Fracture Characterization Via Electrical Impedance and Resistance

Lawrence R. Valverde

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Abstract

The electrical impedance and resistance of several sandstones with and without fractures and initially saturated with 1% brine solution have been measured at four frequencies covering three orders of magnitude. Plots of the data with respect to water saturation have been used to characterize the relationship between fractures in rock and the measured electrical properties of the rock. Data have been compared with previous work involving similar measurements and found to differ. Several analytical circuit models have been developed to explain the results obtained in this research and relate the electrical response to fracture content more quantitatively than has been done in previous work. One unfractured sandstone has been cut into a cube shape and subjected to electrical impedance and resistance measurements over the course of evaporation. Two simulated fractures have been created in the cube with repeated experimentation before and after the creation of each fracture. A model, consisting of a resistor in series with a capacitor and resistor in parallel, has been chosen as the most accurate model and used to characterize all the data acquired from experimentation on the sandstone cube. These experiments have verified that the presence of at least one fracture introduces a frequency dependence to the measured impedance and resistance of the rock at very low saturations. However, quantifying this behavior in more detail—specifically with regard to the number and size of fractures present—has proven difficult.
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Chapter 1

1. Introduction

The characterization of fractures in rock is a critical challenge in the development of geothermal resources. A successful geothermal power generation venture is predicated on the presence of a large network of dense fractures within a reservoir of hot rock. Such a network provides the requisite surface area for efficient and sustainable transfer of energy—over the lifetime of a power plant—from the rock medium to the water that flows through the reservoir. Furthermore, while reinjection of reservoir fluid can significantly increase the power-producing life of a given reservoir, insufficient surface area in the reservoir or inappropriate placement of wells can lead to a condition known as thermal breakthrough. When thermal breakthrough occurs, reinjected water cools the formation to an extent that water produced from the reservoir has not been heated sufficiently over its passage from injector to producer, resulting in decreased enthalpy production. When assessing the viability of a resource for geothermal development, understanding the nature of the fracture network at hand can provide valuable insight into how many producing and injecting wells should be drilled, where the locations of those wells should be, what kind temperature profile the produced water will exhibit over the life of the plant, and many other critical elements of the development. Furthermore, when conducting fracture stimulation for the development of enhanced (or engineered) geothermal systems (EGS), it is important to understand the outcomes of stimulation operations so that the process can be adjusted for optimal effect in a specific reservoir.

The practice of reservoir characterization has taken advantage of the electrical properties of rocks, in-situ fluids, and deliberately injected fluids for a wide variety of specific applications and methods for many years. The flowing fluid electric conductivity logging method introduced by Tsang et al. in 1990 and its subsequently developed intricacies and improvements along with direct current resistivity, magnetotelluric, and self-potential electrical surveys such as those described by Garg et al. (2007) are just a few examples of the benefits of measuring electrical properties in geologic formations. The underlying property upon which many of these methods rely is the electrical conductivity of the objects under consideration. Conductivity of a structure of interest, such as fractures, is not simply a function of the conductivity of the rock and fluids. Rather, initial fracture aperture and contact area, fracture surface geometry, elastic moduli of the rock, and confining pressure or normal stress acting across a fracture are all factors to bear in mind (Stesky, 1986). Thus, the electrical properties of rocks under various conditions have been subjects of investigation for several decades. In particular, there are established methods of using direct current for fracture characterization at the borehole and there is an extensive body of literature investigating the properties of rocks under alternating current, especially variations in dielectric constant. However, there has been no investigation known to the author of this research into the use of alternating current toward a method of fracture characterization. Therefore, the intent of this research has
been to investigate the relationship between fracture geometry and electrical impedance and resistance measured at varying frequencies with the goal of applying the relationships found to the development of a novel fracture characterization technique.

Olhoeft (1981) and Knight (1984) give an excellent overviews of the basic principles governing the electrical properties of rocks. The flow of electrical current is governed by responses to electric fields, the process of which can be described by a set of transfer functions resulting from Maxwell's equations. Among the results of these transfer functions are:

\[
\begin{align*}
D &= \varepsilon E \\
J &= \sigma E \\
B &= \mu H
\end{align*}
\]

where:

- \(E\) = electric field intensity (volt/meter)
- \(H\) = magnetic field intensity (ampere – turn/meter)
- \(D\) = electric displacement (coulomb/m\(^2\))
- \(J\) = electric current density (ampere/m\(^2\))
- \(B\) = magnetic induction (weber/m\(^2\))
- \(\varepsilon\) = dielectric permittivity (farad/m)
- \(\sigma\) = electric conductivity (mho/m or siemens/m)
- \(\mu\) = magnetic permeability (henry/m)

These relations apply for materials that exhibit electromagnetically linear behavior, which is usually the case for rocks and minerals. Because the magnetic properties of most naturally occurring materials are generally negligibly different from those of free space, electrical properties are the focus of study in this research. A further important relation that applies to homogeneous, isotropic, electrically conductive materials is:

\[
\nabla \cdot E = 0
\]

which means that in real materials possessing imperfections, charge accumulates at the places where the electrical field changes—around defects, crystal boundaries, surface interfaces, etc. The electrical response to an applied alternating field of the form:

\[
E = E_0e^{i\omega t}
\]

in which:

- \(i\) = the imaginary unit, \(\sqrt{-1}\)
- \(\omega\) = frequency (radians/sec)
- \(t\) = time (sec)
is composed of loss (or conduction) components in phase with the applied voltage and storage (or displacement) components out of phase. The degree to which storage is out of phase with loss is measured by the loss tangent of the system:

\[ DF = \frac{1}{Q} = \tan(\delta) = \frac{R_s}{X_s} \]  

(1-6)

where \( DF \) is the unitless dissipation factor, and \( Q \) is the quality factor, \( \delta \) is angle by which the storage is out of phase in the complex plane, and \( R_s \) and \( X_s \) are the resistance and reactance, respectively, of a series RC or RL circuit. The total current under such an applied electric field is thus:

\[ J = (\sigma + i\omega\epsilon)E_0e^{i\omega t} \]  

(1-7)

One can define total electrical conductivity in terms of free carrier conduction and dielectric displacement such that

\[ \sigma_T = \sigma'_T + i\sigma''_T = \sigma + i\omega\epsilon \]  

(1-8)

where ' indicates the real part and " the imaginary part of a complex variable. Further complexity arises from the fact that both \( \sigma \) and \( \epsilon \) may be independently complex (Fuller and Ward, 1970) such that:

\[ \sigma'_T = \sigma' + i\omega\epsilon' \]  

(1-9)

\[ \sigma''_T = \sigma'' + \omega\epsilon' \]  

(1-10)

Alternatively, many sources in the literature work in terms of complex resistivity, which is the reciprocal of the complex total conductivity:

\[ \rho' - i\rho'' = (\sigma'_T + i\sigma''_T)^{-1} \]  

(1-11)

which is expressed in terms of ohm-m.

Charge is transported through an electric field via electrons, protons, or ions. These charge carriers are moved through a variety of processes, and the nature of these processes, the accumulation of charge at discontinuities during transport, and the distribution of charge before and after transport can all influence the electrical properties of a given medium. The principal methods of charge transport relevant to this research are migration—the diffusion or percolation of charges in response to an electric field, and tunneling—the quantum statistical movement of charges through an energy barrier (relevant to the movement of charge across interfaces between dissimilar materials). There are many mechanisms by which charge separation occurs as well, including: orientational polarization—reorientation of polar molecules, notably water, in response to
an external electric field; interfacial polarization—separation and accumulation of charges at local irregularities in an applied external electric field during charge migration due to that electric field; Galvani and Nerst potentials—differences between the inner potentials of two materials in contact or a liquid and solid in contact, respectively; and sedimentation potential—due to precipitation of charged particles from suspension in a fluid.

The transport of charge through materials such as rocks can be represented through equivalent electrical circuits. The storage components of the electrical response can result in what are known as leading and lagging current elements. These are cases where capacitive or inductive elements, respectively, store energy such that current appears up to ninety degrees ahead or behind the phase of the applied oscillating electric field. The complex impedance, $Z$, of a series RLC circuit is measured as:

$$Z = R_s + iX_s$$  \hspace{1cm} (1-12)

where $R_s$ represents the loss of the system and $X_s$ represents the storage. For capacitive reactance:

$$X_s = -\frac{1}{\omega C_s}$$  \hspace{1cm} (1-13)

and for inductive reactance:

$$X_s = \omega L_s$$  \hspace{1cm} (1-14)

where $C_s$ and $L_s$ represent the series capacitance and inductance, respectively. These elements are based mainly on the geometry of the system. For example, the capacitance of the essential capacitive element, a parallel-plate capacitor, is given by:

$$C = \varepsilon_0 \varepsilon \frac{A}{d}$$  \hspace{1cm} (1-15)

where $\varepsilon_0$ is the dielectric permittivity of free space ($\sim 8.85 \times 10^{-14}$ F/cm), $A$ is the plate area, and $d$ is the distance between the two charged plates. Equation 1-15 also evinces the importance of understanding the dielectric permittivity of a material in order to fully characterize its capacitance.

The complex nature and myriad permutations of elements in rocks has made the investigation of dielectric permittivity, or dielectric constant, of various rocks in a variety of environments a topic of active research for several decades. Knight and Nur (1987) tested the effects of water saturation on dielectric constant in sandstones measured between 10 kHz and 1 MHz. They identified three regimes of behavior corresponding to monolayer coverage of the pore space, multilayer coverage of the pore space, and filling of the bulk volume of the pore space with water. The limited mobility of water molecules
in the region of monolayer coverage means that \( \varepsilon \) shows little variation from that of dry rock. There is also little variation in \( \varepsilon \) once the rocks are above a critical saturation level. However, in the intervening saturation regime of multilayer surface coverage, insulating components such as sandstone grains and gas pockets contribute capacitive effects leading to increases in \( \varepsilon \). Variation of \( \varepsilon \) with saturation is thus highly dependent on the internal geometry of the rock and can be used as some indication of the surface area-to-volume ratio of the pore space. The capacitive effects of gas pockets and the differences in wettability between water and air, however, result in hysteresis with regard to drainage versus imbibition which must also be taken into account. Knight and Nur (1987b) also find a frequency dependence in \( \varepsilon \) indicative of conductive or Maxwell-Wagner effects—charge accumulation at material interfaces due to different charge carrier relaxation times. Different charge carrier relaxation times can be caused by varying mobility of ions or orientation of polar molecules like water in the adsorbed and bulk phases within the rock (Jonscher, 1999). Bona et al. (2002) present further evidence of the influence of internal geometry on the electrical properties of rocks by noting a power law relation between complex permittivity and frequency on the order 100 to 1000 Hz. They attribute this relation to dispersive transport processes caused by the fractal nature of rocks studied. Rusiniak (2002) investigates relationships between relaxation times in the polarization of water and water saturation in the porous structure of laboratory-made gypsum. These experiments, conducted between 5 Hz and 15 MHz, indicate an increase in \( \varepsilon \) and capacitance at lower frequencies.

Rust et al. (1999) investigate analogous relationships among \( \varepsilon \), capacitance, and pore space in volcanic rocks. A critical point extrapolated upon later in this report is that a pore space consisting almost exclusively of fractures such as that found in the Mount Meager MB1 sample can exhibit a much more pronounced relationship between \( \varepsilon \) and frequency than that of a more homogenous rock with a porous matrix. Cracks can host a layer of adsorbed water on the order of 10 angstroms which increases \( \varepsilon \). This increase in \( \varepsilon \) is likely due to the exaggerated capacitive effect of charge collection along the adsorbed water layer.

Börner et al. (1997) also find a positive relation between \( \varepsilon \) and surface area-to-volume ratio, noting a strong increase in \( \varepsilon \) with increasing saturation. Taking measurements over a frequency range of \( 10^{-3} \) Hz to \( 10^{9} \) Hz, Börner et al. also identify a low- and a high-frequency response explainable by separate phenomena. The low frequency response—in which complex conductivity is relatively constant—is a function of water content, water conductivity, and surface area-to-porosity ratio; meanwhile the high frequency response—in which complex conductivity rises linearly with frequency—depends upon water content and internal surface area.

While the geophysics literature focuses heavily on characterizing dielectric permittivity, and the importance of understanding dielectric permittivity is evident from the discussion until now, most engineering applications focus on more directly measurable properties such as resistance. The measured resistance of a given volume is often treated as a bulk
property and presented in terms of an overall resistivity for the sample under consideration. The most generally accepted relation between saturation and resistivity is Archie’s law (Archie, 1942), which describes resistivity in terms of the resistivity index, $I$, where:

$$ I = \frac{R_t}{R_0} $$

such that:

$$ I = S_w^{-n} $$

where $R_0$ and $R_t$ are the resistivities of fully and partially water-saturated rock, respectively; $S_w$ is the water saturation and $n$ is an empirically derived constant. However, the assumption that the relationship between $I$ and $S_w$ follow a constant and unique power law relation for a given porous medium as well as the assumption that the $n$ values determined are equally applicable at the core and reservoir scales are invalid in many situations. Suman and Knight (1997) found that Archie's law may be realized only in strongly water-wet systems in which a sufficiently thick surface water film is present. Suman and Knight also noticed a negative correlation between the value of $n$ and the standard deviation of pore sizes, attributing this relation to the improved electrical connectivity of a larger number of pores filled with water when there is a high standard deviation of pore sizes. These findings may extend to the fractures as an extreme case of pore size standard deviation, with very low values of $n$ above a critical saturation and a breakdown in Archie's law below that critical saturation. In their investigation of moist limestone samples, Bekhit and Khalil (2007) note that for frequencies between 1 Hz and 107 Hz polarization of water molecules on internal surfaces lead to capacitive effects that often dominate the measured impedances. While a surface monolayer of water leads to Debye behavior at high frequencies (beginning on the order of several kHz), Bekhit and Khalil attribute the overall Warburg impedance behavior to bulk ion diffusion in the filled pore space.

A recent study by Sandler et al. (2009) highlights a marked difference among the frequency responses of rock cores lacking or containing fractures. The study found that in fractured rocks, below a certain level of water saturation, resistivity index was inversely proportional to frequency over the frequency range of 100 Hz to 10,000 Hz. Figure 1-1 illustrates these findings by showing resistivity index as a function of water saturation (for a 1% salt solution) for sandstone lacking (left) and containing (right) fractures. While the unfractured sample demonstrates a power-law relation over the whole range of saturations tested, the resistivity response of the fractured rock splits below a saturation of about $S_w = 0.1$, varying depending on frequency. The initial impetus for the current research was a desire to verify and explain the results obtained by Sandler et al.
Figure 1-1: The frequency dependence of resistivity in fractured sandstone. Core 1 (left) is Berea sandstone without fractures. Core 2 (right) is fractured sandstone. Adapted from Sandler et al. (2009).
Chapter 2

2. Resistance and Impedance Measurement of Rocks

Over the course of the experiments outlined below, several different rock cores were used. The properties of these rocks as well as the numbering system by which they can be identified in this report are presented in Table 2-1. Figures 2-1 through 2-4 show pictures of these rocks.

Table 2-1: Summary of properties for rocks used in this research

<table>
<thead>
<tr>
<th>Core #</th>
<th>Description</th>
<th>Length (mm)</th>
<th>Cross-Sectional Area (mm²)</th>
<th>Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Berea Sandstone</td>
<td>98.43</td>
<td>387.9</td>
<td>18.19</td>
</tr>
<tr>
<td>2</td>
<td>Berea Sandstone</td>
<td>98.43</td>
<td>387.9</td>
<td>18.19</td>
</tr>
<tr>
<td>3</td>
<td>Unfractured Reservoir Rock</td>
<td>49.21</td>
<td>570.0</td>
<td>9.33</td>
</tr>
<tr>
<td>4</td>
<td>Fractured Reservoir Rock</td>
<td>39.69</td>
<td>618.54</td>
<td>27.7</td>
</tr>
<tr>
<td>5</td>
<td>Sandstone Cube</td>
<td>50.8</td>
<td>2580.64</td>
<td>15.0</td>
</tr>
</tbody>
</table>

Figure 2-1: Berea sandstone (core #1). Due to the similarity of cores #1 and #2, no separate photo is shown for core #2.
Figure 2-2: Unfractured reservoir rock (core #3).

Figure 2-3: Fractured reservoir rock (core #4)

Figure 2-4: Sandstone cube (core #5) (secured with experimental setup)
2.1. Reproduction of Sandler et al. (2009)

As the initial portion of this research was intended to replicate the experimental procedures and results of Sandler et al. (2009), the procedure detailed in that paper was followed, with certain details augmented by personal communication with the authors. A Berea sandstone core—Core #1 in Table 2-1—without fractures was saturated with 1% NaCl brine solution. Personal communication with the authors provided information that the core was to be evacuated before saturation to remove all gas from the pore space. Between experimental runs, residual water from the previous run was removed by heating in a vacuum oven at ~70° F. The question of ion buildup between runs was determined to be insignificant and is discussed in greater detail in Appendix B. The core was placed in a vacuum chamber and evacuated to below 200 mTorr before the brine solution was admitted to the chamber and allowed to soak overnight. Figure 2-5 shows a picture of this vacuum setup. The same QuadTech Model 1715 RCL meter as that used by Sandler et al. was used alongside a Mettler Toledo balance with accuracy of ~0.02 g, as opposed to the Satorius balance used by Sandler et al. From personal communication with the authors, electrodes were constructed by soldering wires to two square patches of copper mesh approximately 400 mm². The other ends of the wires were connected to alligator clamp leads from the LCR meter. The electrodes were fixed to either end of the rock core by a hand clamp. The core was then placed upon the balance as shown in Figure 2-6. A rubber sheet between balance and sample prevented electrical connection between the sample and the metal plate of the balance. After having conducted the experiment in the configuration detailed here, later correspondence with the Sandler et al. authors revealed that in the original experiment a piece of filter paper saturated in the same brine solution as the rock was placed between the sample and the copper mesh. The experiment was conducted again for this research with the addition of filter paper and no significant difference was found between experimentation with or without filter paper.
Figure 2-5: Rock evacuation apparatus

Figure 2-6: Balance configuration without filter paper. Wires lead out of frame to LCR meter.
Both the balance and the RCL meter were connected to a computer via RS-232 ports, and programs of the National Instruments LabView software for both measurement devices were used to gather mass, resistance, and impedance data from the core at ten-minute intervals while water was allowed to evaporate from the core at ambient temperature of about 20° C. The LabView vi programs are detailed in Appendix A. Measurements of electrical properties were taken at frequencies of 100 Hz, 120 Hz, 1 kHz, and 10 kHz. These were the frequencies available with the LCR meter used. The change in mass of the sample as measured by the balance was used to determine the mass of water evaporated as a function of time, thereby finding the saturation as a function of time.

2.2. Initial Results with Various Rocks

2.2.1. The Berea Sandstones (Cores #1 and #2)

Figure 2-7 shows water evaporated as a function of time for core #1. The core reached nearly complete desaturation after about 1000 minutes.

\[ S_w = 1 - \frac{m_w(t)}{m_{w,0}} \]  

(2-1)

where \( m_w \) is the mass of water lost at time \( t \), and \( m_{w,0} \) is the initial water mass. Figures 2-8 through 2-10 show the measured electrical properties of core #1 as a function of saturation. Properties are represented as analogues of resistivity index as defined above such that impedance index is the same formula as in Equation 1-16 but with impedance in place of resistance and likewise for reactance.
Both impedance and series resistance were properties measured directly with the LCR meter. Reactance was calculated using vector definitions of these properties such that:

\[ X_s = \sqrt{Z^2 - R_s^2} \]  

(2-2)

These properties were verified by using the LCR meter to directly measure impedance and reactance at several points as well. The measured and calculated values of \( X_s \) were
the same to within the accuracy of the machine. Reactance data are no longer plotted after Figure 2-10 to avoid redundancy in light of the straightforward relation in Equation 2-2.

Figure 2-10: Experimental reactance data measured at four frequencies in core #1.

These results are noticeably different from those obtained by Sandler et al. (2009). Most glaringly, there is no difference in the resistivity index for the unfractured Berea sandstone measured by Sandler et al., yet the resistivity index measured in this research for the same type of rock demonstrates a frequency dispersion very similar to that measured by Sandler et al. for fractured rocks.

To verify the results shown in Figures 2-8 through 2-10 the same procedure was repeated with core #1. The same results as in the first experimental run with core #1 were found again with only a slight deviation in mass change over time. This slight discrepancy in measurements from the balance was a foreshadowing of what was to become the greatest source of uncertainty throughout this research: mass determination. Representing the change in mass in terms of change in saturation over time, Figure 2-11 clearly illustrates the unphysical nature of the results obtained. The saturation falls below 0 between approximately 1600 and 2300 minutes after the start of the experiment. There were several other experimental runs made with cores #1 and #2 as well as other cores in which mass data were too noisy to be useful. The likely source of the problem in the saturation data in Figure 2-11 that will be discussed in greater detail in Section 2.3, was that of relaxation of the wires used to connect the electrodes to the LCR meter. The clearly unphysical saturations obtained, however, do not affect the accuracy of the magnitudes measured for impedance and resistance nor do they significantly change the qualitative nature of the data and their similarity to the first data for core #1 shown above. Figures 2-12 and 2-13 demonstrate that the impedance and resistance indexes remain at nearly the same values as in the first run, demonstrating the same frequency dispersion.
Figure 2-11: Change in water saturation as a function of time in core #1 (unfractured Berea sandstone). The missing data points are between -0.01 and 0.

Figure 2-12: Experimental impedance data measured a second time at four frequencies in core #1.
Figure 2-13: Experimental resistance data measured a second time at four frequencies in core #1. Cores #1 and #2 are both Berea sandstone that originated from one core that was subsequently cut lengthwise to create the two cores of nearly equal dimensions. The same experimental setup as used for core #1 was repeated with core #2, yielding similar results but again exhibiting problems with mass accounting. Figure 2-14 illustrates the problem with collecting data over such long periods of time. After approximately 1800 minutes the mass loss curve shifts downward by ~1 g while the trend in the slope of the curve appears to remain the same. Given the physical impossibility of such a rapid increase in mass as well as the overall shape of the curve otherwise, it is most likely that there was a problem with the mass balance at the time when the curve shifted. Figures 2-15 and 2-16 demonstrate the similarities between the impedance and resistance data collected for core #1 and core #2. Due to the mass problems, these data are slightly difficult to read, but the two most obvious points to compare and contrast between cores #1 and #2 are that while there remains the same order of magnitude in separations between curves measured at 100 Hz, 1 kHz, and 10 kHz at low saturations, the overall orders of magnitude for the curves from core #2 are consistently higher than those for core #1 at low saturations.
Figure 2-14: Water evaporated as a function of time in core #2 (unfractured Berea sandstone).

Figure 2-15: Experimental impedance data measured at four frequencies in core #2.
When the information regarding the use of filter paper in the experiments by Sandler et al. (2009) came to light, the experiments were repeated with the addition of filter paper soaked in brine and placed between the copper mesh electrodes and the rock core sample. Figures 2-17 and 2-18 demonstrate that although the ultimate values reached for impedance and resistance indexes are lower than those from the experiment without filter paper, the same trend in frequency dispersion persists. It was determined that the presence or lack of filter paper was not the source of discrepancy between this research and that of Sandler et al.
Figure 2-18: Experimental resistance data measured at four frequencies in core #2 with filter paper.

### 2.2.2. A Different Unfractured Reservoir Rock

After the confusing results with the Berea sandstone, a different reservoir rock that was also unfractured, core #3, was subjected to the same experimental conditions as cores #1 and #2. Figures 2-19 and 2-20 demonstrate very similar results as those obtained from the Berea sandstones. Similar mass measurement issues as those with the first run on core #2 were encountered, resulting in plateaus at lowest saturations appearing disconnected from, and behind, the main curve, hovering near $S_w = 0.01$ in the following figures. This core was also tested again with filter paper to reinforce the conclusion that the inclusion of filter paper was not responsible for eliminating the frequency dispersion observed. Figures 2-21 and 2-22 below show almost identical results to those for the experiment conducted without filter paper.
Figure 2-19: Experimental impedance data measured at four frequencies in core #3.

Figure 2-20: Experimental resistance data measured at four frequencies in core #3.
2.2.3. A Fractured Reservoir Rock

After similar results for different types of unfractured rock were found, a fractured reservoir rock, core #4, was subjected to the same experiments as the previous cores. Luckily, very clean mass data yield very clear curves in Figures 2-23 and 2-24 which both demonstrate the same frequency dispersion behavior as seen for the unfractured rocks. The notable difference other than the much less noisy data is the fact that onset of the frequency dispersion occurred at higher saturations than in any of the other rocks tested up to that point.
2.3. Sandstone Cube

Over the course of the experimentation documented in Section 2.2, a theoretical explanation for the phenomena observed based on the underlying physics was under investigation simultaneously. While developing the theory outlined in Chapter 3, a problem greater than that of wire relaxation was found with the experimental setup. Electrode effects could dominate the electrical responses reported here in Chapter 2. The electrode effects are rooted in the fundamental complication associated with measuring electrical properties across materials with different modes of conduction. While rocks are ionic conductors, the metal electrodes are electronic conductors, and an accumulation of ions where current crosses the rock/electrode interface can lead to impedances which
might overwhelm behavior of the rock itself. Such a configuration of electrodes is known as blocking electrodes. Ion accumulation is of greatest concern under direct current or low frequency alternating current, where the electric fields force ions to migrate until they contact metallic boundaries which cannot be crossed (Drury, 1978). The frequency dependence of this effect is also partially related to the relaxation time of the polar water molecules and their ability to shift in response to the changing electric fields (Rusiniak 2002). In their investigation of the electrical properties of partially saturated Topopah Spring tuff, Roberts and Lin (1997) note two to three regimes of electrical behavior depending on frequency. At higher frequencies one accesses the response of the rock/water system. For moderate to low saturations this rock water system represents conduction in the water monolayers coating the pore space. At lower frequencies one accesses the electrode response. Additionally, for the relevant saturations, intermediate frequencies sample the behavior of the pore water (Roberts and Lin, 1997). Börner (1997) also points out that low frequency behavior is indicative of water content, water conductivity, and surface area to volume ratio. Meanwhile, the high frequency behavior is related to water content and internal surface area, independent of water conductivity.

The alternative to blocking electrodes is what is known as reversible electrodes. A reversible electrode works through the exchange of charge carriers across the sample-electrode interface via an electrochemical reaction. For example, Scott et al. (1967) tested rock samples with silver-silver chloride electrodes under the assumption that current in the rock is conducted through chloride ions. Chloride reacts with free silver to create silver chloride, thereby incorporating the ions into the electrode. However, in addition to the problem that reversible electrodes are geared toward only specific charge conductors, good contact with the sample is difficult to engineer. These electrodes are usually applied by soaking a blotter in the appropriate solution, leading to a setup very similar to the filter paper setup used here. An air gap, which could be made especially egregious over the course of evaporation, can create a capacitance large enough to dominate the measured response.

Thus, the electrodes were redesigned to minimize electrode effects and ascertain the true influence of fractures on the electrical properties measured. Consultation of Knight and Nur (1987) as well as personal communication with Professor Rosemary Knight at Stanford University prompted a design which incorporated 100 nm of platinum sputtered to either side of the rock sample under study, effectively binding the electrodes to the rock, ensuring the best possible contact with the rock. Platinum is relatively inert and is a blocking electrode, but the capacitances that will be caused by charge build-up at the interface were deemed less problematic than those that are caused by a poor connection to the sample.

The electrodes connecting to the LCR meter were also changed from copper mesh soldered with wires to a mesh of stainless steel, in part due to the observed oxidation of the copper mesh. Two plates of PVC plastic approximately 6.5 cm by 7.5 cm were prepared with four holes each, one in each corner. The stainless steel mesh was attached to the PVC by screwing through the mesh and into the hole at each corner. In one corner the screw was tightened around a wire, the other end of which was connected to the LCR
meter (Figure 2-25). Furthermore, a frame was constructed around the mass balance with an elevated crossbeam to which the electrode wires were secured. From the secured location one end of the wire was allowed to hang loosely and attach to the electrodes and the other end of the wire connected to the RCL meter. A much thinner and less rigid wire was also selected to allow for more quick and less noticeable relaxation of the wire.

![Stainless steel mesh electrode](image)

Figure 2-25: Stainless steel mesh electrode

A new rock was chosen for the new experimental setup: Core #5 began as a sandstone sample cut into a cube approximately 50.8 mm on each side. The cube was chosen with the intention of make several cuts and measuring along different axes as described later in this section. The platinum was sputtered directly onto the rock by using the Metallica sputter system in the Stanford Nanofabrication Lab.

### 2.3.1. Uncut Cube

As a control for testing the new experimental setup, the cube was first subjected to the same experimental procedure as all the other rocks and the electrical properties were measured with the same copper electrodes hand-clamped to the rock sample. Figures 2-26 and 2-27 demonstrate that essentially the same frequency dispersion behavior is observed.

After sputtering, the rock was measured again under the new experimental setup. In addition to the effect of changing to the sputtered platinum electrode being tested, the effect of wire relaxation over the course of an actual experimental run was quantified. The results of the first run on the sputtered cube, presented in Figures 2-28 and 2-29 reinforced two suspicions. Namely, the copper electrodes were indeed introducing dominant capacitive effects that were causing the frequency dispersion observed in all the previous experiments. And, wire relaxation was exerting a significant influence on the mass measurements. Over the course of this experiment—as well as two repetitions of the same experiment—there was never any significant difference in impedance or resistance as a function of frequency. Furthermore, after adjusting for the wire relaxation the impedance and resistance log-log curves follow the linear relationship expected of an unfractured rock. The mass of the associated equipment holding the sample—the
electrodes, hand clamps, and wires—were measured before and after the experiment by finding the difference between the masses of the core without the equipment as measured at the start and end of the experiment. The result of this first run on the sputtered rock was a 0.88 g effective increase in the weight of the equipment. The wires were measured separately, disconnected from any rock samples, and it was discovered that mass changed gradually over the course of ~12 hours. This change in mass is likely due to settling of the wires due to changes in tension at a rate unnoticeable to the eye but recognizable on the mass balance. The behavior appeared to be nearly linear so a linear fit was made to subtract the mass added by the wires over time. As became evident over a second and third run with the same setup, however, the wire relaxation varied between runs and was not exactly linear. In a second run of the experiment, over the first hour or two the mass effectively decreased at a rate faster than what could be ascribed to water evaporation before increasing by the end of the experiment. Figures 2-28 and 2-29 present the raw data collected from the experiment, while Figures 2-30 and 2-31 demonstrate the data after correcting for wire relaxation time.

Figure 2-26: Experimental impedance data measured at four frequencies in core #5 with original copper electrode setup.
Figure 2-27: Experimental resistance data measured at four frequencies in core #5 with original copper electrode setup.

Figure 2-28: Experimental impedance data measured at four frequencies in core #5 with sputtered platinum electrodes.
Figure 2-29: Experimental resistance data measured at four frequencies in core #5 with sputtered platinum electrodes.

Figure 2-30: Experimental impedance data measured at four frequencies in core #5 with sputtered platinum electrodes after adjusting for wire relaxation.
Figure 2-31: Experimental resistance data measured at four frequencies in core #5 with sputtered platinum electrodes after adjusting for wire relaxation.

2.3.2. Cut Cube

After the results from the uncut sample #5 were verified through two repetitions of the experiment yielding the same results, the effect of a fracture in the rock was simulated by cutting the rock completely transverse and perpendicular to the line of applied voltage. As demonstrated in Figure 2-32 the rock was not cut in the middle out of anticipation of a future second cut. The intention of the second cut was to confirm the source of any change in behavior with one simulated fracture by observing changes in magnitude of that behavior in the presence of an additional simulated fracture.

Figure 2-32: Sandstone cube with one simulated fracture.

Experiments on the cut sandstone cube demonstrated the expected return to behavior where the measured impedance and resistance depended upon the frequency at which they were measured. Wire relaxation persisted as an issue as well. Figure 2-33 demonstrates inaccuracies in mass measurement due to changes in wire tension as the
wire relaxed. Both the initial negative mass loss values as well as the decrease in mass loss at long time are both unphysical and attributed to wire relaxation. The problem of wire relaxation is further corroborated by a measured 4.44 g increase in the weight of the equipment between the start and end of the experiment. The electrical property data plotted without correcting for mass in Figures 2-34 and 2-35 reveal the qualitative properties as well as impedance and resistance values that were expected, but the relationship to saturation is clearly inaccurate. Applying a linear subtraction for the wire mass gives the curves in Figures 2-36 and 2-37.

![Graph showing water evaporated as a function of time in core #5 with one cut.](image1)

Figure 2-33: Water evaporated as a function of time in core #5 with one cut. Note how mass loss begins below zero and gradually decreases at long time.

![Graph showing experimental impedance data measured at four frequencies in core #5 with sputtered platinum electrodes and one simulated fracture.](image2)

Figure 2-34: Experimental impedance data measured at four frequencies in core #5 with sputtered platinum electrodes and one simulated fracture.
Figure 2-35: Experimental resistance data measured at four frequencies in core #5 with sputtered platinum electrodes and one simulated fracture.

Figure 2-36: Experimental resistance data measured at four frequencies in core #5 with sputtered platinum electrodes and one simulated fracture after adjusting for wire relaxation.
A wire was found that was lighter and less stiff than the wire used previously, and this wire was substituted in to repeat the experiment on core #5. While this new wire did exhibit less effective mass change over the course of the experiment, the mass of the equipment was reduced, rather than increased, 0.41 g. This reduction in mass had the opposite effect as the wires from the previous run, elongating, rather than compressing the data, as seen in Figures 2-38 and 2-39 Correcting for wire relaxation in this run resulted in the data shown in Figures 2-40 and 2-41.
Figure 2-39: Experimental resistance data measured at four frequencies in core #5 with sputtered platinum electrodes and one simulated fracture and new wires.

Figure 2-40: Experimental resistance data measured at four frequencies in core #5 with sputtered platinum electrodes and one simulated fracture and new wires after adjusting for wire relaxation.
Upon confirmation that a cut in the rock resulted in the frequency-dependent behavior, another cut was made to test whether or not an additional cut would lead to an enhancement in the effects observed with one cut. As shown in Figure 2-42, the second cut was made such that the rock was divided into three pieces. The two cuts were spaced evenly between each other and the edges of the rock. Wire relaxation was again an issue in the first experiment on the twice-cut rock, with an effect of 0.57 g decrease in mass over the course of the experiment. Wire relaxation was again accounted for through a linear subtraction of mass. The data after mass correction are shown in Figures 2-43 and 2-44.
Figure 2-43: Experimental impedance data measured at four frequencies in core #5 with sputtered platinum electrodes and two simulated fractures after adjusting for wire relaxation.

Figure 2-44: Experimental resistance data measured at four frequencies in core #5 with sputtered platinum electrodes and two simulated fractures after adjusting for wire relaxation.

The experiment was conducted again on the rock cube with two cuts, and a 0.39 g increase in mass over the course of the experiment was observed. The data after mass correction are shown in Figures 2-45 and 2-46.
Analysis of the data presented in Figures 2-44 and 2-45 (explained in greater detail in Chapter 5) prompted measurements of impedance and resistance at the lowest saturations as a function of tightness with which the electrodes were clamped to the sides of the rock. The clamps were squeezed in five intervals between compression merely sufficient to hold the rock in place and compression such that the clamps could not be squeezed tighter by hand. At each interval 16 measurements were made at each of the four frequencies under investigation. Figure 2-47 shows the average of these measurements.
for impedance and resistance at each interval. Impedance clearly drops with compression, but the resistance behavior is more complicated. The implications of this behavior are discussed in greater length at the end of Chapter 4.

Figure 2-47: Variation in impedance and resistance with rock compression at saturation about 0.01.
Chapter 3

3. Theory

One of the most instructive methods for describing the electrical responses of rocks is to determine the equivalent electrical circuit that best models the electrical response in the data and then relate the elements of this circuit to the various properties and processes in the rock. Knight (1987) provides an excellent summary and analysis of many rock circuit models presented in the related literature. While the total frequency response of rocks is often approximated as a Debye circuit, inspection of Knight (1987) as well as personal correspondence with Professor Rosemary Knight lead to the selection of the circuit model presented in Figure 3-1. Inspection of Equation 1-13 indicates the capacitive nature of the inverse relationship observed in the data between frequency and impedance. When resistors and capacitors are combined in a more complex manner than simple series connection, the total resistance of the circuit can become frequency-dependent as well. For the model in Figure 3-1, $R_1$ represents the resistance from the electrodes and associated equipment while $R_2$ and $C_2$ connected in parallel represent the pore space of the rock. Equation 3-1 illustrates how frequency is incorporated into resistance. As Börner (1997) describes in his design for an equivalent circuit, the pore space is composed of elements where a poorly conducting, nonwetting phase is at the center—air, in this case—and a conducting wetting phase—in this case, brine—coats the surface of the surrounding, nonconducting rock. The conducting phase is further divided into areas with different modes of conduction, such as the diffusive resistance of the bulk water versus the more restricted charge movement in the diffusive double layer which forms at the liquid solid interfaces where ions of one charge are attracted preferentially to the interface and ions of the opposite charge are then attracted to that first layer of adsorbed charges. Given the complexity of the conductive mechanisms in the pore space, several additional circuit models of increasing complexity were investigated as well and are described in greater length in Section 3.2.

![Figure 3-1: Equivalent circuit model 1.](image-url)
\[
R = \frac{R_2^2}{1 + \omega^2 R_2^2 C_2^2} + R_1 \tag{3-1}
\]

\[
X = \frac{-\omega R_2^2 C_2}{1 + \omega^2 R_2^2 C_2^2} \tag{3-2}
\]

### 3.1. Circuit Modeling

In order to develop an understanding of the relationships and interactions between the possible elements of a model circuit, MathWorks MATLAB was used to create a model of the circuit in Figure 3-1 (model 1) using Equations 3-1 and 3-2 as well as the relationship between impedance, resistance, and reactance. The essential underlying rationale to the model is that pore space resistance increases over time, representing the decrease in conductivity as bulk water evaporates due to the brine being the primary conduit of current in the rock and reduction in the brine is analogous to the thinning of a wire through which current is passing. Thus, the pore space resistor represents the resistance of contiguous bulk water, and the capacitor represents the charge accumulated on microlayers of water as evaporation of the bulk water leads to a situation where regions that previously were joined by contiguous water contact become separated electrically. This electrical separation is due to regions of connected water snapping apart, resulting in myriad microcapacitors which are modeled collectively with the capacitance in \( C_2 \). While the water lining pore spaces after bulk water has evaporated—as described in Section 1—provides capacitive effects, fractures are considered the dominant capacitive factor. Fractures can be thought of as analogous to parallel plate capacitors with a capacitance proportional to the quantity and aperture thickness of fractures perpendicular to the direction of applied voltage as defined by the relationship in Equation 1-15. Graphs of the parameter space were used to gain insight as to which parameters would influence specific behavior in the overall resistance and impedance measured. Inspection of Figures 3-2 and 3-3 prompted making the slices shown in Figures 3-4 and 3-5. With an appropriate choice of parameter values, the slices bear remarkable resemblance to the data plotted in Section 2. Furthermore, it is possible that while the bulk water is evaporating there is a relatively stable relationship between saturation and internal resistance such that a monotonic decrease in saturation results in a monotonic increase in the value for \( R_2 \). However, after a certain critical saturation, enough of the pore water snaps off from itself to increase \( R_2 \) dramatically due to the sudden disconnectedness of the conductive fluid. The result should be something like what is shown in Figure 3-6. In the case of this figure, the x-axis does not truly represent \( R_2 \), rather it is a qualitative representation of decreasing saturation where there exists some relationship between saturation and \( R_2 \) up until \( \log(R_2) = 4.5 \), after which \( R_2 \) plateaus or asymptotes to one value for the remaining lowest saturations.
Figure 3-2: Variation of overall impedance measured for circuit model 1 at varying values of $R_1$ and $R_2$ with $\omega$ and $C_2$ held constant.

$\omega = 1, \ C_2 = 1$

Figure 3-3: Variation of overall resistance measured for circuit model 1 at varying values of $R_1$ and $R_2$ with $\omega$ and $C_2$ held constant.

$\omega = 1, \ C_2 = 1$
Figure 3-4: Qualitative behavior of impedance with decreasing $R_2$.

Figure 3-5: Qualitative behavior of resistance with decreasing $R_2$. 
Figure 3-6: Resistance behavior under the assumption that after a certain point (in this case \( \log(R_2) = 4.5 \)) \( R_2 \) remains constant. After this point, the x-axis no longer represents resistance, rather the overall x-axis is equivalent to the saturation of the rock.

3.2. Alternative Circuits

In addition to the circuit model in Figure 3-1, four other models were considered to account for the great complexity of the actual unknown equivalent circuit to the rocks' electrical response.

Model 2, shown in Figure 3-7 incorporates an additional capacitor in series before the bulk rock section of the circuit in order to separately account for capacitance at the rock/electrode interface.

Figure 3-7: Equivalent circuit model 2.

Model 3, shown in Figure 3-8 incorporates a Debye circuit for the bulk rock circuit. This change might better account for the varying capacitances due to varying pore size, particularly if the pores represent one internal capacitance and the fractures represent another.
Circuit model 4, show in Figure 3-9, is a variation of model 3 such that $C_3$ is now a resistor, $R_3$. This change reflects the possibility of varying resistances within the pore space being more dominant than varying capacitances. In this case $C_2$ represents the capacitance of the fractures.

Finally, circuit model 5, show in Figure 3-10, represents a combination of models 3 and 4. Although the complexity of this model already is likely too great for the sake of making a simplified model that provides intuitive insight into the fracture properties of the rock, this model was used when fitting the data to the models to determine if significantly greater accuracy in matches could be obtained by the increased complexity.
Chapter 4

4. Fitting the Model to the Data

Given the highly nonlinear nature of the impedance and resistance curves, nonlinear optimization methods were used to fit the curves. The free parameters to be optimized were the various circuit elements other than whichever resistor represented decreasing saturation in the rock. In order to avoid the extreme computational difficulty of a mixed integer optimization problem of this size, circuit elements were effectively shut off by reaching values with extreme negative or positive orders of magnitude. The Sparse Nonlinear OPTimizer (snopt) solver for AMPL (A Mathematical Programming Language) was used for optimization operations. The fit of the representative circuit equation to the experimental data was performed by minimizing the error between the real portion of the complex admittance and the experimental resistance data as well as the error between the bulk rock resistance used for the model and a power law approximation of the pore space resistance based on saturation data. Error is determined by the method of least squares. The objective function for the optimization is thus:

Minimize $\text{Error} =$

$$\sum_{i=1}^{n} \sqrt{[\log(R_{\text{data}}(t,\omega)) - \log(R(t,\omega))]^2 + [\log(S_{w}(t)^{-\lambda(t)}) - \log(R_{2}(t))]^2} +$$

(4-1)

$$\sum_{i=1}^{n} \sqrt{[\log(Z_{\text{data}}(t,\omega)) - \log(Z(t,\omega))]^2 + [\log(S_{w}(t)^{-\lambda(t)}) - \log(R_{2}(t))]^2}$$

where:

- $E = \text{The sum of error between the data and model fit. [ohm]}$
- $R_{\text{data}}(t,\omega) = \text{The experimentally measured resistance index for a given saturation and frequency at time } t. \text{ [ohm]}$
- $Z_{\text{data}}(t,\omega) = \text{The experimentally measured impedance index for a given saturation and frequency at time } t. \text{ [ohm]}$
- $R(t, \omega) = \text{The total resistance index of the representative circuit as defined by the characteristic equations for that circuit, such as Equations 3-1 and 3-2 for model 1, Figure 3-1, at a given time and frequency. [ohm]}$
- $Z(t, \omega) = \text{The total impedance index of the representative circuit as defined by the characteristic equations for that circuit, such as Equations 3-1 and 3-2 for model 1, Figure 3-1, at a given time and frequency. [ohm]}$
- $R_{2}(t) = \text{The equivalent resistance of the pore space corresponding to decreasing saturation at time } t. \text{ [ohm]}$
- $S_{w}(t) = \text{The experimentally measured saturation at time } t.$
- $\lambda(t) = \text{A variable parameter relating saturation to resistance.}$
$t = 0\ldots T$, The time steps corresponding to saturation points for which resistance and impedance data were measured, where $T$ is the last time step.

$n = 1\ldots T$, The time steps over which the error was summed. The first step was left out because this would invariably be zero by definition of the problem statement, and snopt has trouble evaluating the square root of zero.

$\omega = \text{The frequency at which measurements were made.} \ [\text{rad}]$

Each resistance or impedance index was defined as shown in the example in Equation 4-2 in which $R$ is the relevant property and $R_0$ is the value of that property at time 0.

$$\text{Resistance Index} = \frac{R}{R_0}$$ (4-2)

The power law relationship for pore space resistance was inspired by Archie's law. Archie's law refers to the overall resistance of a rock sample as a function of overall saturation and the resistance in question for this optimization is an internal resistance. Yet, any relationship could be chosen, and Archie's law was itself initially an empirical observation, so an analogy was drawn from Archie's law to internal resistance as a function of saturation at different points in the rock. The decision variables for the optimization were the various other circuit elements of the circuit model under consideration. Taking model 1 as an example, the decision variables would be $R_1$ and $C_2$, in ohms and farads, respectively, as well as $\lambda(t)$ to match the $R_2$ with $S_w(t)$. The two constraints imposed were that $\lambda(t)$ must be greater than or equal to zero and that $R(t)$ be greater than or equal to $R(t-1)$. The limited number of constraints resulted in a large feasible solution space; this fact combined with the nonlinearity of the problem means that there were many unstable solutions. Sensitivity analysis was conducted to characterize the robustness of the optimization as well as to help in finding the region of the solution space with the lowest errors.

For the sensitivity analysis of the model fits, the starting points for all parameters except $\lambda$ were varied at each order of magnitude between $10^{-4}$ and $10^5$. Due to inability of the program to handle inordinately large exponents, and given the fact that $\lambda$ is likely on the order of magnitude of one, $\lambda$ was varied between $10^{-8}$ and $10^2$. A deliberately wide range of parameter starting points was chosen to scan as much of the solution space as possible and to gain insight into how precise of a best possible fit might be obtained. Data chosen for the first attempts at curve fitting were those from the first experimental run on core #5 with the initial copper electrode setup. In a further attempt to make the problem more tractable for AMPL, initial sensitivity analysis was conducted on optimization of a fit for only the overall resistance data. The resistance data were chosen as opposed to the impedance data because the behavior of the resistance curves was more varied than that of the impedance curves. Hence, it was assumed that fitting the resistance curve data would better constrain the parameter values for the model circuit. Initial analysis of model 1 revealed relatively little dependence on $\lambda$ and $R_1$, but there was a noticeable drop in the errors achieved at lower values of $C_2$. Surfaces relating $C_2$ to $R_1$ at each of the ten
starting values of \( \lambda \) are plotted on top of each other in Figure 4-1. Notice that for nearly all values of \( C_2 \) over \( 10^{-2} \) there is almost no variation in the surfaces. For the sake of error comparison with model fits to different datasets, error values reported are followed by values in parenthesis normalized by the number of data points considered. The minimum error obtained was 145.9 (0.7332) for values of \( R_1 = 1.501 \) and \( C_2 = 1.077 \times 10^{-8} \).

Figure 4-1: Sensitivity analysis for circuit model 1. All ten surfaces for each value of \( \lambda \) are plotted on top of each other, revealing little change for all by the lowest values of \( C_2 \).

Thus, sensitivity analysis was conducted again but shifted such that the range of starting values of \( C_2 \) was \( 10^{-12} \) to \( 10^{-2} \). The result of this second analysis was a slightly improved error of 144.5 (0.7261) obtained at values of \( R_1 = 4.907 \times 10^{-12} \) and \( C_2 = 2.648 \times 10^{-8} \). While the order of magnitude of \( R_1 \) seems to have relatively little impact on the ultimate error, the values of \( C_2 \) in both cases are of order \( 10^{-8} \). These values were obtained with a starting value for \( C_2 \) of \( 10^{-4} \). Figure 4-2 demonstrates relatively wide ranges of possible starting values for both \( R_2 \) and \( \lambda \) which provide low error values. Furthermore, Figure 4-3 demonstrates the disappearance of any trend indicating lower errors toward any particular range of \( R_2 \) or \( C_2 \). The optimization was next conducted for both the resistance and impedance data and yielded a total error of 299.0 (0.7512), note that due to the fact that twice as many data points as were fit for the previous analysis were being fit here, this error is equivalent to half its own value for comparison to the resistance curve fits, making the normalized error comparable. Furthermore, while this error was obtained with a value of \( R_1 = 4.279 \times 10^{-2} \), the capacitance was still very similar to the previous analysis at \( C_2 = 2.460 \times 10^{-8} \).
At $C_2 = 10^{-4}$ there is relatively little variation in the lowest values of error with respect to $R_2$ and $\lambda$.

Model 2 was investigated after model 1 using the same initial range of values for $R_1$, $C_1$, and $C_2$ as for $R_1$ and $C_2$ in model 1, but the model yielded higher errors, with a minimum of 179.7 (0.9030) for the resistance data fit. As Figures 4-4 and 4-5 show, the error was relatively independent of starting values for $\lambda$ or $C_1$, but tended to be lower toward lower starting values of $R_1$ and $C_2$. A second round of sensitivity analysis on model 2 was conducted by shifting the ranges of $R_1$ and $C_2$ both to $10^{-9}$ to $10^9$. This new range yielded a slightly lower error at 175.8 (0.8833) for values of $R_1 = 1.417 \times 10^{-12}$, $C_1 = 10^{-9}$, and $C_2 = 47.451$. Figure 4-6 shows that the trend for where the lowest error values lie is relatively unchanged by the new sensitivity analysis. Interestingly, $C_1$, the added capacitance in
series in front of the bulk rock part of the circuit tended toward the lowest values of the given range.

Figure 4-4: Sensitivity analysis for circuit model 2. All ten surfaces for each value of $R_1$ and $C_2$ are plotted on top of each other, revealing little change with respect to $\lambda$ or $C_1$ for all by the lowest values of $\lambda$, and even then lowest value planes are relatively irrespective of $\lambda$.

Figure 4-5: Sensitivity analysis for circuit model 2. All ten surfaces for each value of $\lambda$ and $C_1$ are plotted on top of each other, again revealing little change with respect to $\lambda$ or $C_1$. Furthermore, values for the error fairly erratic except toward the lowest values of $C_2$, and $R_1$. 

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Figure 4-6: Second sensitivity analysis for circuit model 2. Error values at the lower values of $R_1$ and $C_2$ are relatively lower but still erratic and give little sign that investigation of even lower starting values will yield lower errors.

Given the erratic behavior of the models with respect to starting points for the various parameters as well as the difficulty that snopt had in conducting sensitivity analysis on models more complex than model 2 without encountering an error and aborting analysis, the constraints of the model were revisited. Given the resistivity of brine and the dimensions of the rocks under consideration, it was deemed reasonable to set a lower bound for any circuit $R$ terms to 1 Ω. Furthermore, all good fits for $\lambda$ were of order 1 and it is unusual for a physical relationship to have power terms of high order, so $\lambda$ was constrained to lie between 0.5 and 3. While these tightened constraints did not lead to substantially less erratic sensitivity—see Figure 4-7—the problem became more tractable for AMPL and the snopt solver, resulting in slightly better fits and greater stability in the AMPL run script for sensitivity analysis. This added stability allowed for assessment of more complex models. For model 1, this tightened analysis resulted in an error of 321.3 (0.8073) at values of $R_1 = 1.2739$ and $C_2 = 2.4421 \times 10^{-8}$. Notably, the capacitance remained on the order of $10^{-8}$.

Under the more constrained parameters snopt was not able to provide an acceptable match to the data using model 2. Given that model 2 is simply model 1 with the addition of a capacitor, the fact that model 2 did not at least provide results similar to those from model 1 reveals a flaw in the assumption that allowing parameters like the additional capacitance in model 2 to vary unrestrained would make them reach values such that a sufficiently high or low value could be read as that element being turned off. Furthermore, after running models 3, 4, and 5 under the more constrained and stable versions, no matches significantly better than the simpler model 1 were found. Thus, in the interest of elegance model 1 was chosen for analysis with data from the other experimental runs.
The best fit for model 1 obtained by the initial match with only the resistance data is plotted over the data in Figures 4-8 and 4-9, while the best fit for model 1 obtained by matching both the resistance and the impedance curves under the tightened constraints is plotted with the data in Figures 4-10 and 4-11. While the initial fit seems to lie closer to the data especially at lower saturations, the more constrained fit captures more of the complexity in the data's behavior. Primarily, the horizontal lines in Figure 4-8 at the lowest frequency makes the model resistances and impedances qualitatively too similar. None of the matches were able to fit the lowest frequency impedance data very well.

Figure 4-7: Tightened sensitivity analysis for circuit model 1. All ten surfaces for each value of $\lambda$ are plotted on top of each other, revealing a model which is still very ill-behaved.

Figure 4-8: Resistance match for the best fit for model 1 to the data by matching only resistance data.
Figure 4-9: Impedance match for the best fit for model 1 to the data by matching only resistance data.

Figure 4-10: Resistance match for the best fit for model 1 to the data by matching both the resistance and impedance data under the more tightly constrained analysis.
Once model 1 was chosen as the most appropriate model to describe the phenomena observed in the unsputtered core #5 data, the model was then applied to the subsequent experimental runs with core #5. The model provides a qualitatively excellent match to the data from Figures 2-30 and 2-31 with an error of 346.6 (0.5467) at values of $R_1 = 1.0000$ and $C_2 = -6.5741 \times 10^{-19}$. Most notably, the extremely small value of $C_2$ indicates that the capacitance is effectively zero, confirming conclusions made after first viewing the data given the better connection of the electrode to the rock with the sputtered platinum. The model is shown alongside the data in Figures 4-12 and 4-13. The slight deviation from the curve is due to the minimum value allowed for $R_I$ being too high; by allowing $R_I$ to go to zero the nearly perfect match shown in Figure 4-14 can be obtained. This result indicates that while the minimum bound on $R_2$ is justified by the maximum conductivity of the brine being on the order of magnitude of $10^5$, perhaps the resistance of the equipment and associated experimental apparatus, represented by $R_I$, can be negligible. Thus, sensitivity analysis for the data from subsequent runs was also adjusted to allow $R_I$ to approach zero.

Figure 4-11: Impedance match for the best fit for model 1 to the data by matching both the resistance and impedance data under the more tightly constrained analysis.
Figure 4-12: Resistance match to the uncut, sputtered core for the best fit using model 1 by matching both the resistance and impedance data under the more tightly constrained analysis.

Figure 4-13: Impedance match to the uncut, sputtered core for the best fit using model 1 by matching both the resistance and impedance data under the more tightly constrained analysis.
Figure 4-14: Resistance match to the uncut, sputtered core for the best fit using model 1 under the more tightly constrained analysis but allowing $R_j$ to be zero.

Model 1 was next fit to the first experimental run of core #5 with one simulated fracture using the data with the corrected mass measurements, and a minimum error of 953.8 (1.355) was obtained at $R_j = 0$, $C_2 = 1.899 \times 10^{-9}$. As expected the capacitance rose to a significant value with the addition of the fracture. The fit for these data to model 1, shown in Figures 4-15 and 4-16, are not quite as good as the previously found fits; however, this experiment had particularly egregious mass error due to wire relaxation, and the model nonetheless fits the qualitative trends in the data well.

Figure 4-15: Impedance match for model 1 to the sputtered core with 1 cut.
Figure 4-16: Resistance match for model 1 to the sputtered core with 1 cut.
The fit of model 1 to the second experimental run on core #5 with one cut is shown in Figures 4-17 and 4-18. The error for these curves is 398.7 (0.7222) obtained at $R_i = 0$ and $C_2 = 1.526 \times 10^{-08}$.

Figure 4-17: Impedance match for model 1 to the sputtered core with 1 cut, second run data.
The match for model 1 to the data from the rock with two cuts is shown in Figures 4-19 and 4-20. The error for these curves is 398.7 (0.7355) obtained at $R_1 = 0$ and $C_2 = 9.338 \times 10^{-10}$. The capacitance for this fit is surprisingly less than the capacitance for previous matches.
Figure 4-20: Resistance match for model 1 to data from the sputtered core with 2 cuts. Model 1 was fit to the data from the second experiment on the rock with two cuts and is shown in Figures 4-21 and 4-22. The error for these curves is 668.8 (0.8574) obtained at $R_1 = 2.755 \times 10^{-8}$ and $C_2 = 5.096 \times 10^{-9}$. While greater than the previous run, the value for the capacitance for this fit is still less than the capacitance for the matches to the data from the rock when it was cut only once. Also of interest is the fact that $R_1$ is not 0 in this instance. However, changing $R_1$ to 0 produced no graphically noticeable difference in the curve fit.

Figure 4-21: Impedance match for model 1 to data from the second run on the sputtered core with 2 cuts.
4-22: Resistance match for model 1 to data from the second run on the sputtered core with 2 cuts.
Chapter 5

5. Discussion

5.1. Conclusions

The behavioral change of the electrical properties for core #5 before and after sputtering as well as the transition in behavior again after cutting the rock support the conclusion that fractures in rock provide a capacitance which results in different values of impedance and resistance depending upon the frequency at which a rock is measured at very low saturations. When the initial copper electrodes based on those used by Sandler et al. (2009) were used, all rock cores demonstrated frequency-dependent behavior at very low saturations. The imperfect connection afforded by this electrode setup likely facilitated air gaps between the sample and the electrodes, resulting in a capacitance which led to the frequency dependence. This frequency dependence was no longer manifest in core #5 after the core had been sputtered with platinum. This shift is likely due to the improved connection between the electrodes and the sample, with the sputtered metal eliminating air gaps between the ionically conducting rock and the electronically conducting platinum and stainless steel. Any air gaps that might exist between the stainless steel mesh and the platinum are likely not as significant as in the nonsputtered case because of the electronic nature of the conduction in both of these materials. Upon cutting the rock, a new capacitor was introduced to the system, allowing air gaps to form between either side of the fracture after a sufficient amount of water had evaporated. Microlayers of water adsorbed to the surfaces of the fractures provide surfaces for charge collection as on a parallel plate capacitor.

Given these changes in behavior, it was expected that measurements after the rock had been cut a second time would yield an even larger capacitance than what was observed for the rock with one cut. This rise in capacitance was expected much in the same way the addition of two capacitors in series doubles the effective capacitance. However, the resulting data were not remarkably different from the data collected when the rock had one cut, and the model fit to the data yielded confusingly low values of capacitance. It was suspected that perhaps variation in the compression on the rock across the fractures due to the experimental setup might have altered the apertures of the fractures sufficiently to influence the capacitances of those fractures. The data presented in figure 2-47 indicate that there is indeed a noticeable change in the measured impedance and resistance of the rock depending on how tightly the clamps are secured, but the difference in value appears negligible in comparison to the variation in values of the data collected over an entire experimental run. Furthermore, it was suspected that greater compression would influence capacitance by reducing the fracture aperture, thereby lowering the capacitance, and a lower capacitance would be manifested in higher values for both impedance and resistance. However, the impedance demonstrates the opposite relation—decreasing monotonically with tightness—and the resistance increases before decreasing at the
The most likely explanation for the deviation between the results obtained by Sandler et al. (2009) and the results obtained here is that Sandler et al. simply might not have taken measurements at low enough saturations to observe the frequency dependence in the unfractured Berea sandstone they used. There is only one data point at large time from which to infer the plateau in water evaporation. It is possible that the curve measured by Sandler et al. is merely the beginning portion of the curve measured for the same type of rock in this research. However, this one data point at very low saturation should be at sufficiently low saturation such that at least the last point in the resistivity data presented by Sandler et al. should exhibit the frequency dispersion recorded in this research. It is not known whether this last point of datum was plotted on the Sandler et al. resistivity index figures. Furthermore, the shapes of the curves are qualitatively different for some of the rocks measured here, with the data from Sandler et al. nearly linear on the log-log scale and some data here having a more curved, swinging shape. One possible explanation for the difference in curve shape could lie in the fact that a consistent problem with the experiment setup used here as well as in the research from Sadler et al. was the issue of wire relaxation and its effect on mass measurements and saturation determination. While it is possible that different methods of calibration for wire relaxation might affect the shape of the curves, these differences should not impact the qualitative differences of the frequency dispersion.

An alternative explanation offered by the model circuit developed above is that the specific sandstones used by Sandler et al. (2009) had much lower internal capacitances than those used in this research. As shown in Equation 1-13, frequency and capacitance are linked parameters; a change in capacitance will be manifested the exact same way as an identical change in frequency. Thus, a much lower capacitance would give equivalent results to the case of measuring at frequencies too low to observe the frequency dependence in $R$ for the range of $R_2$. This behavior can be conceptualized as equivalent to shifting the plot in Figure 3-3 diagonally in the positive $x$, $y$ and $z$ directions, and were it possible for the rocks to achieve even lower values of $R_2$, a frequency dependence would have eventually been observed. Had Sandler et al. observed at higher frequencies, similar frequency dependence may have been observed for all rocks. One concern with this explanation is that the Berea sandstone samples used by Sandler et al. and in this research were from the same source, and it seems unusual that properties should vary so greatly from sample to sample of the same rock type. However, a critical difference between the samples, discovered only after the experiments with the cores #1 and #2 were conducted, is the history of the samples. Namely, the Berea sandstone used here was fired, affecting the clay content of the samples. As elucidated in the literature (Drury, 1978; Knight and Nur, 1987; Roberts and Lin, 1997; Bekhit and Khalil, 2007; Garg et al., 2007), clay content can have a significant effect on the electrical properties of rocks, influencing surface polarizations and internal capacitances.
5.2. Future Work

The greatest impediment to collecting reliable data has been the issue of wire relaxation, uncertain mass measurements, and subsequently uncertain saturation of the rock for each measurement of electrical properties. The ideal experimental setup would have all of the equipment and wiring on top of the mass balance with perhaps only a couple power cords and computer connections leading off the balance. Because these few cords and cables would not need to be adjusted over the entire course of experimentation they could be allowed to relax into position well before the first experiment is conducted. Furthermore, the hand clamps should be replaced by clamps which would allow precise determination of the compressive force on the rock for each experiment, allowing for greater consistency in measurements. Spacers could also be used to prop the fractures open to more reliably measurable apertures than what is provided by simply allowing the rock grains to prop the fracture open.
Nomenclature

\[ E = \text{electric field intensity (volt/meter)} \]
\[ H = \text{magnetic field intensity (ampere-turn/meter)} \]
\[ D = \text{electric displacement (coulomb/m}^2\text{)} \]
\[ J = \text{electric current density (ampere/m}^2\text{)} \]
\[ B = \text{magnetic induction (weber/m}^2\text{)} \]
\[ \varepsilon = \text{dielectric permittivity (farad/m)} \]
\[ \sigma = \text{electric conductivity (mho/m or siemens/m)} \]
\[ \rho = \text{electric resistivity (ohm-m)} \]
\[ \mu = \text{magnetic permeability (henry/m)} \]
\[ i = \text{the imaginary unit, } \sqrt{-1} \]
\[ \omega = \text{frequency (radians/sec)} \]
\[ t = \text{time (sec)} \]
\[ DF = \text{dissipation factor} \]
\[ Q = \text{quality factor} \]
\[ \delta = \text{angle by which storage is out of phase in the complex plane} \]
\[ R = \text{resistance (ohm)} \]
\[ X = \text{reactance (ohm)} \]
\[ Z = \text{impedance (ohm)} \]
\[ R_s = \text{resistance of a series circuit (ohm)} \]
\[ X_s = \text{reactance of a series circuit (ohm)} \]
\[ \text{RC circuit} = \text{a circuit composed of resistors and capacitors} \]
\[ \text{RL circuit} = \text{a circuit composed of resistors and inductors} \]
\[ C = \text{capacitance} \]
\[ L = \text{inductance} \]
\[ I = \text{resistivity index} \]
\[ S_w = \text{water saturation} \]
References

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Appendix A

A. LabView vi Modification

The QuadTech Model 1715 RCL meter came with its own suite of LabView vi programs that could be edited or assembled in different ways to access the various functions of the RCL meter remotely via RS-232 port. The basic example vi which to a great degree replicates the front controls of the physical RCL meter was used in conjunction with loop structures to appropriately cycle through measurements of all four frequencies every ten minutes. The bottom left of Figure A-1 contains a structure that cycles through four iterations and connects to the appropriate frequency controls indicated in Figure A-2 by the orange lines leading into part 0 of the scrolling frame structure which contains modifications for the adjustable parameters of the RCL meter. The bottom of Figure A-2 also shows the 10-minute timed loop (in units of milliseconds) which encloses the entire vi structure. A string concatenating element and file-writing elements were added to part 1 of the scrolling frame, shown in Figure A-3, in order to output the measured impedance and resistance data to a text file. Appropriate labels and time stamps were included in tab-delimited format such that they could easily be interpreted by programs such as Microsoft Excel, which was used extensively in data cleanup. For the sake of completeness the rest of the vi, which is contained in part 2 of the scrollable frame structure, is shown in Figure A-4.
Figure A-1: LabView block diagram frequency cycling.
Figure A-2: LabView block diagram 10-minute loop and part 0 of the scrolling frame.
Figure A-3: LabView block diagram, part 1 of the scrolling frame.
Figure A-4: LabView block diagram, part 1 of the scrolling frame.
Appendix B

B. Ion Diffusion Calculation

When saturating, drying, and resaturating rock cores over many cycles it is likely that ions will accumulate over time and possible that this accumulation will affect the resistances measured. Sodium and chloride ions from the brine precipitate out of solution as the water evaporates and are adsorbed to the internal surfaces of the rock. However, these ions are weakly bound to the rock relative to their attraction to the water upon resaturation, due in large part to the polar nature of water molecules. An initial simplifying assumption was that over the course of saturation the water in the rock comes to ionic concentration equilibrium with the water in the saturation chamber. However, this assumption is not trivial and the diffusion of these ions was investigated in greater detail. Diffusion coefficients for sodium and chlorine ions in solution in water are provided in *Diffusion: Mass Transfer in Fluid Systems* (Cussler, 2009), calculated from the data of Robinson and Stokes (2002). The diffusivity of Na$^+$ is $1.33 \times 10^{-5}$ cm$^2$/s and of Cl$^-$ is $2.03 \times 10^{-5}$ cm$^2$/s. As a first approximation the root mean square motion of the particle under diffusion gives the distance any given particle is likely to travel after a certain period of time. This distance is given by

$$x = \sqrt{2nDt}$$  \hspace{1cm} (B-1)

where $x$ is the distance travelled, $n$ is the dimensionality of the space under consideration, $D$ is the diffusivity, and $t$ is the time the particle spends travelling. Over the typical period of saturation of about 12 hours, the sodium and chlorine ions would travel approximately 1.86 cm and 2.29 cm, respectively. Although these distances are enough that most ions within in the pore space of the rocks would be able to make it outside the rock and facilitate equilibrium with the surrounding solution, the distances aren’t quite enough for ions at the center of the sandstone cube to leave the cube. Thus, a more detailed analysis of the diffusion equation was conducted. Given Fick’s first law:

$$J = -D \nabla \varphi$$  \hspace{1cm} (B-2)

where $J$ is flux and $\varphi$ is the concentration of ions, and the continuity equation:

$$\frac{d}{dt} \varphi + \nabla \cdot J = 0$$  \hspace{1cm} (B-3)

combining equations yields the diffusion equation in multiple dimensions:
\[
\frac{d}{dt} \varphi = \nabla \cdot (D \nabla \varphi) \quad \text{(B-4)}
\]

which simplifies to:

\[
\frac{d}{dt} \varphi = D \nabla^2 \varphi \quad \text{(B-5)}
\]
due to \( D \) being a constant in all directions. The analytical solution to this partial differential equation can be elegantly solved by multiplying the appropriate Green's function by the initial condition and integrating over the space of the problem. This form of solution is especially useful given the nonsmooth initial condition assumed. Given the relative affinity of the ions for the imbibed water, the initial condition was assumed to be that of a cube of 2% concentration surrounded by a larger cube of 1% concentration. The interior cube represents the ions from the imbibed water in addition to the dissolved ions which were previously adsorbed to the pore space. The exterior cube is a simplification of the saturation environment. Thus, the concentration as a function of time can be expressed as:

\[
\varphi(\vec{x}, t) = \int G(\vec{x}, \vec{x}', t) \varphi(\vec{x}', 0) d\vec{x}' \quad \text{(B-6)}
\]

where \( \vec{x} \) is a vector of all spatial coordinates, \( \vec{x}' \) is the Green's function impulse location, and \( G \) is the Green's function, which in Cartesian coordinates can be expressed as:

\[
G(\vec{x}, \vec{x}', t) = \frac{e^{-|\vec{x} - \vec{x}'|^2}}{(4\pi Dt)^{n/2}} \quad \text{(B-7)}
\]

where \( n \) is again the dimensionality of the space. The model used for this analysis was scaled such that the rock cube was represented by a cube 200 units on each side and centered at the origin with the diffusivities scaled accordingly. Furthermore, the saturation chamber was represented as a cube 1000 units on each side also centered at the origin. While this setup ignores the fact that the rock cube is actually resting on the floor of the saturation vessel, the answer does not likely deviate far from this assumption and the symmetry provides a valuable simplification. The concentration is expressed in units of percent, with 1 being equal to 1%. Thus, the initial condition can be expressed as:

\[
\varphi(\vec{x}', 0) = (H(x - 100) - H(x + 100)) \times \\
(H(y - 100) - H(y + 100)) \times \\
(H(z - 100) - H(z + 100)) + 1 \quad \text{(B-8)}
\]

where \( H \) is the Heaviside step function and \( x, y, \) and \( z \) are the spatial coordinates in three-dimensional space. The boundary condition is assumed to be that of a container wall which allows zero flux at the boundary. Thus, superposition of Green's functions can be
used to represent a perfect reflecting boundary. The full form of the equation becomes cumbersome in multiple dimensions so it is displayed in Equation B-9 in its one-dimensional form which can readily be generalized to multiple dimensions:

\[
G_N(x, x', y, y', z, z', t) = G(x, x', y, y', z, z', t) + G(x, x' + 1000, y, y', z, z', t) + G(-x, -x' + 1000, y, y', z, z', t)
\] (B-9)

where \(G_N\) is the Green's function for the Neumann boundary conditions in this problem. The result for sodium ions after 12 hours is shown in Figures B-2 and B-3, with Figure B-1 showing the initial concentration distribution as a reference. These figures illustrate the fact there is indeed a certain amount of ion accumulation from run to run. Furthermore, these models do not take into account the tortuosity of the paths which the ions must follow to leave the pore space and equilibrate with the brine bath. However, as described by Ucok et al. (1980) and summarized by Olhoeft (1981), resistivity varies by approximately only 0.1 \(\Omega\cdot m\) over the range of concentrations which might vary due to ionic accumulation. Furthermore, the relationship between resistivity and NaCl ion concentration decreases nearly exponentially at greater concentrations. The resistances measured for the cores, however, are on the order of \(10^3\) \(\Omega\) to \(10^6\) \(\Omega\). Hence, accumulation of ions between experimentally runs was determined to be inconsequential to the investigations of this research.

Figure B-1: Initial distribution of Na\(^+\) ion concentration at \(z = 0\).
Figure B-2: Distribution of Na\(^+\) ion concentration at \(z = 0\) after 12 hours of diffusion.

Figure B-3: Distribution of Na\(^+\) ion concentration at \(z = 100\) after 12 hours of diffusion.