# SHALE CHARACTERIZATION USING COMPLEX PERMITTIVITY MEASUREMENTS

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## Abstract
Geomaterials exhibit a wide range of electrical properties depending on their structure, mineralogical composition, porosity, water content, ionic concentration, and anisotropy. In this study, low and high-frequency complex permittivity measurements are used to analyze shale cores and to extract fundamental engineering and physical properties.

## Complex permittivity
The real permittivity is related to the density of dipole moments aligned in the direction of the applied electric field. Therefore, the real permittivity is sensitive to the presence of water (a dipolar molecule) and the existence of different polarization mechanisms (processes that induce dipole moments inside the material). On the other hand, losses due to polarization and ion movement govern the measured imaginary permittivity.

## Experimental work
Preserved shale samples are prepared parallel and normal to the bedding plane for complex permittivity measurements. Tested samples include preserved shales (Queenston, Mancos, and Pierre) and a pure clay (kaolinite). A two-electrode sample holder (10 Hz – 1 MHz) and a surface coaxial termination probe (0.02 GHz – 1.30 GHz) are used in this study. An impedance analyzer and a network analyzer measure the complex impedance and the complex reflection coefficient of tested cores, respectively.

## Results and analyses
*Moisture content.* The real permittivity of a solid-water system at high frequencies is related to the volumetric fraction of the water phase. Different theories have been proposed to determine the relationship between the real permittivity at a specific frequency and the volumetric moisture content (van Beek 1967; DeLoor 1983). Generally, the relationship is non-linear; however, a linear approximation between high-frequency real permittivity of shales ($f$ 1.30 GHz) and the volumetric moisture content $d_w$ can be written as (Fam and Dusseault 1998-b):

$$d_w \approx 0.017 \cdot \kappa' - 0.019 \tag{1}$$

Equation 1 is applicable for mixtures with $d_w$ less than 0.5.

*Free water content.* Free water shows a relaxation phenomenon at high frequencies ($f = 17$ GHz; Pottel 1973). However, the presence of bound water in clays and shales may lead to

a reduction in the relaxation polarization of water dipoles and a lower complex permittivity (De Loor 1983; Adamson 1990). This concept can be used to extract the free water content of clay-fluid mixtures and to assess the reactivity of the system; high percentages of bound water indicate reactive systems with high specific surfaces. Figure 1 shows an example of an imaginary permittivity spectrum of a kaolinite specimen with volumetric water content $d_w = 0.60$. Knowing that the imaginary permittivity of free water at 1.30 GHz is $\kappa'' \approx 5.3$ and assuming a linear system, the volumetric free moisture content $d_{fw}$ can be approximated by:

$$d_{fw} \approx 0.2 \Delta \kappa'' \qquad (2)$$

where $\Delta\kappa''$ is the deviation in the imaginary permittivity trend at 1.30 GHz because of free water polarization. The procedure to extract the free water content is sketched in Figure 1, showing that more than 66% of the pore water in the kaolinite specimen can be considered as free water. Shale cores showed similar trends and the estimated free water contents are summarized in Table 1 (Fam and Dusseault 1998-b).

Table 1. Estimated free water contents of clays and shales

| Specimen | Kaolinite clay | Queenston shale | Mancos shale | Pierre shale |
|---|---|---|---|---|
| Volumetric water content, $d_w$ | 0.60 | 0.086 | 0.062 | 0.143 |
| Estimated free water content, $d_{fw}$ | 0.40 | 0.04 | 0.02 | 0.004 |
| Percentage of free water (%) | 66.7 | 46.5 | 32.2 | 2.8 |

As shown in Table 1, Pierre shale shows the minimum free water content, which agrees with its high specific surface and high smectite content (Fam and Dusseault 1998-a).

*Surface conductivity.* Relaxation phenomena have been observed for preserved Queenston and Mancos shales at frequencies near 1 MHz. These polarization mechanisms are attributed to Maxwell-Wagner effects and the enhanced surface conductivity of clay particles. Grosse's model can be used to determine the surface conductivity of such systems using the measured relaxation frequencies (Grosse 1988).

$$\tau_{HF} = \frac{\varepsilon_o \kappa'_f (\kappa_r + 2)}{\sigma_f (\lambda_r + 2)} \qquad (3)$$

where $\kappa'_f$ and $\sigma_f$ are the permittivity and conductivity of the pore fluid, $\lambda_r = 2\lambda/R\sigma_f$, and $\lambda$ is the surface conductivity, and $\varepsilon_0$ is the permittivity of vacuum. Back-calculated surface conductivity values for Queenston and Mancos shales range between $1.1 \cdot 10^{-9}$ and $2.7 \cdot 10^{-9}$ Sm/m (Fam and Dusseault 1998-c).

*Characteristic length.* Air-dry shales may show double layer polarization because of the displacement of the cationic cloud relative to the negatively-charged clay particles. The relaxation time of this polarization mechanism depends on the characteristic length and the diffusion coefficient (Dukhin and Shilov 1975; Garrouch and Sharma 1994). Air-dry

Queenston shale showed double layer polarization at 8 kHz. Back-calculated characteristic length was 0.5 µm. This is a typical value for clay mineral particles.

*Anisotropy.* Low-frequency permittivity data acquired normal and parallel to the bedding plane can be used to study permittivity anisotropy of shales. Figure 2 shows complex permittivity data of Pierre shale parallel and normal to the bedding plane at low frequencies. As shown, both real and imaginary permittivity data normal to the bedding plane are higher than those measured parallel to the bedding plane. This behavior is opposite to the expected trend in sedimentary rocks and shales with low clay contents (e.g., Mancos shale) where tortuosity effects reduce ion mobility normal to the bedding plane. Accumulation of charges on clay interfaces normal to bedding (Maxwell-Wagner polarization) is responsible for this behavior. The high specific surface of Pierre shale ($S_a$ = 220 $m^2$/g) acts as a charge barrier, enhancing polarizability normal to the bedding plane.

## Conclusions

It is shown that permittivity spectra in the low-frequency range can provide information related to characteristic length, surface conductivity, and shale reactivity through previously-developed models. Also, high-frequency measurements may be used to extract volumetric water content. Using a simple approach based on the low polarizability of bound water, the imaginary permittivity at high frequencies is used to estimate the free water content of tested cores. Cores with high-specific surfaces show the lowest free water content. Finally, permittivity measurements are proposed as simple techniques to provide micro-level information related to tested cores.

## Acknowledgments

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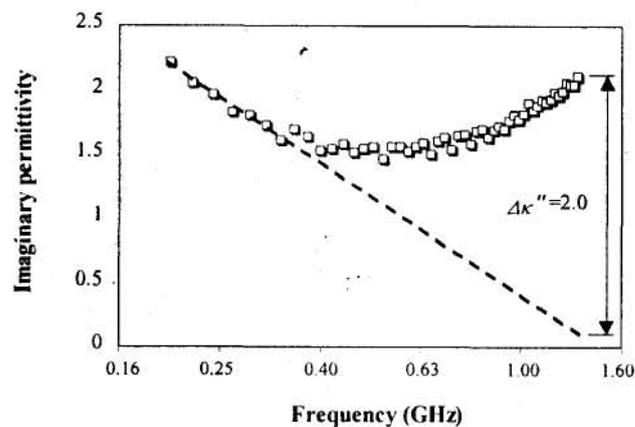
Figure 1. High-frequency imaginary permittivity spectrum of a kaolinite clay with volumetric water content = 0.6 (the procedure to extract free water content is sketched).
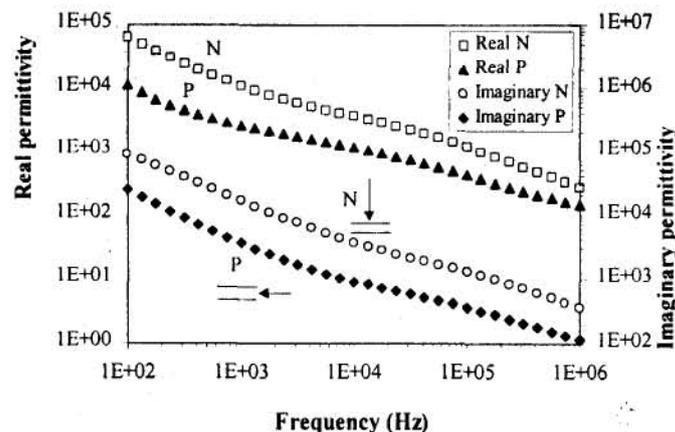


Figure 2. Low-frequency complex permittivity spectra of Pierre shale parallel (P) and normal (N) to the bedding plane.