Fluids, Soils and Rocks: Electromagnetic and NMR Signatures

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Summary

Electromagnetic phenomena support the development of exceptional methods for non-destructive, non-contact geomaterial characterization and subsurface process monitoring. Such phenomena include Ohmic conduction, electromagnetic wave propagation and nuclear magnetic resonance. The physical parameters involved include DC conductivity σ, complex permittivity $\varepsilon^*(\omega)$ and complex permeability $\mu^*(\omega)$ spectra, and nuclear magnetic resonance relaxation. These parameters reflect mineralogy, particle and pore size distributions, pore network topology and anisotropy, fluid composition, state and fluid-mineral interaction. Proper data interpretation requires careful understanding of underlying physical concepts and measurement methods.

Ohmic DC Conduction in Geomaterials

In general, dry minerals, dry salt and deionized water are non-conductive, i.e., dielectrics. Electrical conductivity emerges when dipolar water molecules hydrate salts and counter-ions resting on dry mineral surfaces. Then, the electrical conductivity of wet soils and rocks reflects the availability and mobility of hydrated anions and cations in the pore water (ionic concentration) and in double layers (surface conduction – Pfannkuch 1972, Klein and Santamarina, 2003).

The electrical conductivity of low specific surface porous media saturated with high conductivity fluids is determined by porosity φ, degree of saturation S and pore fluid conductivity σ_{fl}; a first order approximation predicts $\sigma=\varphi \cdot S \cdot \sigma_{fl}$. The expression is generalized as $\sigma=a(\varphi S)^b \sigma_{fl}^c$, where fitting parameters a, b and c account for other internal effects such as tortuosity and pore topology (often known as Archie's equation).

Surface conduction gains relevance in low-porosity geomaterials made of high specific surface clay minerals, when the pore fluid has low ionic concentration (Santamarina et al. 2019). Under these conditions, electrical conductivity becomes inversely proportional to porosity (Note: this trend is opposite to Archie's equation).

Permittivity Spectrum

The "hindered displacement" of negative and positive charges in a geomaterial subjected to an electric field causes internal polarization (Santamarina et al., 2001 & 2020) Polarization mechanisms and the extent of polarization depend on the electric field frequency and reflect temporal and spatial scales within the material.

Molecules can experience multiple vibration and bending modes (see the case of water in Popov et al, 2016). Dry minerals experience electronic (UV-frequencies – 1 to 100 PHz) and ionic polarizations (IR-frequencies - 10 THz to 1 PHz). The combined effects of electronic and ionic polarization in dry geomaterials results in low permittivity values, typically $\kappa' < 10$.

In addition to electronic and ionic polarization, fluid molecules (e.g., water) or molecular segments (e.g., some organic fluids) can rotate and follow the external electric field. Orientational polarization occurs at microwave frequencies (20 MHz–20 GHz).

Spatial Maxwell-Wagner polarization takes place in wet geomaterials when hydrated ions (in pore fluids or in double layers) move in response to an external electric field but reach transverse pore walls. Spatial polarization typically manifests at radio-frequencies (10 kHz - 20 MHz).

The various polarizations accumulate with decreasing frequency. Polarizations have a phase shift with respect to the applied field. Then, the relationship between polarization and applied field can be captured through a complex permittivity to reflect the response amplitude and phase shift: $\kappa^*=\varepsilon^*/\varepsilon_0 = \kappa' + j\kappa''$ where $j^2=-1$. The out-of-phase "imaginary" component of the permittivity κ'' captures polarization losses and is in phase with Ohmic conduction losses. Consequently, the measured "effective imaginary permittivity" adds polarization and conduction losses, $\kappa''_{eff}=\kappa''_{pol}+\sigma_{DC}/(\omega\varepsilon_o)$.

It follows from the previous analysis that the complex permittivity at microwave frequencies (~0.1 to 1 GHz) is dominated by the pore fluid polarizability and volume fraction; then, a first order approximation to the permittivity of geomaterials at microwave frequencies is $\kappa' \approx \varphi \cdot S \cdot \kappa'_{fl}$. (Note: the Complex Refractive Index Model is a better predictor - Santamarina et al. 2019).

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On the other hand, the relaxation of Maxwell-Wagner spatial polarization is based on ionic diffusion. Therefore, the relaxation time depend on pore size r and the ionic diffusion coefficient D, and scales as $\tau \propto r^2/D$. Various authors have used these concepts to infer pore size distribution from permittivity spectra (Kruschwitz et al, 2009; Revil et al, 2014).

Experimental methods vary with frequency f and wavelength $\lambda=V/f$, where V is the propagation velocity. At high frequencies (say, f >200 MHz), measurement methods are based on wave propagation and the wavelength is similar or shorter than the specimen size λ<d (resonance techniques apply when $\lambda\approx$d). Measurements at low frequencies $\lambda>>$d use capacitor-type configurations and adopt a lump parameter model analysis in terms of resistance and capacitance.

Unfortunately, polarization measurements at radio frequencies are dominated by the interaction between hydrated ions and the specimen boundaries. In particular, the mismatch between electronic conduction in peripheral electronics and ionic conduction in geomaterials results in charge accumulation at the specimen-electrode interfaces creating a pronounced polarization bias known as electrode polarization; we highlight that this polarization is not a material property but an inherent measurement bias. The threshold frequency when electrode polarization obscures permittivity measurements increases with the material conductivity. In water saturated geomaterials the threshold frequency can exceed 100 kHz (Klein and Santamarina, 1997; see Hakiki, 2020 for a detailed analysis and comprehensive data). Contrary to frequent claims, 4-terminal measurement methods do not cancel electrode polarization; furthermore, they aggravate the effects of inductive coupling in the upper radiofrequency range.

Complex Magnetic Permeability

The third electromagnetic parameter is the magnetic permeability μ. Analogous to polarization, the material magnetization (1) is proportional to the imposed magnetic field, (2) there may be a phase shift between the applied field and the material response, and (3) various spatial and temporal scales can be involved. Consequently the magnetic permeability of a material is a complex quantity $\mu^*=\kappa'+j\,\kappa''$ with a characteristic spectral response.

High frequency measurements are based on wave propagation analysis and require reflection and transmission data to deconvolve the effects of complex permittivity and complex permeability. At low frequencies, measurements are based on imposed magnetic fields, typically within a solenoid, and data reduction is based on lump parameters (Klein and Santamarina, 2000).

Most geomaterials are non-ferromagnetic but either para-, or diamagnetic; therefore, $\mu^*=1+j0$. However, ferromagnetic components can be found in mine tailings, fly ash and hydrothermal sediments (Santamarina et al. 2019). Furthermore, ferromagnetic impurities can be purposely added for engineered applications that require monitoring (see magnetic drilling muds in Hakiki 2020). In such cases, the wave propagation constant must involve both, the complex permittivity κ^* and permeability μ^*.

Nuclear Magnetic Resonance

Nuclear magnetic resonance NMR targets the intrinsic magnetic spin of atomic nuclei with an odd number of protons or neutrons, such as hydrogen ^{1}H. Nuclear spins precess in the presence of a magnetic field B_{oz} with Larmor frequency $\omega_L=\gamma B_{oz}/2\pi$ where the gyromagnetic ratio is $\gamma=2.675\times10^8$ Hz/Tesla for hydrogen ^{1}H (Levitt, 2001). Most NMR systems that target hydrogen ^{1}H impose an external magnetic field B_{oz}= 0.05-to-23 Tesla and the Larmor frequency falls in the radio frequency RF range (2 MHz to 1000 MHz).

Then, a basic measurement consists of three steps: (1) impose a static external magnetic field B_{oz}, (2) apply a transverse magnetic pulse B_x, and (3) monitor the recovery of the longitudinal z-magnetization ($T1$ relaxation time) and the decay of the transverse x-magnetization ($T2$ relaxation time).

The relaxation of bulk fluids following the transverse pulse B_x results from thermal random vibration and renders long relaxation times (e.g., $T2$ =2.8s for bulk water). However, the interaction between water molecules and mineral pore walls expedite relaxation when minerals contain paramagnetic ions such as Fe^3 and Mn^2. Random walk brings molecules close to pore walls, hence, the relaxation time is a function of pore size and diffusivity.

The brief introduction above allows us to anticipate valuable applications of NMR for the characterization of geomaterials and process monitoring, including: pore fluid fingerprinting (Hakiki 2020); water mass, state and spatial distribution (magnetic gradient - Zhao and Santamarina 2022); and pore size distribution (from the decomposition of the time response in terms of a spectrum of relaxation times - Brownstein and Tarr, 1969; Ridwan et al, 2020).

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