ADVANCED CHARACTERIZATION OF SHALE GAS ROCKS USING DUAL RANGE FTIR AND DIELECTRIC DISPERSION

A. Marzoug[1], T. AlGhamdi[1], K. H. Sassi[2], and M. Badri[2]
[1] Saudi Aramco
[2] Sclumberger Dhahran Research Center

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ABSTRACT

Dielectric properties were measured on a suite of shale gas rock samples selected from several wells across the Silurian source rock formation. The dependence of minerals in the shale-gas rocks on the interpretation of the dielectric response was studied. The dual-range Fourier transform infrared (FTIR) technique was used to accurately quantify the mineralogical composition of the studied samples, including pyrite. Pyrite has a significant effect on the dielectric responses, and an accurate estimation of its volume is crucial for an enhanced interpretation of the dielectric response. A workflow was developed to accurately estimate the effective permittivity of the rock matrix to enhance the estimate of water volume from the dielectric response. The high-resolution retort was also used for the quantification of water content, and the results were compared to the water saturations from dielectric dispersion. NMR T_2 was also measured on the selected shale gas rock samples using very short echo spacing and showed that the most abundant pore-body size is in the range of 0.2 to 0.3 μm in diameter.

INTRODUCTION

Extensive shale gas reservoir characterization is essential for accurate estimates of the original gas-in-place (OGIP), the production rates, and the storage capacity of depleted reservoirs. Shale gas systems are relatively low porosity and ultra-low permeability, and comprise wide ranges of pore sizes. The latter is associated with the diversity of minerals that make up shale, such as clays, carbonates, and organic material (i.e., kerogen). The complexity in mineral content leads to fundamental questions, and often uncertainties, related to the calculation of the petrophysical properties: the total amounts and spatial distribution of original fluids in the reservoir, their thermodynamic states (i.e., adsorbed or free). The industry requires new techniques to be developed that would assist in accurate predictions of the rock and fluid properties, leading to much better description of the reservoir properties of the shale plays. The dielectric dispersion along with the dual-range Fourier transform infrared (FTIR) method, are the main focus in this study to

evaluate the organic-rich shale formations. The dependence of the dielectric dispersion to the mineralogical composition of the rock was studied in this work, in order to accurately estimate the effective permittivity of the rock matrix, hence, a better estimate of the water saturation from dielectric. The NMR measurements at very short echo spacing were also conducted to capture the micro- and nano- porosities of the rocks in order to accurately estimate the total porosity of the shale gas rocks. New workflows are developed and will be detailed in this paper.

DIELECTRIC LABORATORY MEASUREMENTS

Numerous dielectric forward models have been developed and used in our work to convert the dielectric measurements into water saturation, water salinity and rock matrix. The models are so-called bimodal, Stroud-Milton-De (SMD), shaly sand, and complex refractive index model (CRIM) (Seleznev et al., 2006). Each type of model has inherent strengths and weaknesses based on the assumptions intrinsic to the model. The CRIM model could be summarized by Eq. 1:

$$\sqrt{\epsilon^*} = (1-\emptyset)\sqrt{\epsilon_m} + \emptyset \mathrm{Sw}\sqrt{\epsilon_w} + \emptyset(1-\mathrm{Sw})\sqrt{\epsilon_h} \quad (1)$$

Where S_w is the water volume fraction, $\emptyset$ is the total porosity. ϵ^* is the measured complex permittivity. $\epsilon_m, \epsilon_w, \epsilon_h$ are, respectively, the permittivities of the matrix, water and hydrocarbon. As shown in Eq. 1, the matrix permittivity is a crucial parameter to estimate the water-saturation. The estimated water volume is strongly dependent on the permittivity of the rock matrix. A sensitivity analysis was performed and demonstrates that a one-unit error in matrix permittivity results in a 2-p.u. error in the water-filled porosity estimate, a huge error for the low porosity rocks of less than 10 p.u. total porosity. The results of the sensitivity analysis are shown in **Fig. 1**.

The laboratory dielectric measuring device used in this study comprises an Agilent network analyzer (NA), ENA series E50771C, controlled using a laptop. The network analyzer is calibrated using a set of short, open, and 50-ohm standards from Agilent. The coaxial probe operates in reflection mode; thus, reflection coefficient S11 (scattering parameter) is measured in sequence on the flat ends of the core plug. The measured complex reflection coefficients (S11) are recorded in the form of an amplitude (in decibels) and phase (in degrees) as a function of frequency which was varied from 10 MHz to 1 GHz, and inverted into conductivity and permittivity, dielectric characteristics of the core sample.

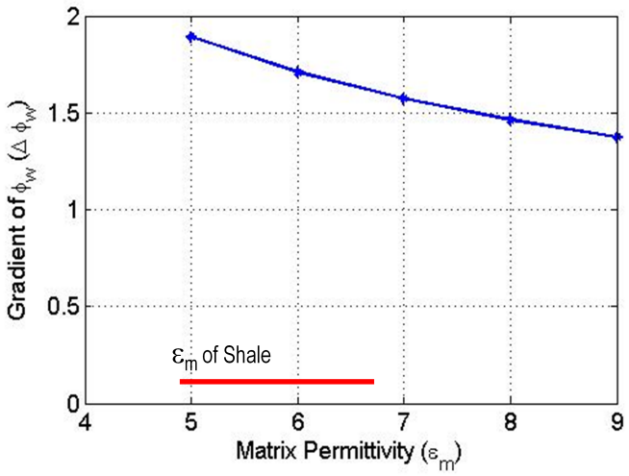

Fig. 1—Sensitivity analysis showing the error propagation of the matrix permittivity into water-filled porosity for a given rock property. The variation of the water filled porosity is plotted vs the matrix permittivity of the rock samples.

In this study, 10 shale gas core samples from the Saudi Arabia organic-rich shale formation were analyzed. The samples were selected from different wells and at different depths to cover the vertical and lateral variability of the Silurian source rock units. From each sample, a plug was drilled, approximately 1.5 in. in diameter. Small pieces were also cut from each sample. These small trim pieces were crushed sufficiently small to remove all the microfractures and coring-induced artifacts, yet large enough to be representative of the rock matrix. The crushed samples were used for the laboratory measurements of the total and effective porosity, LECO TOC, mineralogy analysis, and high-resolution retort for direct quantification of water content. More details of the petrophysical measurements of the crushed samples are given by Handwerger et al. (2011). All laboratory measurements were made on samples in their "as received" state of saturation, meaning that these may not be in their unaltered "native state".

The dielectric measurements were conducted on the selected samples and inverted to estimate the water-filled porosity. A default value of effective matrix permittivity was applied as the input parameter for the inversion. This study was conducted to intuitively validate the dependence of the water-filled porosity to the matrix permittivity. The estimated water saturations were compared to the saturation measurements from high-resolution retort using the crushed samples. **Fig. 2** shows an overestimation of the dielectric-derived water saturation compared to the retort saturations. This overestimation of water volume is mainly caused by the default choice of the matrix permittivity. Therefore, an accurate estimation of water content of the shale gas rocks requires a better estimation of the effective matrix permittivity. In the next section, a workflow will be discussed to enhance the estimation of the rock matrix permittivity and demonstrate its dependence to the mineralogical composition.

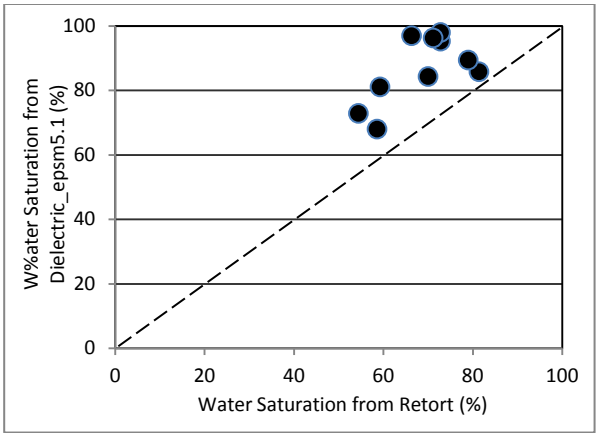

Fig. 2—Comparison between the saturations measured from retort and those estimated from dielectric measurements using the default matrix permittivity value of 5.1.

EFFECTIVE MATRIX PERMITTIVITY OF SHALE GAS ROCKS

The effective matrix permittivity of each rock sample was estimated by inverting the measured permittivity and conductivity dispersions. The dielectric physics model used for the inversion is the so-called bimodal model which attributes the dielectric dispersion to rock grains with plate-like shapes. These grain shapes properly describe the dielectric behavior of most rocks, but are even more appropriate for the clay particles, which are plate like and are abundant in the shale samples. The water saturations measured from the high-resolution retort technique was used as input property in this inversion process. The workflow applied to estimate the effective matrix permittivity is described in **Fig. 3.**

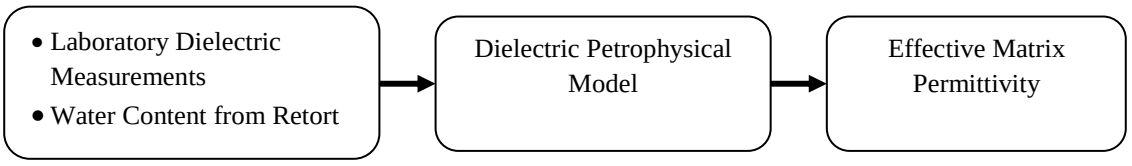

Fig. 3—Workflow applied to estimate the effective matrix permittivity.

MINERALOGY COMPOSITION

Mineralogy is a fundamental part of formation description. In this study, a quantitative mineral analysis of the selected samples is based upon the Fourier transform infrared (FTIR) spectroscopy measurements. More details on the FT-IR technique could be found in Herron et al. 1997. During the course of the FTIR analysis, few characteristics have been noted. Illite is the most commonly observed clay mineral in the samples we studied. It represents almost half the weight percent of the captured minerals. The muscovite concentrations average 20 weight percent. This is a high number. The experiment shows that the FTIR procedure is able to quantitatively distinguish illite from muscovite, even

when present in the same sample. Kaolinite is also present within the studied samples with an average concentration of 7 weight percent. The pyrite is in the range from 0 to 6.9 wt%.

DEPENDENCE OF MATRIX PERMITTIVITY ON MINERALOGY

The complex refractive index (CRI) dielectric mixing law is the forward modeling technique that will be used to investigate the dependence of the effective matrix permittivity $\in_{meff}$ on the mineralogy composition of the rock. The CRI law could be expressed as

$$\sqrt{\in_{meff}} = \sum_{i=1}^{n} V_i \sqrt{\in_i} \,. \tag{2}$$

V_i represents the volume fraction of rock minerals, $\in_i$ is the dry matrix permittivity of each mineral, and *n* is the number of minerals measured by the FTIR system.

A deterministic misfit data approach was developed in this study to investigate the effect of mineralogy composition of the rock on its effective matrix permittivity. This study aims at computing the dry permittivity of each mineral of the selected samples measured by FTIR system. The workflow is shown in Fig. 4.

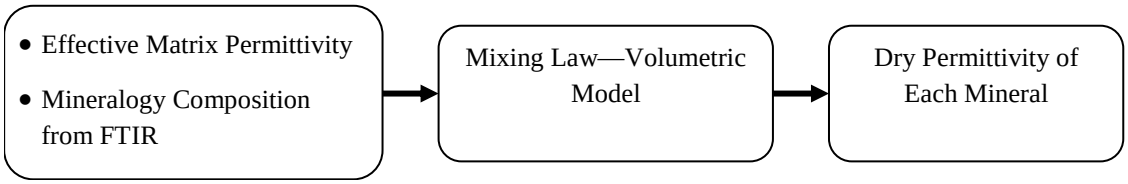

Fig. 4—Workflow applied to estimate the dry permittivity of minerals.

The dry permittivity values determined from the workflow in Fig. 4 are summarized in Table 1.

PERMITTIVITY VALUES OF KEY MINERALS IN THE STUDIED SAMPLES

Mineral	Permittivity value
Quartz	4.65
Illite	5.8
Kaolinite	5.08
Chlorite	5.02
Muscovite	5.67
Pyrite	37.43

Pyrite is a relatively heavy mineral in shale gas rocks. It influences the formation evaluation when it is present at large quantities (Anderson et al., 2006; Clennell et al., 2010). Pyrite has a significant effect on the dielectric responses, enhancing conductivity and permittivity very significantly. Therefore, an accurate estimation of dry permittivity

of pyrite is crucial to account for it in the interpretation of dielectric responses. The analyzed samples contain a broad range of pyrite. The estimation of the dry permittivity of pyrite along with the key shale minerals through the workflow developed above represents a major element in this paper.
The dry permittivity estimated above was used to reconstruct the effective matrix permittivity of the shale gas rocks. These are used to redetermine the water saturation from the permittivity and conductivity dispersions.

CONCLUSIONS

The dependence of correct identification of minerals in shale gas rocks on the interpretation of the dielectric response was studied in this paper. The dual-range FTIR technique was used to accurately quantify the mineralogical composition of the studied samples, including the pyrite. The pyrite has an obvious effect on the dielectric responses, and an accurate estimation of its volume is crucial for an enhanced interpretation of the dielectric response. The dependence of the effective matrix permittivity to the shale gas minerals was well investigated in this work. A workflow was developed to accurately estimate the effective permittivity of the rock matrix to enhance the estimate of water volume from the dielectric response. NMR T_2 was also measured on the selected shale gas samples using very short echo spacing to capture all the inorganic and organic porosity at the nanometer scale. An accurate estimation of total porosity from NMR and total water content from dielectric response enhance the estimation of the gas in place of the shale gas rocks.

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