Complex Permittivity of Common Minerals and One Soil at Low Water Contents

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Abstract—The complex permittivity of quartz, feldspars, calcite, and non-crystalline gypsum at 4%–7% volumetric water content shows low conductivity and low-frequency dispersion. At similar water contents, montmorillonite, gypsum crystallites, and a desert soil all show unusually strong and broad low-frequency dispersion, and strong attenuation rates above 100 MHz. The desert soil contained 80% quartz, 10% feldspars, and 10% gypsum, with the gypsum appearing as crystallites and crustations on the quartz particles. Despite insignificant salt or clay mineral content, the dispersion and attenuation rates of the soil exceed those of its constituents and are similar to that of montmorillonite, with a rate exceeding 100 dB m⁻¹ by 1 GHz. We attribute this to a Maxwell-Wagner type of relaxation, likely caused by polarization resulting from surface charge and from the charge separation caused by dielectric and conductivity contrasts between the gypsum and quartz. The conductivity contrasts were likely enhanced by ions dissolved from the gypsum and feldspars into free water within, and water adsorbed on the quartz surfaces.

Keywords—ground-penetrating radar; complex permittivity; attenuation rate; dispersion

I. INTRODUCTION

Desert soils in the United States, and in the evaporite basins of the Middle East, allow insignificant penetration of ground-penetrating radar (GPR) signals, which suggests one-way attenuation rates exceeding several tens of dB m⁻¹. These soils commonly contain calcite, gypsum, quartz, and feldspars. In addition, the soils are usually suspected of having a high salt, or magnetic and clay mineral content, to make them exceptionally conductive and therefore, lossy. We have found some desert soils within 1 m of the surface to have volumetric water contents of about 4%–7%, sufficiently high enough to suspect that conductivity, σ , is the cause. However, conductivity mapping during the 1950s [1], based on ground wave attenuation near 1 MHz, estimated that effective σ values in the U.S. Southwest were 0.015–0.008 S m⁻¹, which would allow a few meters of penetration at 100 MHz. Consequently,

relaxation processes must often be present and are the subject of this paper.

We discuss the frequency (f)-dependent complex permittivities, $\epsilon^*(f)$, of a desert soil and of common soil minerals at low water contents. The soil exhibited a very high attenuation rate yet had insignificant salt content, no magnetic or clay minerals, and an insufficient conductivity to explain its high attenuation rate. We wanted to find if dielectric relaxation was strong, the likely relaxation process and where relaxation was centered in frequency. Our objective was to compare $\epsilon^*(f)$ of this soil with that of its constituent minerals (and with two common clay minerals) at similarly low water contents and range of grain size, and over a bandwidth that would cover the common GPR center frequency bandwidth of 100–1,000 MHz. We used time domain reflectometry (TDR) to measure ϵ^* , modeled the results to interpret the relaxations, and used scanning electron micrographs to determine grain structure.

II. METHODS

We measured $\epsilon^*(f)$ from 1.6 MHz–10 GHz. We express $\epsilon^*(f)$ as

$$\epsilon^*(f) = \epsilon'(f) - j \epsilon''(f), \quad (1)$$

where ϵ' is the real part, ϵ'' is the imaginary part, and $j = \sqrt{-1}$. The quantity ϵ'' includes the term $\sigma/\omega\epsilon_0$, where σ is measured in Siemens per meter (S m⁻¹), $\omega = 2\pi f$, and $\epsilon_0 = 8.854 \times 10^{-12}$ F m⁻¹. As frequency decreases, the σ term dominates ϵ'' , should ϵ'' exhibit a linear frequency dependence on a log-log scale below 100 MHz. In this case, an effective σ is then computed from the asymptotic low frequency value of ϵ' , as we do here. The complex refractive index $n^* = \sqrt{\epsilon^*}$. The attenuation rate, β , is derived from the imaginary part of n^* (n'') such that

$$\beta \text{ (dB m}^{-1}\text{)} = 8.686 \omega n''/c, \quad (2)$$

where $c = 3 \times 10^8$ m s⁻¹.

TDR records the complete reflection sequence of a step pulse incident upon and transmitted through a sample within a coaxial waveguide in which the center conductor, known as the pin, is of finite length, d (see Appendix and [2; 3]). The end of the pin makes the waveguide effectively open-ended because reflections originate from the front sample face and from the

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end of the pin, which is well-covered by the sample. The highest reliable frequency is determined by an effective calibrated d , and by ϵ , both of which must keep the phase propagation quantity, $\omega d \sqrt{\epsilon}/c < \pi/2$. Our effective d was close to $d = 2$ mm, and our highest reliable frequency was about 6 GHz due to noise. Calibrations with ethanol showed errors less than 8% in ϵ up to 5 GHz [3]. We recorded reflections for 4 ns, digitized the waveforms at several sampling intervals, and averaged all results.

Table 1 lists relevant parameters for our soil and for sample minerals common in soils. The non-clay mineral samples can be classified as a medium size silt because all had grain sizes less than 53 microns (μ) with a small percentage $< 2 \mu$. The grain sizes of Na-kaolinite (sodium saturated with sodium-exchangeable ion) and Ca-montmorillonite (calcium-saturated montmorillonite) were likely $< 10 \mu$. Median grain sizes of the quartz and crystalline gypsum were 8μ . Our anorthoclase and oligoclase feldspars are both plagioclases that contain varying amounts of sodium and calcium. Gypsum (calcium dihydrate) and calcium are dielectrically anisotropic; the gypsum more so. We determined the σ values from the asymptotic values of ϵ .

We used two different gypsum samples. The first was non-crystallite gypsum that we ground, and the second was crystallite grains supplied by Sigma Scientific. Particle analysis supplied by an independent laboratory indicated a possible maximum surface area of $5 \text{ m}^2 \text{ gr}^{-1}$. This suggests, at the volumetric water content (v) of 0.075 and mineral content of 0.67, that at least 90% of water was in a free state. The ϵ^* results for the two gypsum samples are significantly different. Our target volumetric water content was 5%, but the final values were within 2% (Table 1). The water was doubly distilled, with $\sigma < 0.001 \text{ S m}^{-1}$.

Our soil sample was from an unspecified location in Iraq. X-ray diffraction analysis found, by weight: gypsum (10%), quartz (80%), unspecified plagioclase feldspars (10%), and insignificant salt or salt elements. Scanning electron microscope (SEM) micrographs (Fig. 1) and electron dispersion analysis (EDA) showed significant sulfur, and calcium within one crystalline crust and within several crystallites on the quartz grains surfaces. We therefore assumed these elements are the gypsum constituents. At the low pH of

TABLE I. PROPERTIES OF THE SAMPLES.

Sample	Vol. Wat. %	Por.	$\sim \epsilon'$	$\sigma (\text{S m}^{-1})$
Gypsum crystallites	7.5	0.33	11.2, 12.0, 5.4 [4]	0.025
Gypsum ground	6.7	0.31	11.2, 12.0, 5.4 [4]	0.0024
Calcite	5.0	0.30	8.5, 7.6 [4]	0.0029
Quartz	5.6	0.41	4.5 [4]	0.0026
Anorthoclase	4.7	0.33	7.1 [5]	0.0048
Oligoclase	3.7	0.36	6.1 [5]	0.0018
Na-Kaolinite	4.3	0.40	5.1 [6]	0.0025
Ca-Mont. ³	4.4	0.45	5.5 [6]	Not asymptotic
Iraq soil	7.3	0.37	—	0.0450

the distilled water, the gypsum and quartz surfaces are negatively charged so that they should not be intrinsically strongly attracted.

III. RESULTS

We present our results according to the range of ϵ . The lower values occurred for calcite, the feldspars, quartz, and the ground gypsum, and the higher for the clay minerals, the complex soil, and the crystalline gypsum. The first group displayed consistent ϵ and β behavior, while the second group displayed inconsistent and higher values of ϵ and β .

We show the frequency dependence of ϵ in Figs. 2 and 3. Fig. 2 shows ϵ for five of the minerals. All five have reasonably constant ϵ from 100–2,000 MHz. The variations are caused by the differences in water content, porosity, pure mineral values of ϵ (Table 1), and likely texture. Consistent with the ϵ' solid bulk values [4–6] in Table 1, calcite and ground gypsum showed the highest ϵ' for all frequencies. Above about 6 GHz, system resonances appear. Below 100 MHz, all values rise with decreasing frequency. This behavior has been attributed to electrode polarization [7], which would

Figure 1. SEM micrographs of Iraqi soil grains which show crystalline gypsum encrusted on quartz (left) and adhering to quartz in small crystallites. The crystalline crust at left is the white part of the grains. EDA showed sulfur and calcium in section B (crust) but not A.

Figure 2. The real part for several minerals vs. log of frequency between 1 MHz and 10 GHz. Percentages refer to the volumetric water contents.

mean either a response to charge on the coaxial casings because of impeded flow into the sample, or polarization where pore spaces are blocked by a metallic particle, which we preclude. However, experiments with changing voltage at lower frequencies [8], consistent behavior of clay minerals from 1 kHz to > 1 GHz [2; 3; 9], and analysis of low-frequency dispersion in solids [10] argue against this. In our frequency range, the dispersion is likely related to strong contrasts in σ between different materials, and which polarized charge along grain boundaries and formed large dipole moments [11], both within individual grains and across grain boundaries. This process characterizes Maxwell-Wagner (MW) type relaxations. As frequency decreases, the induced moments more easily stay in phase with the applied field. For our more dielectric grains (e.g., quartz, feldspars), the σ contrast likely formed between them and the water layer films that contained ions in solution [10; 11]. This effect mildly began just below 1 GHz for the higher-solubility calcite and appears to have approached a low-frequency peak for the feldspars and quartz.

Fig. 3 shows ϵ' for the clay minerals, gypsum crystallites, and soil. In contrast with Fig. 2, as frequency decreases, strong dispersion begins at about 600 MHz or higher for the soil, gypsum, and montmorillonite. The gypsum curve approaches a peak near 1.5 MHz, whereas the others show little decrease in their rate of increase. The soil dispersion and high loss (shown later) is surprising, in view of the low amount of gypsum and strong presence of quartz. In fact, its high effective $\sigma = 0.045$ S

Figure 3. The real part for three other minerals and the soil. Percentages refer to the volumetric water contents.

Figure 4. The imaginary part for five minerals, and model behavior for only a conductive contribution (bold line); i.e., no water relaxation. Values in parentheses are the σ values we computed from the low frequency asymptotes. We assumed $\epsilon' = 5$ for the simple σ model.

m^{-1} (Fig. 5) is also unexpected, as is that of the gypsum crystallites. Consequently, the association of the two minerals, with possibly distinctly different values of ϵ' and likely different values of σ enhanced by the presence of water, makes this soil a candidate for any generic MW dispersion. The soil behavior is most similar to that of the montmorillonite, even at the highest frequencies where ϵ' drops to near 4, but montmorillonite was not present.

We show the frequency dependence of ϵ'' in Figs. 4 and 5. The same five minerals of Fig. 2 show asymptotic linear behavior at low frequency in the log-log formats (Fig. 4), from which we computed their labeled effective values of σ . The bold line shows ϵ'' for only $\sigma = 0.0025$ S m^{-1} . The deviation of the minerals' plots from this bold line shows that, by as low as 2 MHz, the relaxation of free water (centered at 20 GHz) strongly contributed to loss for all minerals. However, as we will see from modeling of the other minerals, relaxation centered well below 1 GHz [2; 3; 9] was also present. Although the effective σ is low, neither the minerals nor the added water had values as high. Consequently, conduction ions were likely added by dissolution.

Figure 5. The imaginary part for three minerals and the soil. Two of the minerals and the soil are labeled for the σ computed from the low frequency asymptotes. The montmorillonite appears to approach asymptotic behavior at a very low value of σ . The solid line plots the value for a model in which only $\sigma = 0.045$ determined ϵ'' , and $\epsilon' = 5$.

Figure 6. The one-way attenuation rate for the first five minerals. The solid lines plot attenuation rates for a model in which only σ determined ϵ'' , and $\epsilon' = 5$.

Fig. 5 shows ϵ'' for the two clay minerals, the gypsum crystallites, and the soil, along with theoretical behavior based on only the value of σ for the soil. The montmorillonite failed to reach asymptotic behavior at the low frequency end, but it appears that it would have done so at a very low effective σ . In contrast, at the high frequency end, the montmorillonite reached nearly the same value as the soil. The kaolinite behaved similarly to the previous minerals at low frequency. Only the gypsum crystallites paralleled the soil behavior at all frequencies. The theoretical plot for only σ dependency shows that the soil deviated from conductive behavior above 10 MHz.

We show the frequency dependence of β in Figs. 6 and 7, along with values based only on σ . The same five minerals of Figs. 2 and 4 show similar behavior (Fig. 6); conduction losses provide a base of only a few dB m^{-1} above 10 MHz, above which another process takes over. For example, β for the anorthoclase, with the highest $\sigma = 0.0048 \text{ S m}^{-1}$, reached 13.1 dB m^{-1} by 1 GHz, which is 9.6 dB m^{-1} higher than the rate caused by only σ . Only anorthoclase and calcite exceeded 10 dB m^{-1} by 1 GHz. In contrast, β for the other minerals and the soil generally exceeded 10 dB m^{-1} above 100 MHz (Fig. 7), and by 1 GHz the crystallites, montmorillonite, and soil reached 47, 70, and 101 dB m^{-1} , respectively. Most importantly, the soil rate is almost linear for all frequencies. The large loss for this

Figure 7. The one-way attenuation rate for three minerals plus soil, and the theoretical rate for a model in which only σ determined ϵ'' , and $\epsilon' = 5$.

soil above 100 MHz is obviously not caused by only σ .

Fig. 8 shows ϵ^* for relatively dry quartz, gypsum crystallites, and soil. All show the same range of relaxation frequency. The CaSO_4 is the lossiest. By comparison with Fig. 7, the addition of water further activated these crystallites to make the soil lossier than the crystallites themselves despite its presence at only 10%. Even in this virtually dry state, the soil shows a loss of 11 dB m^{-1} by 1 GHz. The quartz attenuation curve still shows evidence of free water relaxation but the rate is much less than in Fig. 6, having dropped from 10 to 4.5 dB m^{-1} at 1 GHz. Even at such low water content, a median grain size of 8μ would support more than 20 monolayers of water, and thus most was likely still free.

IV. MODELS

We used the complex refractive index model (CRIM) [3; 12], in which the complex refractive index, n^* , of a material is determined by the sum of the indices of the constituent components, weighted by their volumetric content, v , such that

$$n^* = n_m v_m + n_w v_w + n_a v_a, \quad (3)$$

where subscripts m, w, and a stand for mineral, water and air, respectively, and $n_a = 1$. The CRIM does not present a mechanism by which different materials interact; it is justified by the assumption that statistically, each grain acts independently upon a wave. The formula comes close to predicting the correct values for the quartz, montmorillonite, kaolinite, and gypsum at 1 GHz (reasonably above the low frequency dispersion and below that of free water). This result assumed that $\epsilon' = 5.4$ as in Table 1, but the values are

Figure 8. Permittivity of quartz (dotted), CaSO_4 (dashed) and Iraq soil (solid) at extremely low water contents of 0.11, 0.69 and 0.64%, respectively. The bold solid line is theoretical at the soil value of ϵ' and σ .

Figure 9. The real part for two minerals, the soil, and matching CRIM models (bold curves). CRIM parameters are given in Table 2.

significantly higher by almost 50% for the feldspars (6.2–6.9 vs. 4.0–4.7 measured) and calcite (6.9 vs. 4.7 measured) if water is considered to be in a free state. Following the Cole-Cole relaxation expression, $n_m = \sqrt{\epsilon_m^*}$ and $n_w = \sqrt{\epsilon_w^*}$, where

$$\epsilon^* = \epsilon_{\text{inf}} + (\epsilon_{\text{st}} - \epsilon_{\text{inf}}) (1 + (j f_{\text{rel}})^{-\alpha}) - j \sigma / \omega \epsilon_0, \quad (4)$$

ϵ_{inf} is an asymptotic value of ϵ^* at very high frequency, ϵ_{st} is the very low frequency value, f_{rel} is a relaxation frequency, and α is a broadening factor, caused by collisions or multiple close relaxations.

We modeled the soil, gypsum crystallites, montmorillonite, and quartz. Within any one sample material, we considered the mineral and water to have the same σ because it is a bulk property, and the quartz, gypsum (as explained above) and soil grains to be coated with adsorbed water but with most in a free state. For the montmorillonite, we considered all water to be adsorbed onto the silicate layers ($v = 0.044$ would cover only 16% of the available surface area of $\sim 800 \text{ m}^2 \text{ gr}^{-1}$), which thereby enhanced the effective mineral volume. The continued decrease in ϵ^* for this mineral above 1 GHz (Fig. 3) suggests this was the case.

We show our model results in Figs. 9–11, and the parameters we used to match our results in Table 2, in which the relaxation frequencies are in MHz. We assumed that all

TABLE II. PARAMETERS FOR THE MODEL COMPONENTS.

Comp.	Vol.	σ	ϵ_{mf}	ϵ_{st}	f_{rel}	α
Soil min.	0.629	0.045	2.3	105	1.1	0.50
Soil water	0.073	0.045	5.3	78	20000	0.0
Mont. min.	0.594	0.0003	4.0	145	1.1	0.53
Mont. water	0.0	–	–	–	–	–
Quartz min.	0.592	0.0026	3.0	12	4	0.3
Quartz water	0.056	0.0026	5.3	78	20	0.0
Gypsum min.	0.673	0.025	5.0	27	18	0.24
Gypsum water	0.075	0.025	5.3	78	20000	0.0

Fig. 10. The imaginary part for two minerals, the soil, and matching CRIM models (bold curves). CRIM parameters are given in Table 2.

montmorillonite water was adsorbed and became part of the mineral fraction. Values of σ are those measured except for the montmorillonite, which was fitted because none could be determined. The decimal value of v for air is 1.000 minus the sum of that for the mineral and water; the water having displaced air.

The models fit ϵ^* , ϵ'' , and β well for all samples, with $f_{\text{rel}} = 1$ –20 MHz, as found for clay minerals [2], and theoretically [13]. As expected, ϵ_{st} and f_{rel} for the soil and montmorillonite are similar, as is f_{rel} for the quartz and gypsum. This similarity between the soil and montmorillonite is unexpected because no clay minerals were in the soil. A low ϵ^* above 1 GHz is expected in the real data, but the low soil and quartz ϵ_{inf} was unexpected. The gypsum $\epsilon_{\text{inf}} = 5.0$ is consistent with Table 1.

The models show different influences on β . Free water relaxation affected the quartz above 10 MHz, but only at the highest frequencies for the soil. It is mainly the low frequency relaxation, broadened by $\alpha = 0.50$, which provided high soil β above 100 MHz; i.e., Fig. 7 shows σ supplied a maximum 33 dB m^{-1} yet by 1 GHz, the soil β is near 100 dB m^{-1} . The low ϵ'' and β for montmorillonite at low frequency are unexpected. Although an effective σ cannot be found, $\sigma = 0.0003 \text{ S m}^{-1}$ for the model match, which means either few ions were available

Figure 11. The one-way attenuation rate for two minerals, the soil, and matching CRIM models (bold curves). CRIM parameters are given in Table 2.

for conduction, or little ionic mobility. Consequently, we assumed no free water. The montmorillonite model also required a large $\alpha = 0.53$; the crystallites needed only $\alpha = 0.24$.

The failure of CRIM to better predict ϵ' for the feldspars and calcite is of concern. A $2\ \mu$ particle size would support 200 monolayers of water at 4% water content. Consequently, it is unlikely that most water was adsorbed, which would lower the CRIM values if it was. A possible cause is texture; the more plausible results for the fine clays, crystallites, and quartz was possibly due to better contact with the waveguide and pin. Therefore, it may be safer to use grain sizes $< 30\ \mu$ with TDR.

V. CONCLUSIONS

The association of crystalline gypsum and quartz at low water content caused attenuation rates characteristic of conductive or clay mineral-rich soils, such as montmorillonite. The $\sigma = 0.025$ for the gypsum crystallites suggests that strong contrasts in σ also existed within the crusts or particles themselves. When associated with quartz, the loss exceeded that of the montmorillonite, although the water contents were not exactly the same. The presence of gypsum does not appear to account for the soil behavior of ϵ'' below about 10 MHz.

We conclude that a Maxwell Wagner type of relaxation process was present and enhanced by the small amount of water because of: 1) lack of clay minerals and significant amount of clay-size particles; 2) low conductivity and lack of salts; 3) the deionized water; 4) the dissimilarity of electrical properties between minerals yet the likelihood of all negatively charged surfaces; 5) the lack of significant adsorbed water; 6) the low solubility of gypsum and the almost zero solubility of quartz; 7) the greater loss of the soil relative to quartz in a dry state, and 8) the centering of the relaxation at 1–20 MHz.

Other minor relaxations were surely present; quartz at similar water content showed little loss but similar dispersion, while the gypsum crystallites showed high loss but were only 10% of the soil by weight. The σ contrast between the common, dielectric minerals with distilled water may have been enhanced by some ions formed in solution from the gypsum, and so polarization likely existed within electrical water double layers. Consequently, ϵ^* includes several minor relaxations because of the many ways to polarize charge. These mechanisms are expressed through the large value of α , which spread the loss below, into, and above the GPR bandwidth.

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APPENDIX: COAXIAL SAMPLE HOLDER

Fig. A1 diagrams the TDR (also known as Time Domain Spectroscopy, TDS) process, sample holder and pin. In (a) $V(t)$ is the incident waveform with a 40-ps rise time, and $R(t)$ is the waveform reflected from the sample. The full structure of a $d = 10.53$ -mm pin is shown in (c). More details are found in [2; 3].

Fig. A1. Schematic of TDR system and dynamics of pulse reflection (a), of the sample cell (b), and photograph of sample cell and pin (c).