 the compaction cell is connected to high-pressure tubing and installed in the press frame.

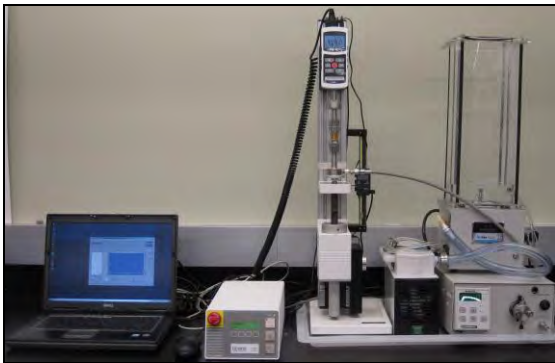
### *Sample Preparation:*

The samples analyzed in the present study were prepared by combining clay minerals and quartz. The clay minerals were obtained from the source clay repository at Purdue University. The clay repository allows researchers access to clay samples from large, reasonably homogenized rocks that are well characterized<sup>6</sup> and have less variability in their physical properties compared with natural deposits. We used the following source clays: kaolinite (KGa-1b), illite (IMt-2), and smectite (SWy-2).

The sample preparation procedure began with mixing dry clay and quartz powders in Retsch MM-400 mixer mills for 30 minutes to produce homogeneous samples. The powders were combined in various proportions to mimic lithological variability of natural shaly sand reservoirs. Next, the mixed powder was loaded into the compaction

cell and weighed. The compaction cell was installed into the compaction press and the dry sample was precompacted to 500 psi to limit mobility and prevent segregation of the clay and quartz particles during saturation.

After the precompaction phase, brine of the desired salinity was pumped into the sample at 300- to 400-psi pressure through the bottom piston. The excess brine was allowed to leave the sample through the top piston and was collected in a container. The brine pressure was maintained until at least 20 pore volumes of brine passed through the sample to achieve complete saturation. The final step included compaction at a higher pressure to further consolidate the sample. Then the compaction pressure was gradually released until it reached a low level of 50 psi. During this process the sample expanded and excess brine from the outlet in the top piston was taken back into the sample to fill the voids. Once the compaction pressure was reduced to 50 psi small volume of brine was pumped through the sample at low pressure to ensure complete saturation in the unstressed conditions. A typical artificial clay-quartz sample produced after the compaction and saturation cycle is shown in Figure 2.



**Figure 1.** Compaction setup with the measurement cell installed.



**Figure 2.** Artificial compacted clay-quartz sample removed from the compaction cell.

#### *Dielectric Measurements:*

The dielectric spectra were collected using a transmission line technique that allows accurate broadband measurements. The scattering parameters measured by the network analyzer were inverted to the complex dielectric constant at multiple frequencies based on a published algorithm.<sup>7</sup> The accuracy of the dielectric measurement was verified by conducting measurements on ethanol and comparing the results with data obtained at the National Physics Laboratory.<sup>8</sup> The results are shown in Figure 3 and the good correspondence between the NPL data and the present measurements is evident.

After the sample compaction cycle was completed, the measurement cell was removed from the press and connected to an Agilent E5071C network analyzer. The metal pistons utilized for sample compaction and saturation were replaced with Teflon washers that maintained sample in a fixed position within the airline and prevented fluids from escaping the sample. Any residual amount of brine remained on the outside of the sample was forced by washers to fill possible gaps between the sample and measurement cell electrodes. Small gaps filled with conductive brine produce minimum adverse effect on the dielectric measurements on saturated samples compared to air-filled gaps.

The dielectric spectra were obtained on the collection of clay-quartz mixtures with variable clay content and saturating brine salinities. For example, the dielectric and conductivity spectra obtained on compacted smectite-quartz mixtures with 10.8% and 21.2% smectite content are compared in Figure 4. Both samples were saturated with 10-ppk NaCl brine. The effect of increasing clay content is evident on both the dielectric and conductivity spectra.

*Porosity Measurements:*

After the dielectric measurements were completed, the sample size was determined from caliper measurements of the distance between the end of the cell and top of the Teflon washers. The length of the Teflon inserts was precisely known. Then Teflon contained was connected to the measurement cell and the sample sandwiched between the Teflon inserts was pushed out into a Teflon<sup>®</sup> container with a piston. This procedure minimized any potential alteration of the sample and fluid loss during its transition. The saturated sample weight was determined by weighting the Teflon container and from precise weights of the dry Teflon container and washers. Alternatively, the sample height can also be measured in the press itself during compaction. The difference between the dry and saturated sample weights provides the fluid weight that is converted into volume via density. The sample porosity is calculated as a ratio of the fluid volume to the sample bulk volume from caliper measurements.

The accuracy of the porosity determination was tested on solid rock samples. Five rock cores were cut into similar shape as the artificial compacted clay-quartz samples and standard gas porosity measurements were obtained. The gas porosity is determined from caliper measurements of the sample bulk volume and the solid grain volume measured in a pycnometer. The samples were subsequently weighed, saturated with brine and weighed again in a Teflon container used for pressed samples. Comparison of the standard gas porosity to values determined from the weights is given in Table 1.

**Table 1.** Comparison of the porosity values determined with gas pycnometer and by weighing dry and saturated cores.

Sample	Gas Porosity, %	Weight Porosity, %
Limestone 1	26.9	27.9
Sandstone 1	8.2	9.1
Sandstone 2	14.5	15.2
Dolomite 1	20.5	22.8
Dolomite 2	9.5	9.5

The porosity values for all but one sample agreed within 1%. The maximum difference between the gas and weight porosity values was 2.3% for “Dolomite 2.” The porosity based on weights was further benchmarked on a set of 11 samples from a shaly sand reservoir, and porosity values determined with gas pycnometer and weights agreed within 1.2% or better.

## CONCLUSIONS

In the present paper we have introduced an experimental apparatus for compaction and saturation of artificial clay-quartz samples. The experimental design was optimized to conduct accurate broadband dielectric measurements on the resulting compacted and saturated samples. Developed experimental protocols enable the preparation of artificial samples with controlled and repeatable properties. The dielectric measurement cell has been benchmarked on independently characterized dielectric standards and the high accuracy of the measurements confirmed. The developed protocols for determination of the compacted samples' porosity were benchmarked on two collections of solid rock samples with independently measured gas porosity.

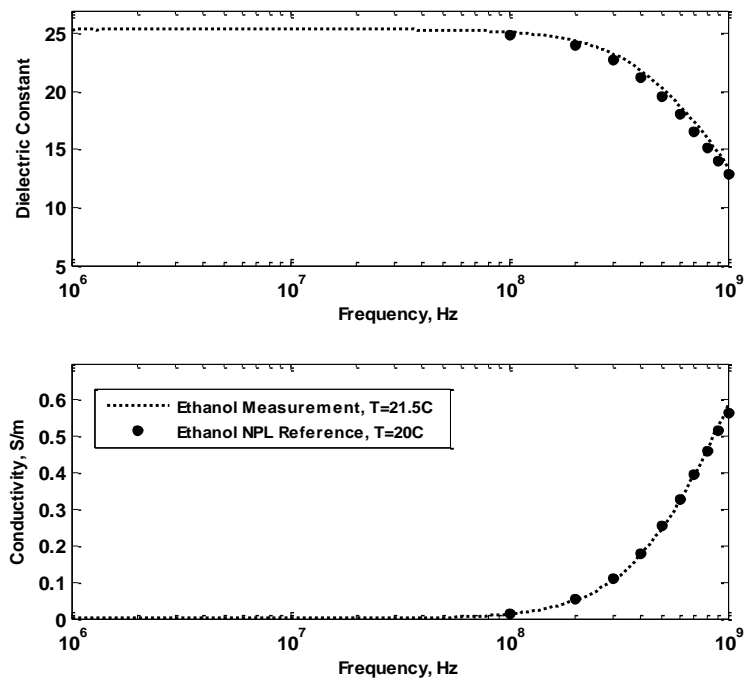
The developed experimental hardware and procedures allowed systematic variation of the sample lithology, clay content, and brine salinity that were used to obtain a measurement database and study dielectric properties of clay-containing rocks.

## ACKNOWLEDGMENTS

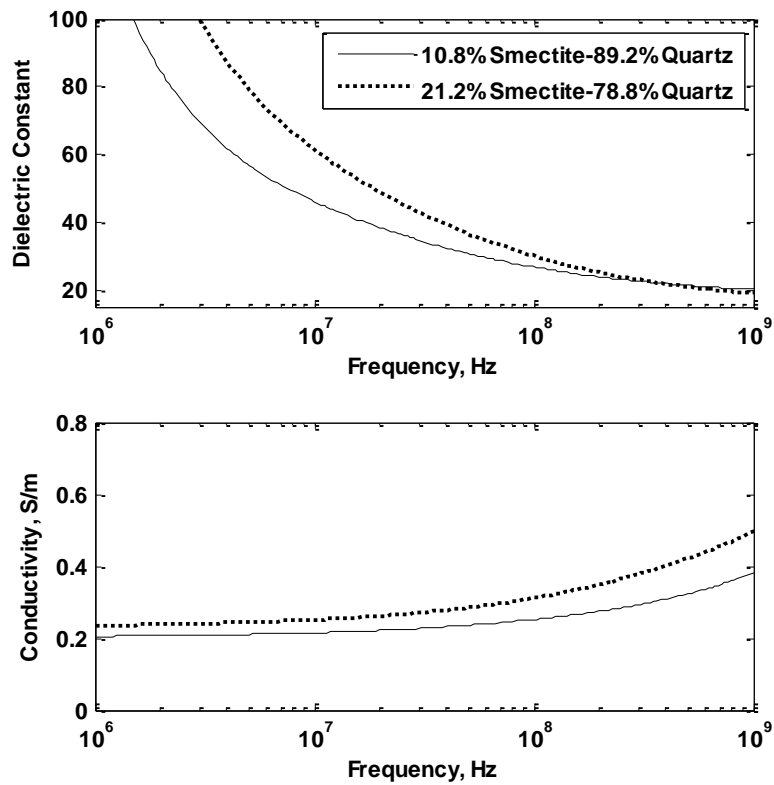
The authors thank Benjamin Costanza and Kamilla Fellah, who carried out a large part of experimental work after the initial setup and experimental procedures were developed. We are also grateful to Abigail Matteson, Lisa Meeks, and Tim Sime for their invaluable contribution in sample preparation and extensive characterization. We also thank Pabitra Sen and Michael Herron for stimulating discussions on the experimental procedures and clay minerals.

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**Figure 3.** The dielectric and conductivity dispersion curves measured with the dielectric measurement cell on ethanol



**Figure 4.** Dielectric spectra obtained on smectite-quartz mixtures with 10.8% and 21.2% smectite content.