# Electrical Properties Estimation of Oil Sands

By

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# Abstract

With conventional sources of oil depleting, non-conventional oil resources such as heavy crude oil, oil shales, and oil sands are gaining importance as alternatives. Various extraction processes have been employed to separate bitumen from oil sands. The relatively unexplored electromagnetic heating of oil sands can be a major breakthrough for extraction of bitumen from oil sands. The objective of this work is to study the electrical properties of Athabasca oil sands with variations in frequency and temperature. The electric properties of a material are described by the impedance parameters and complex permittivity. To obtain the electrical properties, a set of experiments were performed for frequencies ranging from 100 Hz to 1 MHz with temperatures varying between $20^0$C - $250^0$C. Electrical properties such as dielectric constant and dissipation factor were derived from the experimental results obtained using a parallel plate capacitor with an impedance analyzer. Temperature dependence results were also measured at different frequencies. Characterization and estimation of different properties of oil sands will turn out to be useful in experimental as well as modelling related studies of electromagnetic heating.

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# List of Symbols

| | |
|---|---|
| $\varepsilon_r$ | Complex Permittivity [F/m] |
| $\varepsilon_r'$ | Dielectric Constant |
| $\varepsilon_r''$ | Dielectric Loss |
| A | Cross sectional area of the electrode [$m^2$] |
| Cp | Measured Capacitance [F] |
| $Cp_{o/s}$ | Specific heat capacity of oil sands [J/deg.g] |
| D | Dissipation factor |
| d | Diameter of electrode [m] |
| I | Current [A] |
| R | Resistance [ohm] |
| t | Thickness of the sample [m] |
| $tan(\delta)$ | Loss tangent/ Dissipation Factor |
| Y | Admittance [S] |
| Z | Impedance [ohm] |
| θ | Phase Angle [degree] |
| μ | Viscosity [Pa.s] |
| ρ | Density [$kg/m^3$] |
| ω | Angular frequency [Hz] |
| $\sigma$ | Electrical Conductivity [S/m] |

# List of Abbreviations

| | |
|---|---|
| CSS | Cyclic Steam Simulation |
| DSC | Differential Scanning Calorimetry |
| FTIR | Fourier Transform Infrared Spectroscopy |
| GPIB | General Purpose Interface Bus |
| IOSI | Institute for Oil Sands Innovation |
| RAT | Resonant Auto-Transformer |
| SAGD | Steam Assisted Gravity Drainage |
| SCO | Synthetic Crude Oil |
| TGA | Thermogravimetric Analysis |

# Chapter 1
# Introduction

## 1.1 Energy

The oil and gas sector has witnessed a tremendous growth and expansion worldwide in the last few decades with ever increasing demand for energy (BP Energy 2013). Due to growing energy needs, highly fluctuating oil prices and increasing importance of renewable energy sources, large amounts of money is being invested in the research and development area to understand the science behind oil production and develop safe techniques to produce oil in an environmentally friendly manner (Alberta Government 2011, Alberta Government Sept 2013). Issues like the volatility of the crude oil prices, switching from light to heavy crude oils, meeting stringent product specifications and sustainable development pose very challenging and technologically demanding problems before the petroleum industry. Currently, increasing attention is being given to the characterization of heavy oil and bitumen to maximize the oil recovery from a hydrocarbon reservoir. As the conventional sources of oil are getting depleted, non-conventional oil resources such as heavy crude oil, oil shales and oil sands are gaining in importance as alternatives.

Canada's oil sands deposits have proven to be an immensely important source for providing a stable and reliable energy supply to Canada and the United States. Canada has the largest oil sands deposits in the world with reserves of 173 billion barrels, while 170 billion barrels are located in Alberta itself. Out of all the reserves, 168 billion barrels are recoverable with today's technology (Alberta Energy, 2014), (Canada National Energy Board 2004). In terms of proven crude oil reserves, Canada ranks third after Saudi Arabia and Venezuela (Alberta Government 2011) . Oil sands deposits are mainly located in three oil sands areas (OSAs): Athabasca Wabiskaw-McMurray, Cold Lake Clearwater and Peace River. Athabasca Wabiskaw-McMurray, also known as Athabasca oil sands, is the largest reserves in Alberta (Masliyah, Czarnecki et al. 2010).

Oil sands are a mixture of sand, water, minerals and highly viscous, heavy organic material known as bitumen. Bitumen can be characterized as a mixture of heavy hydrocarbons with very high densities and viscosities. Bitumen percentage is variable in the oil sands typically from 0% to 18% by weight. Extracted bitumen is upgraded and refined to get synthetic crude oil (SCO) and other final products (Bearden, Aldrige 1979).

Oil sands are extracted either by open pit mining or by in-situ extraction (recovery of bitumen while the sand remains in the deposits) depending upon the depth of oil sands deposits. The deposits at depths more than 80 meters are not accessible for open pit mining. In situ recovery techniques are used to recover the bitumen from the oil sand reserves. Bitumen is highly viscous at normal temperatures; but the viscosity decreases rapidly when the temperature is increased from $20^0$C - $250^0$C. As bitumen in oil sands is highly viscous at normal temperatures, it is hard to separate bitumen from oil sands. Incorporation of thermal recovery methods in these deposits is highly essential to reduce the viscosity and to extract bitumen from oil sands deposits. Two commercial in-situ recovery processes, steam assisted gravity drainage (SAGD) and cyclic steam simulation (CSS) are used in the industry to extract bitumen from deep reservoirs (Butler 1991).

SAGD and CSS both work on a similar principle. In SAGD, two parallel horizontal well pairs run through the oil sands reservoir. High pressure and high temperature steam is injected in the reservoir by the injector well that is placed parallel and above the producer well. As a result of this steam injection, the temperature of the reservoir increases, thereby greatly reducing the viscosity of the oil sands. Then the producer well is opened to start the oil production. The recovery in SAGD process is observed to be higher than the CSS process. The recovery can still be improved by using other methods. Research is ongoing to optimize the recovery of bitumen by coupling existing methods with solvent assisted recovery, electrical heating, and in situ combustion (Alberta Government 2013).

Electromagnetic heating can be an effective alternative method in cases where in situ extraction techniques by steam injection are not efficient because of heat losses to the reservoir, low permeability, and low thermal conductivity (Sahni, Kumar et al. 2000, Marx, Langenheim 1959). Depending on the frequency, electromagnetic heating can be divided as low frequency heating with frequency less than 60 Hz and high frequency heating with frequency within the range of a few kHz to GHz (radio wave and microwave frequencies). Low frequency heating is also known as resistive or Joule heating where power dissipation takes place due to resistive heating in the deposits. In the high frequency range, dielectric-heating phenomenon prevails. When an electric field is applied across the dielectric media, polar molecules in the oil sands tend to align themselves with the changing electric field. This phenomenon is called dipolar rotation or polarization. As the field oscillates, polar species also oscillate which leads to the losses. In dielectric heating, the heat is produced due to dielectric losses occurring in the medium.

Electromagnetic heating process has advantages like instantaneous heating of oil sands and its independence from the low thermal conductivity of the reservoir. Although thorough investigation has been prevalent for a long time to quantify the efficiency of electromagnetic heating (Davison 1991, Kasevich, Price et al. 1994), the process has not yet developed to a commercial scale. Relatively unexplored electromagnetic heating of oil sands can be a major breakthrough for oil sands extraction. Estimation of dielectric properties assists in experimental as well as in modelling studies to make this technique commercially applicable.

## 1.2 Studies on dielectric properties

Despite electrical heating being one of the common fields of interest, very little research has been done to study the dielectric properties of oil sands. Initial studies for finding out dielectric properties of Athabasca oil sands were carried out by (Chute, Vermeulen et al. 1979). In this experimental study, the electrical conductivity and relative dielectric constant of oil sands were determined over a frequency range of 50 Hz to 1 GHz. The experiments were carried out on different grades of Athabasca oil sands which had variable moisture and bitumen contents. These properties are essential to carry out the feasibility of electrical heating of oil sands and its advantages at different frequencies and temperatures.

## 1.3 Motivation

The research done by (Chute, Vermeulen et al. 1979), for finding out electric properties in the frequency range of 50 Hz to 1 GHz motivated us to further investigate the dielectric properties.

An electrical system was built in the Nano-Interfaces & Molecular Engineering (NIME) group at the Department of Chemical and Materials Engineering, University of Alberta which has the ability to produce very high voltages. The system is also known as Resonant Auto-Transformer (RAT), and can produce high potential fields with low current consumption. The voltage produced is in the order of $10^6$ volts while the resultant frequency is around 200 kHz. Resonant

Auto-Transformer can be used to heat Athabasca oil sands electromagnetically. Experiments were conducted to demonstrate the application of Resonant Auto-Transformer in heating of oil sands. An increase in temperature was observed during the experiments. At such high frequency and high voltage electromagnetic heating will be dominant and is dependent on the electrical properties of oil sands. Thus to understand more about the mechanisms involved in electromagnetic heating, it is necessary to study the electrical properties of oil sands.

The objective of this work is to estimate the electric properties of Athabasca oil sands with variation in frequency as well as temperature. Electric properties essential for evaluation of electromagnetic heating such as dielectric constant, dissipation factor and electrical conductivities were obtained. As Resonant Auto-Transformer is operated around the frequency of 200 kHz, it was necessary to gather electrical properties data near this frequency regime. Thus, experiments were performed over a frequency range of 100 Hz to 1 MHz with temperatures varying between $20^0$C - $250^0$C. Capacitance and dissipation factor were measured using a parallel plate capacitor with an impedance analyzer. The dielectric constant was evaluated from measured capacitance. Temperature dependence results were measured at different frequencies. Empirical relations for estimation of the dielectric properties can be derived from these results. The results obtained from the analysis will prove to be useful in understanding how the electromagnetic heating system behaves for oil sands.

## 1.4 Thesis Outline

The thesis is divided into several chapters, covering the conceptual design for measurement of electric properties.

Chapter One: This chapter includes a short introduction on oil sands, brief information of existing technologies for oil sands extraction, objective and outline of the thesis. The background of electromagnetic heating is discussed. It is correlated with the study of dielectric and the previous studies on the topic are reviewed further.

Chapter Two: This chapter comprises the review of electromagnetic heating, its advantages and limitations. A detailed description of the fundamentals behind the dielectric properties and their dependence on parameters like temperature and frequency is given.

Chapter Three: This chapter includes the literature review of oil sands properties. This chapter is dedicated to the characterization of oil sands. The important characterization techniques are also discussed in the chapter.

Chapter Four: This chapter presents the experimental work involved in the study. The description about the experimental setup, sample preparation, calibration and the working of associated software has been included in this chapter.

Chapter Five: This chapter includes the results and discussions of the characterization of oil sands and measurements of dielectric properties obtained for various grades of oil sands based on temperature and frequency dependence.

Chapter Six: The final chapter gives the summary of the work done so far in this study. The future scope of the work is also discussed in this chapter.

# Chapter 2
# Electromagnetic Heating and Dielectric Property Estimation

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As mentioned in chapter 1, steam injection *in-situ* processes such as steam assisted gravity drainage (SAGD) and cyclic steam simulation (CSS) are used in the industry to extract bitumen oil sands deposits. Steam injection is an effective method for recovery of bitumen; however, there are a few situations where it might not be as effective. The possible situations could be one of the following as discussed by (Sahni, Kumar et al. 2000).

- Very deep formations, where steam reaching the reservoir is low in quality.
- Thin pay-zones, where significant amount of heat is lost to adjacent formations.
- Low permeability formations, where injected steam cannot easily penetrate through the formation.
- Reservoir heterogeneity, which might lead to fracture in the rocks and thus reduce recovery.

Electromagnetic heating can prove an effective alternative method for heating oil sands reservoirs in the cases where steam injection techniques are not efficient. A brief analysis of the existing literature related to electromagnetic heating of oil sands is presented in the following section. Depending on the frequency, electromagnetic heating can be divided as low frequency heating (with frequency ranging from 0.1 Hz to 60 Hz) and high frequency heating (with frequency ranging from few kHz to GHz).

## 2.1 Electromagnetic Heating

### 2.1.1 Joule Heating

Electrodes are inserted into the reservoirs wells and alternating current is passed through the formation (Vermeulen, Chute 1983). This low frequency heating (with frequency ranging from 0.1 to 60 Hz) is also known as Joule heating. As the current flows through the formation, power is lost in the formation due to resistive heating and the oil sands formation heats up (Mcgee, Vermeulen 2007, McGee 2008). The water and free ions in the solution provide a conductive passage for current while oil sands act as the resistive element. Heating depends upon resistivity and thus, on electrical conductivity through oil sands formation. Heating in the formation can be explained with the simple formula

$$P = I^2 R \tag{2.1}$$

where I is the current flowing and R is effective resistance offered by oil sands formation. Power dissipated is dependent on the current as well as the resistance offered by oil sands matrix. One of the problems associated with Joule heating is the possibility of a circuit break, which may occur if the temperature of water close to the electrodes reaches its boiling point. The formation of water vapour reduces the heating efficiency of the electrodes. This problem is overcome by the continuous supply of water in heat prone areas near the electrodes. After sufficient heat is introduced in the reservoir, the viscosity of bitumen is reduced and it can be extracted with the help of external agents like solvent or steam.

### 2.1.2 Inductive Heating

Inductive heating can be described as heating of electrically conductive metal by electromagnetic induction caused by the coil, which leads to the generation of eddy currents leading to resistive heating in the metal object (Vermeulen, Chute 1983). As alternating current passes through the coil, it generates heat in the region inside the coil. A comparative study of resistive heating and inductive heating has been conducted by (Vermeulen, McGee 2000).

### 2.1.3 High frequency heating: Dielectric Heating

High frequency heating includes microwave and radio wave frequency in the range of few kHz to GHz. Dielectric heating dominates in this frequency range as compared to Joule heating (Vermeulen, Chute 1983). High frequency heating depends on the material properties such as permittivity ($\boldsymbol{\varepsilon_r}$), magnetic permeability (μ), and electrical conductivity ($\boldsymbol{\sigma}$). Maxwell's equation explains the average power dissipated per unit volume given by electromagnetic heating

$$\boldsymbol{P = \omega\varepsilon_0\varepsilon_r\, tan(\delta)\, |E|^2} \tag{2.2}$$

Where P- Power dissipated per unit volume [J/m$^3$.s]

ω – Angular frequency [Hz],

$\varepsilon_0$- Free space dielectric constant [F/m],

$\varepsilon_r$- Relative permittivity,

$tan(\delta)$- Loss tangent,

$|E|$ - Electric field [V/m] and

Conductivity $\sigma = \omega\varepsilon_0\varepsilon_r\, tan(\delta)$

As the oil sands sample is heated by absorbing the electromagnetic energy, the increase in the temperature for the medium can be calculated from the equation

$$\sigma\, E^2 dt = \rho C_P dT \tag{2.3}$$

Where $C_P$= Specific heat capacity of oil sands $\frac{J}{\deg . g}$

$\rho$= density of oil sands medium in kg/m$^3$,

In high frequency electromagnetic heating, the heat is produced due to dielectric losses occurring due to the constantly changing electric field. The heating is dependent on the polar species present in the system and dielectric constant of the material. The principle for dielectric heating is similar to microwave heating.

When a high frequency electric field is applied, free electrons, ions and dipoles of polar molecules in the oil sands tend to align themselves with changing electric field. The rotational motion, vibrational motion and molecular collisions with constantly changing electric field result in heating of the medium. Unlike low frequency heating, this process is independent of electrical conductivity through oil sands formation; it does not require pore water to provide a conductive path. To understand the mechanism of electromagnetic heating it is necessary to discuss the electrical properties of an oil sand system.

## 2.2 Impedance analysis

When a signal $V = V_m \sin(\omega t)$ is applied across the load, $i = I_m \sin(\omega t + \theta)$ is the resultant current. The applied voltage and resultant current differ by the phase angle of θ. If the load applied is purely resistive then the lag between applied voltage and measured current will be zero. If the load applied is purely capacitive or inductive the applied voltage is out of phase with current by the phase angle of 90°. If the system consists of combination of resistor, inductor or capacitor, the effective resistance offered by the system is complex and it is known as electrical impedance (Z). For a sinusoidal voltage or current the complex resistance, also known as impedance (Z), is given by Ohm's law

$$Z = \frac{V}{I} \qquad (2.4)$$

As impedance is a complex quantity, it can be represented by real and imaginary parts as follows

$$Z = Z' + jZ'' \tag{2.5}$$

$$\theta = \tan^{-1}\left[\frac{Z''}{Z'}\right] \tag{2.6}$$

$$Z' = |Z|cos\theta \tag{2.7}$$

$$Z'' = |Z|sin\theta \tag{2.8}$$

where $Z'$, $Z''$ and θ represent the real and imaginary parts of impedance and phase angle respectively.

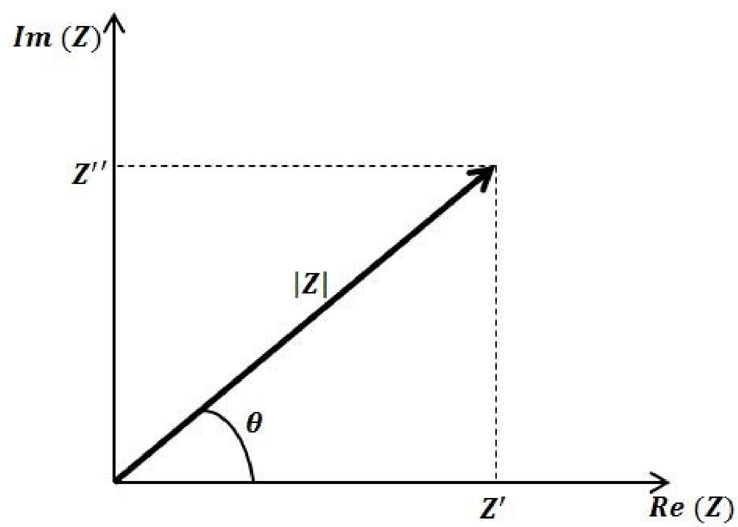



**Figure 2.1: Simple vector diagram for Impedance**

Complex resistance is plotted in a vector diagram (see figure 2.1); real part and imaginary part of impedance are $90^0$ out of phase. Resultant of these vectors forms angle θ with the real part of impedance. Impedance analysis of oil sands is done using an impedance analyzer to extract more information about the electrical behaviour of the sample. Complex permittivity analysis is done to analyze the dielectric response of oil sand sample. Basics of dielectrics and its dependence is discussed in the next section.

## 2.3 Basics of Dielectric

A material can be classified as a dielectric depending upon its ability to interact with the applied external field. The dielectric properties of a material are described by its complex permittivity. Complex permittivity of material gives information as to how a dielectric material reacts, as well as to how a material stores or dissipates electric energy when an external field is applied (Zhang, Huo et al. 2013, Erdogan, Akyel et al. 2011). When an external field is applied across the simple parallel plate incorporated with dielectric material, polarization takes place within the material for different frequencies. Polarization leads to redistribution and neutralization of charges at the electrode surfaces leading to increase in the energy stored in the material. Thus, the capacitance of the system will increase when a dielectric is incorporated in a capacitor (Paraskevas, Vassiliou et al. 2006).

Complex permittivity is defined in terms of dielectric constant $\varepsilon'$and dissipation factor $\varepsilon''$. The permittivity for dielectric substance is given by

$$\varepsilon = \varepsilon' - j\varepsilon'' \tag{2.9}$$

Permittivity of a material is complex number and its value is defined relative to the absolute permittivity of free space.

$$\varepsilon = \varepsilon_r * \varepsilon_0 \tag{2.10}$$

Where $\varepsilon_0$ is the permittivity of free space, 8.854*$10^{-12}$ $F/m$.

Similarly, complex relative permittivity is defined relative to the absolute value of permittivity of free space.

$$\varepsilon_r = \frac{\varepsilon' - j\varepsilon''}{\varepsilon_0} = \varepsilon'_r - j\varepsilon''_r \tag{2.11}$$

Relative complex permittivity represents the interaction of dielectric material when an electric field applied, whose real part $\varepsilon'_r$ represents the storage while its imaginary part $\varepsilon''_r$ represents losses in the system. Real part $\varepsilon'_r$, also known as dielectric constant, provides information about how much energy is stored in the system when an external electric field is applied. Imaginary part $\varepsilon''_r$ of complex relative permittivity, also known as dissipation factor, represents how much energy is dissipated in the presence of external field. $\varepsilon''_r$ represents the dielectric losses due to polarization as well as ionic conduction.

It is evident that the power lost in the material is directly dependent on loss factor $\varepsilon''_r$, while it might as also be dependent on dielectric constant $\varepsilon'_r$ which plays an important role in the electric field in the dielectric medium.

As discussed earlier, power dissipated per unit volume is given by electromagnetic heating in equation (2.2), it can be seen that power dissipation is directly dependent on dissipation factor $\boldsymbol{\varepsilon''}$. Although the dependence of $\boldsymbol{\varepsilon'}$ can not been seen in the equation directly, the heating may be dependent on $\boldsymbol{\varepsilon'}$ as electric field inside the dielectric is a function of $\boldsymbol{\varepsilon''}$ (Nelson 1991).

## 2.4 Variation in dielectric properties

Dielectric properties of the materials depend on various factors. For hygroscopic materials the water content plays a major role. Dielectric properties are dependent on other important factors such as frequency of the external applied electric field and temperature of the material. At the microscopic level the dielectric properties depend upon the structure of the material, shape of the grains present. Dielectric properties are dependent upon the chemical composition and the presence of the ions in the material, which determine the permanent dipoles moments in the system. In this section, we will discuss the dependence of dielectric properties on frequency of the applied external field and the temperature variation in the material.

### 2.4.1 Frequency dependence

Dielectric properties of the materials vary with the change in the frequency of the applied electric field. The frequency dependence of the dielectric properties can be explained by the phenomenon of polarization arising from the orientation of dipoles when an external electric field is applied. The system can be considered to contain permanent dipoles and free ions at different chemical compositions. As the frequency applied is varied the polarization inside the dielectric material is changed, thus, changing dielectric properties. The mathematical for the phenomenon of relaxation of polarization for a dielectric material is given below. The studies were performed for pure polar materials, which contain a set of non-interacting dipoles free to rotate in the medium. The equation for complex permittivity is given by

$$\varepsilon = \varepsilon_\infty + \frac{\varepsilon_0 - \varepsilon_\infty}{1 + i\omega\tau} \qquad (2.13)$$

Where $\varepsilon_0$= Dielectric constant at very low frequency i.e. zero frequency

$\varepsilon_\infty$= Dielectric constant at high frequency

$\omega$ = Angular frequency

$\tau$ = Relaxation time

$\varepsilon_0$ represents static permittivity i.e. dielectric constant at dc value while $\varepsilon_\infty$ represents the dielectric constant at very high frequencies. $\varepsilon_\infty$ reaches a constant value since very high frequencies dipoles and free ions do not have time to realign themselves with a rapidly changing external electric field. $\tau$ represents the relaxation time, which is the time period required for the dipole to go into the random state after the removal of applied electric field.

At very low and very high frequencies the dielectric constants do not vary much and dissipation factor reaches zero. For intermediate values, the dielectric constant decreases continuously while dissipation factor attains a maximum value and drops down to zero for very high frequencies with relaxation frequency $\omega = 1/\tau$.

### 2.4.2 Temperature dependence

Dielectric properties vary significantly with variation in the temperature. The properties are dependent on the dielectric relaxation and the applied frequency. At lower frequencies, the dissipation factor increases as the temperature is increased due to ionic conductance and decreases at high frequencies with an increase in the temperature. As temperature increases, the loss peak is shifted towards higher frequencies Thus, reducing the relaxation time.

Electrical properties of oil sands are dependent on various factors which will be discussed in this study.

# Chapter 3
# Oil sands Characterization

## 3.1 Oil sands

As the conventional sources of oil are getting depleted, non-conventional oil resources are gaining in importance as an alternative. Canada has vast oil sands deposits mainly in Alberta. Oil sands are being utilized as the main source for getting synthetic crude oil from oil sands extraction as well as upgrading processes (Gray 2010).

Oil sands are mixture of sand, clay, fine minerals, formation water and electrolytes along with a dense and viscous form of petroleum referred as bitumen (Masliyah, Czarnecki et al. 2010). Oil sands are also known as tar sands or bituminous sands. Oil sands are sedimentary deposits with complex, viscous fluid with very high molecular mass.

In oil sands, sand grains (with sizes in mm range) are coated with a thin film of water envelope, the presence of which is not yet experimentally proven but has been postulated in the early development of oil sands. The bitumen layer, as the outermost layer, surrounds wet sand grains. A schematic structure of oil sands is shown in Figure 3.1 (Czarnecki, Radoev et al. 2005).

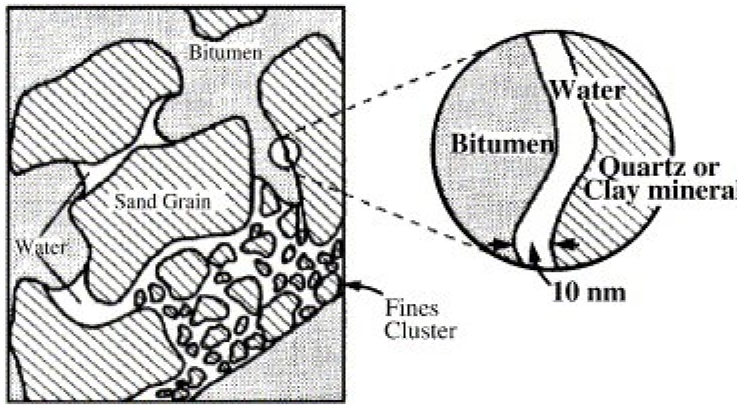



**Figure 3.1: Structure of oil sands (Reprinted with permission from Copyright (2005) Wiley-(Czarnecki, Radoev et al. 2005) )**

The quality of oil sands also known as grade and is dependent on the weight percentage of bitumen present in the deposit. Bitumen percentage in the oil sands may vary from 0% to 18%. The grade of oil sand is determined by bitumen percentage. Oil sands should contain at least 7% bitumen to be extracted for the conversion to synthetic crude oil economically. High-grade oil sands contain about 13-18% bitumen by weight, medium-grade oil sands contain about 10-12% bitumen while the low-grade oil sands consist of less than 7% bitumen by weight.

Dielectric properties of oil sands depend upon such various parameters as frequency, temperature, moisture content and bitumen content in the samples (Chute, Vermeulen et al. 1979). To find out the relationship between oil sands properties with electromagnetic heating, one needs to look into the characterization of oil sands. Thus, a few physical properties of oil sands which might affect the process of electromagnetic heating in the reservoirs are taken into the consideration in this work. These properties include the bulk properties such as density, viscosity, specific heat, heat of combustion and electric properties such as dielectric constant, dissipation factor and electrical conductivity.

## 3.2 Bitumen Characteristics

(Speight 1991) and (Gray 2010) compare the physical properties of bitumen and conventional crude oil, elemental and fractional composition as well as carbon residue and metal percentage. It can be seen that bitumen has higher sulphur content, metal composition and carbon residue than the conventional crude.

Analysis is carried out to separate bitumen into saturates, aromatics, resins and asphaltenes (SARA) fractions based on its solubility into different solvents. Bitumen is made of a mixture of hydrocarbons which can be divided into following four groups:

- Saturates: This class contains straight or branched chain hydrocarbons known as Paraffins and Isoparaffns as well as cyclic hydrocarbons known as Napthenes. Due to biodegradation, these classes have minor content in bitumen.
- Aromatics: Contain hydrocarbons with aromatic rings such as benzene, toluene, naphthalene, etc. Heavy members of this class contribute to bitumen composition.

- Resins: Resins contain polar aromatic rings which have nitrogen, oxygen, or sulphur
- Asphaltenes: Asphaltenes include complex organic compounds of high molecular weight. This class is the major contributor to bitumen and are important in the conversion processes due to their complex structures.

Depending upon the insolubility in n-alkanes, a class in bitumen is known as Asphaltene class as we have discussed previously. Asphaltenes are complex molecules with high molecular weight. High content of asphaltenes indicates that thermal treatment may lead to reduction in stability of liquid products causing asphaltenes to precipitate out (Gray 2010).

### 3.2.1 Viscosity

Bitumen is highly viscous at low temperature. It has a viscosity of more than 1,000,000 mpa.s at reservoir conditions ($20^0$ C). Viscosity is strongly dependent on the temperature; it shows a decrease of about five orders of magnitude over the temperature range of $20^0$ - $200^0$ C. (Puttagunta, Singh et al. 1993) gave the following correlation for bitumen viscosity

$$ln\mu = 2.30259 \left[\frac{b}{\left(1+\frac{T-30}{303.15}\right)^s} + C\right] + B_0 Pexp(dT) \quad (3.1)$$

$$s = 0.0066940b + 3.53641 \quad (3.2)$$

$$B_0 = 0.004742b + 0.0081709 \quad (3.3)$$

$$d = -0.0015646b + 0.0061814 \quad (3.4)$$

$$b = log_{10}\mu_{30^0C} - C \quad (3.5)$$

Where T is temperature in $^0$ C, P is pressure in MPa, $\mu_{30^0C}$ is the viscosity at 30 $^0$ C and atmospheric pressure.

### 3.2.2 Heat Capacity

Specific heat for bitumen is given by the equation 2.3 (Smith, Gowan et al. 1982)

$$C_{p,b} = 1.557 + 5.219 * 10^{-3}T - 8.686 * 10^{-6}T^2 \quad (3.6)$$

For 50<T<300 $^0$ C

## 3.3 Oil sands Characteristics

### 3.3.1 Heat Capacity

Specific heat capacities of Athabasca oil sands can be found out by the general equation given by (Smith, Gowan et al. 1982).

$$C_{P,o/s} = f_{cs}C_{P,cs} + f_{fs}C_{P,fs} + f_bC_{P,b} + f_wC_{P,w} \quad (3.7)$$

where $f_{cs}$, $f_{fs}$, $f_b$, $f_w$ represent the weight fractions of coarse solids, fine solids, bitumen and water in oil sands. Specific heats of the components with temperature are given as follows

$$C_{p,cs} = 0.738 + 1.518 * 10^{-3}T - 2.026 * 10^{-6}T^2 \quad (3.8)$$

$$C_{p,fs} = 0.778 + 1.400 * 10^{-3}T - 0.964 * 10^{-6}T^2 \quad (3.9)$$

$$C_{p,b} = 1.557 + 5.219 * 10^{-3}T - 8.686 * 10^{-6}T^2 \quad (3.10)$$

where specific heats are measured in J/deg g and temperatures are measured in $^0$C. Specific heat of water is taken to be $C_{p,w} = 4.19 \frac{J}{\text{deg}.\,g}$ upto 100$^0$ C.

# 3.4 Thermophysical Characteristics

## 3.4.1 Thermogravimetric Analysis

The aim of our study is to characterize oil sands with frequency as well as temperature. As discussed earlier, oil sands are mixture of sand, clay, fines, water and bitumen, which contain volatile petroleum components. As temperature increases, water as well as bituminous components undergo fractionation and volatiles are lost during the process, thus leading to changes in the properties of oil sands samples. So it would be essential to study how oil sands and its components behave with variation in the temperature. The influence of temperature on the properties of bitumen and oil sands were characterized using a Thermogravimetric Analyzer (TGA) and differential scanning calorimeter (DSC) (Rosenvold, DuBow et al. 1982, Rajeshwar 1983). Thermogravimetric Analyzer is used for the purpose of thermal analysis where the physical or chemical properties of sample vary with increasing the temperature. TGA records the weight of the sample with time as the sample is subjected to heat. As the temperature is increased, different components in the sample start to decompose and the percentage weight loss of the sample with temperature can be measured.

Athabasca oil sands samples were obtained from Institute for Oil Sands Innovation (IOSI) at University of Alberta. Thermal behaviour of oil sands and bitumen has been characterised by using TGA-DSC instrument. Calibration of DSC was done by taking a sample of Indium. For the reference in DSC, an empty measurement pan was used. Signals for the oil sands samples were observed in the temperature range 20°C-600°C with heating rate 10° C/min. The experiments were carried out for three grades of oil sands as well as on bitumen sample. The information about the system is given below:

Purge Gas used: Nitrogen 50.0 ml/min

1: Data storage off to erase unwanted data from ambient temperature conditions.

2: Equilibrate at 20.00 °C

3: Isothermal for 10.00 min to establish constant temperature for DSC.

9: Isothermal for 10.00 min

10: Ramp 10 °C/min from 20 to 600 °C

The results obtained are compared by using TA universal analysis software. The heating curves obtained from TGA analysis are shown in the figures 3.2 and 3.3 while the heating curve for DSC is shown in 3.4. TGA curves give the information about the percentage weight loss with temperature and time. The figure 3.2 shows about 14% weight was lost when the temperature was increased to 600°C while figure 3.3 shows around 4% weight was lost when temperature was increased to 250°C. This shows that the major component lost until 250 °C was water, while the major components lost were bituminous products in the temperature range 250°C to 600°C. It can be seen that the heating curve consists of a peak around 105°C in figure 3.4. It represents the endothermic event where phase transition i.e. evaporation takes place. The information extracted from the thermophysical characteristics will prove to be useful in interpreting the temperature dependence of dielectric properties.

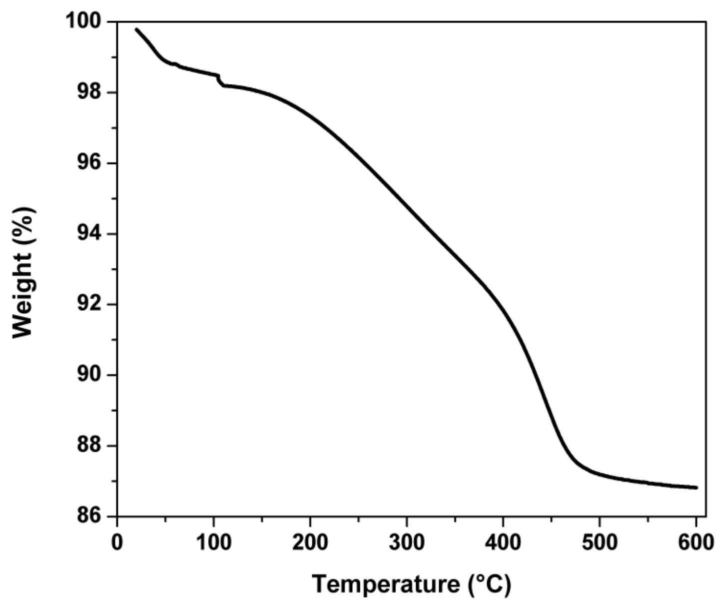



**Figure 3.2: TGA graph of oil sand sample (temperature range 25$^0$C - 600$^0$ C)**

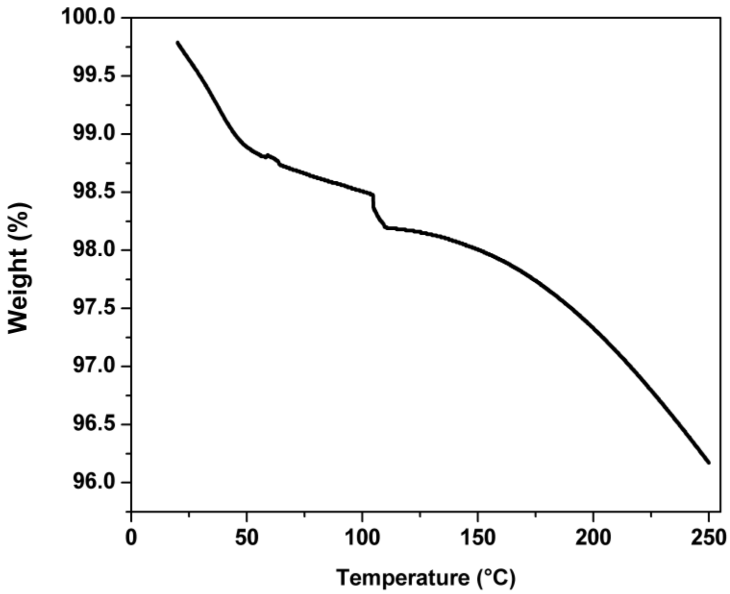



**Figure 3.3: TGA graph of oil sand sample (temperature range 25$^0$C -250$^0$C)**

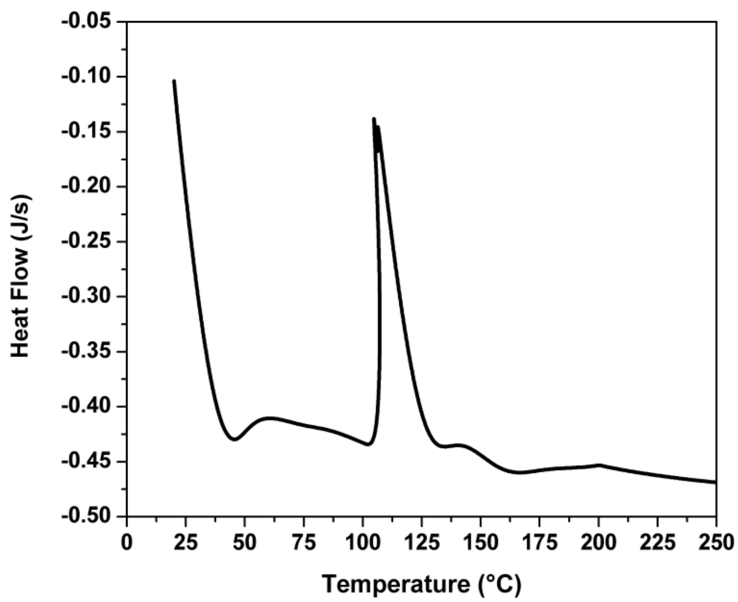



**Figure 3.4: DSC thermograph of oil sand sample**

### 3.4.2 Dean Stark Apparatus

Dean Stark apparatus was used to determine the composition of water, bitumen and solids in the oil sands. (Jia 2010, Zhu 2013) A simple Dean Stark apparatus consists of an assembly of round bottom flask, reflux condenser and a trap. The bottom flask contains the organic liquid (toluene) which is used for the extraction process. The top vertical glass tube acts as a condenser for a volatile component and refluxes it back to the trap. The top of the trap is connected to the bottom of the reflux condenser. The trap acts as a separator for the liquids recovered from the condenser based on the density differences. The side arm of the trap consists of a thimble where the sample in this case oil sand sample is placed.

For the analysis, 50 gm of oil sands sample is taken into the thimble placed in the side arm of the trap. Around 200 ml of pure toluene is taken into the round flask and is subjected to heat. The vapours containing toluene and water rise to the condenser; get cooled and condense down to the separating trap. The immiscible liquids form two layers with toluene on top and water in bottom. As more vapours pass through the condenser, the level of the condensate rises in the trap too. Condensed toluene starts to flow back to the flask once the level at the side arm is crossed. This reflux stream of toluene comes in contact with the oil sand sample placed on the side arm of the trap. Bitumen in the sample gets dissolved in the reflux toluene and comes down in the flask. The analysis is carried out till the reflux stream coming out of thimble becomes colourless and the water level in the trap is stabilized.

Once the process is complete, the water is carefully drained from the trap and weighed to get the weight of water in oil sand sample. Dark coloured solution containing bitumen in toluene is then transferred to 250 ml flask. Pure toluene is added to make it up to the 250 ml level mark. The solution was well shaken and 5 ml of the dark solution was taken and sprayed on the filter paper weighed before. Filter paper was dried for some time to ensure all volatile toluene has gone off. The filter paper was weighed again. The difference between initial and final weights of filter paper gave the weight of bitumen present in 5 ml of the solution. For getting the weight of bitumen in the oil sand sample, the result was multiplied by 50. The remains in the thimble were dried and weighed to get the solid content.

Oil sands sample were taken from Institute for Oil Sands Innovation (IOSI) at the University of Alberta. Dean Stark Apparatus was used to find out the weight contribution of the components in the oil sands samples. The extraction analysis reported that the low-grade oil sand sample was comprised of 3-4% bitumen and 8-10% water by weight, with the remainder being fine-grained solids and fines.

Similar studies were performed for medium-grade and high-grade oil sands samples and the results are tabulated below:

| | Proximate analysis (wt. %) | | |
|---|---|---|---|
| | Water | Bitumen | Solids |
| **High Grade** | 2-2.5% | 13-14% | Remaining |
| **Medium grade** | 4-5% | 6-7% | Remaining |
| **Low grade** | 8-10% | 3-4% | Remaining |

**Table 3.1: Proximate Analysis of oil sands**

### 3.4.3 Fourier Transform Infrared Spectroscopy (FTIR)

Spectroscopy is the study of interaction between the incident rays and the compound to be analyzed. Infrared spectroscopy is a technique used to identify the chemical bonds present in the compound and thereby determining the functional groups present. Infrared rays are passed through the samples; some part is absorbed by the sample while some part is reflected back. The spectrum obtained represents the molecular interactions between the radiation and sample. There are different FTIR techniques for extracting the information. The best-suited technique for powders and rough surfaces is Diffuse Reflectance Infrared

Fourier Transform Spectroscopy (DRIFTS) technique. Thus, DRIFTS is used for getting the information from oil sands samples.

Diffuse reflectance is caused by incident radiation on the rough surface, which then travels through the sample and gets reflected or scattered from internal surfaces. The intensity of reflectance signal is observed to be low at the frequencies where the absorption is seen to be maximum. Sample preparation for DRIFTs method is very simple. The powdered sample is placed into a sample cup and the signal from the sample is observed. As the sample used was too absorbent, it was diluted by mixing with KBr to get a better signal. The spectrum was observed in the wavelength ranging from 650nm to $4000nm^{-1}$.

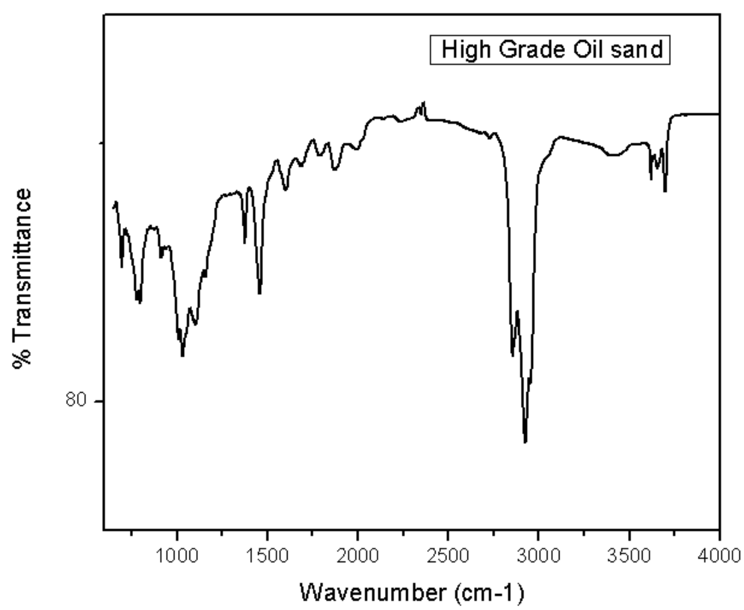



**Figure 3.5: FTIR spectrum for high-grade oil sand sample**

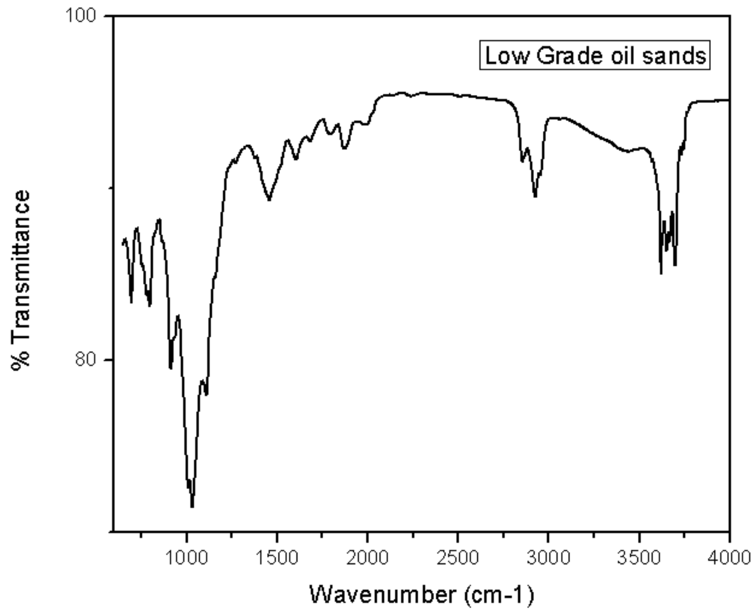



**Figure 3.6: FTIR spectrum for low-grade oil sand sample**

Figure 3.5 and 3.6 show the FTIR spectra for high-grade and low-grade oil sand sample respectively. The peaks obtained at different frequencies are the representatives of particular bonds present in the sample (Jiang, Zhao et al. 2007, Yoon, Son et al. 2009). The peak near 3500 $cm^{-1}$ is assigned for Si-OH and adsorbed water on clay. The bands near 1600 $cm^{-1}$ are due to distortion vibrations of the water molecules adsorbed. The band observed at 1083 $cm^{-1}$ is the characteristic of Si-O-Si bond which represents the clay present in the sample. The strong band observed near 2924 $cm^{-1}$ is the characteristic of C-H bond which represents the presence of significant amounts of bitumen in the sample (Bukka, Miller et al. 1991).

# Chapter 4

# Experimental setup

This chapter illustrates a comparison between the different permittivity measurement techniques. The selection of one of the techniques used in our experimental process, depending upon different factors, is also explained. The detailed information about the experimental setup, sample preparation, calibration and associated software has been included in this chapter. The experimental setup and the working principle for the Impedance Analyzer have been explained in detail. In the later part, the calibration of the setup has been described followed by the discussion on data collection software and step by step experimental procedure.

Different techniques for measurement of dielectric properties are mentioned in the literature. The most common techniques used are the transmission line technique, the coaxial probe technique, the cavity perturbation technique, free space technique and the parallel plate technique (Agilent Technologies 2009), (Agilent Technologies August 2012). Many factors are involved in the selection of a particular measurement technique for desired measurements. A few of them are listed below:

- Desired frequency range
- Expected values of dielectric properties
- Material properties, shape of the material
- State of the material (i.e. Liquid, powder or solid)
- Desired temperature range
- Measurement accuracy
- Method of measurement (contacting or non-contacting)
- Cost effectiveness

## 4.1 The parallel plate capacitance technique

Our experimental setup consists of a 4294A Precision Impedance Analyzer by Agilent with operating frequency range of 40 Hz to 110 MHz for the purpose of permittivity measurement.

The parallel plate capacitance method is the most common technique for measuring dielectric properties at low frequencies. By considering the sample to be oil sands and the operating frequency range as the lower frequency range for the measurement, it was concluded to use the parallel plate capacitance technique.

## 4.2 Oil Sand Samples

Oil sands sample were taken from Institute for Oil Sands Innovation (IOSI) at the University of Alberta. The samples were stored in the refrigerators at around $4^{o}$ C - $5^{o}$ C in airtight containers. Sufficient amount of oil sands sample were taken in small containers or in petri dishes to make pellets as test samples. The measurements were performed at room temperature i.e. around $20^{o}$ C. Solvent extraction process was used to determine the composition of water, bitumen and solids in the oil sands, which has been described in the previous chapter.

## 4.3 Experimental setup

4294A Precision Impedance Analyzer by Agilent is used to measure the dielectric properties of the oil sand samples. The parallel plate capacitance method is taken into account to measure the impedance of the system. The principle of this method is to shape the sample in such a way that it is possible to measure the capacitance of the desired sample. The capacitance for the assembly is measured by sandwiching the samples (in the form of thin sheets or pellets) between two electrodes. Experiments were performed for a frequency range of 100 Hz to 1 MHz. The effect of variation in the temperature of the samples was also observed on the dielectric properties for particular frequencies. Capacitance and dissipation factor were measured using a parallel plate capacitor with an impedance analyzer. The dielectric constant was evaluated from the measured capacitance. Moreover, the dissipation factor was found out from the values of D in the measurements.

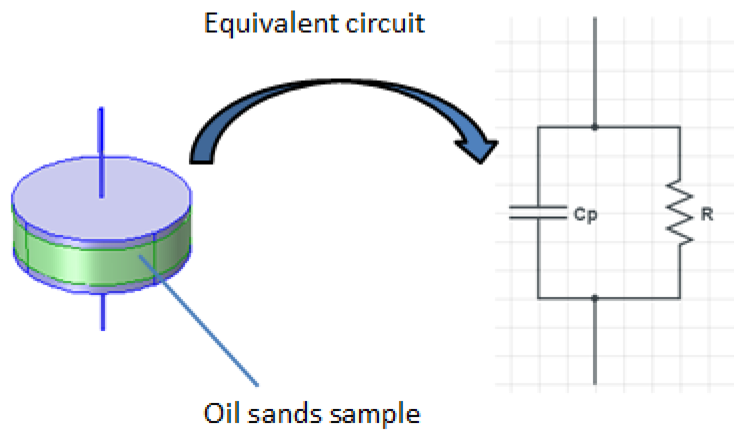



**Figure 4.1: Representation of dielectric material as an equivalent circuit**

The capacitive system can be simplified as the equivalent circuit of capacitive as well as resistive elements (Agilent Technologies May 2014) as shown in the figure 4.1. The complex permittivity and relative complex permittivity for the system are given by equations

$$\varepsilon = \varepsilon' - j\varepsilon'' \tag{4.1}$$

$$\varepsilon_r = \frac{\varepsilon' - j\varepsilon''}{\varepsilon_0} = \varepsilon'_r - j\varepsilon''_r \tag{4.2}$$

The effective admittance of parallel plate pellet is given by

$$Y = \frac{1}{Z_{effective}} = G + j\omega C_P \tag{4.3}$$

$$Y = j\omega C_0 \left(\frac{C_P}{C_0} - \frac{G}{\omega C_0}\right) \tag{4.4}$$

Where $C_0$ is the capacitance of the air

Thus,

$$\varepsilon_r = \left(\frac{C_P}{C_0} - j\frac{G}{\omega C_0}\right) \tag{4.5}$$

$$\varepsilon''_r = \frac{t}{\omega\ R_P\ \varepsilon_0 * A} \tag{4.6}$$

$$\varepsilon'_r = \frac{C_P}{C_0} = \frac{C_P * t}{\varepsilon_0 * A} \tag{4.7}$$

$$tan\delta = \frac{\varepsilon''_r}{\varepsilon'_r} = \mathrm{D} \tag{4.8}$$

Where

$C_P$: Measured capacitance of the system [F]

$t$ : Thickness of the sample [m]

$A$ : Cross sectional area of the electrode [$m^2$]

D : Measured value of dissipation factor

## 4.4 Experimental setup

The experimental setup is shown in the figure 4.2. Overall, experimental setup consists of main components listed below i.e.

- Impedance Analyzer for measuring the impedance parameters such as impedance, phase angle, capacitance, dissipation factor
- Test fixture for the measurement of dielectric properties with the variation in frequency as well as temperature
- Oil sands samples made in shapes of pellets used as parallel plate capacitors
- Data acquisition software and high performance GPIB (General Purpose Interface Bus) interface for data collection

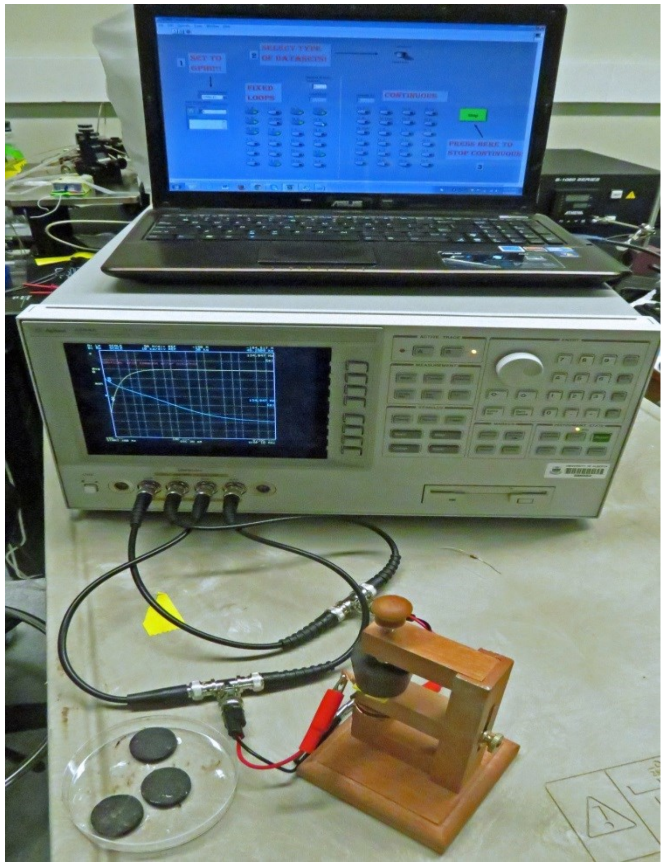


**Figure 4.2: The experimental setup for measurement of dielectric properties**

### 4.4.1 4294A Precision Impedance Analyzer

The 4294A precision impedance analyzer is used for the accurate impedance measurement and analysis of circuits. The Impedance Analyzer can be operated in the wide frequency range of 40 Hz to 110 MHz (Agilent Technologies 2006, Agilent Technologies 2008) and it can measure the impedance of the system ranging from 10 mΩ to 200 MΩ. The device can be operated in the temperature range of 20°C to 200°C. It is a very effective instrument for evaluating the characteristics of circuit components like capacitors, inductors, resonators and semiconductors. Different parameters can be evaluated by using the impedance analyzer. The measured parameters are the Impedance Z, capacitance $X_C$, inductance $X_L$ for different frequencies. Other measurement parameters are Admittance |Y|, Phase angle (θ), Conductance (G), Dissipation factor (D), Quality factor (Q). Experiments for dielectric properties measurement were performed for a frequency range of 100 Hz to 1 MHz. The effect of variation in the temperature of the samples was also observed on the dielectric properties for fixed frequencies.

### 4.4.2 Test fixture and Oil sands pellets

As discussed earlier, dielectric properties were measured using a parallel plate capacitor with an impedance analyzer. The dielectric constant was evaluated from the measured capacitance of the dielectric system. For the evaluation of capacitance of the system, a few parallel plate assemblies have been constructed in house. These cells consisted of copper sheet cut into two electrodes depending upon the shapes of capacitor assembly sandwiched between them.

Oil sands are a mixture of sand, clay, fine minerals, formation water and bitumen. Typical oil sands samples contain 4%-16% bitumen, 1% -10% water and the remainder is sands and clays. Oil sands samples are loose and the grains are of varying sizes. It was difficult to make uniform physical contact between the copper sheet electrodes and the uneven granular surface of the oil sand sample.

The major challenge was making the parallel plate assembly, which can evaluate the permittivity of oil sands samples. In order to overcome the problem of non-uniform contact, a compact system is designed. The assembly consists of two thin copper sheet electrodes of 0.15 inch thickness and 2.54 cm in diameter. These electrodes were separated by inserting the oil sand pellets used for the measurements. The pellets of oil sands with diameters 1 inch and 2 inch and different thickness were prepared by using the Hydraulic Press and Pressing Die. Around 3 gm of loose oil sand sample is taken from the airtight container and weighed before compressing. The force of 8 tonne was applied to the oil sand sample and the prepared pellets were gently taken off. The thickness of oil sands pellets is dependent on the weight of the loose oil sands sample taken. The pellets prepared from the samples using the hydraulic press assembly.

### 4.4.3 Data acquisition software and GPIB interface cable

Agilent 4294A Impedance Analyzer is connected to an external controller (computer) through a General Purpose Interface Bus (GPIB) cable. General Purpose Interface Bus (GPIB), the interface connecting a computer with Impedance Analyzer is one of the interface standards accepted worldwide. The direct control over Impedance analyzer can be attained through GPIB interface. The computer sends the commands through GPIB to impedance analyzer and the data obtained after the measurements is sent back to the computer. The data stored in the computer is then processed to get the desired dielectric properties.

## 4.5 Calibration of Impedance Analyzer

The measurements done by Impedance Analyzer may contain some errors due to instrument inaccuracies, noise or due to additional residuals in the test fixture and the cables used for the connections. In order to get rid of the measurement errors, proper measurements techniques should be used (Agilent Technologies 2009) . Calibration is the process of verification of the accuracy by comparing it with the measurements of standard devices. Calibration of the instrument should be done to eliminate the measurement errors in the dielectric properties. The accuracy of the instrument may be reduced after few measurements.

4294A Impedance Analyzer is calibrated by using "User Calibration" process as mentioned in the Operation Manual by Agilent. It consists of three steps

1. OPEN Calibration: The connections are made according to the OPEN standards and then Open Calibration mode is activated to get OPEN calibration
2. SHORT Calibration: After OPEN calibration is done, SHORT standard connections are made and the SHORT calibration is carried out.
3. LOAD Calibration: A load of known parameters is connected with the Impedance Analyzer. LOAD mode is activated to start LOAD calibration data measurement. The calibration data measured generally matches with the parameters known beforehand.

Once the calibration process is complete, the calibration coefficient is automatically saved to the internal memory. Thus, it is desirable to calibrate the instrument every time it is powered on or any input setting is changed.

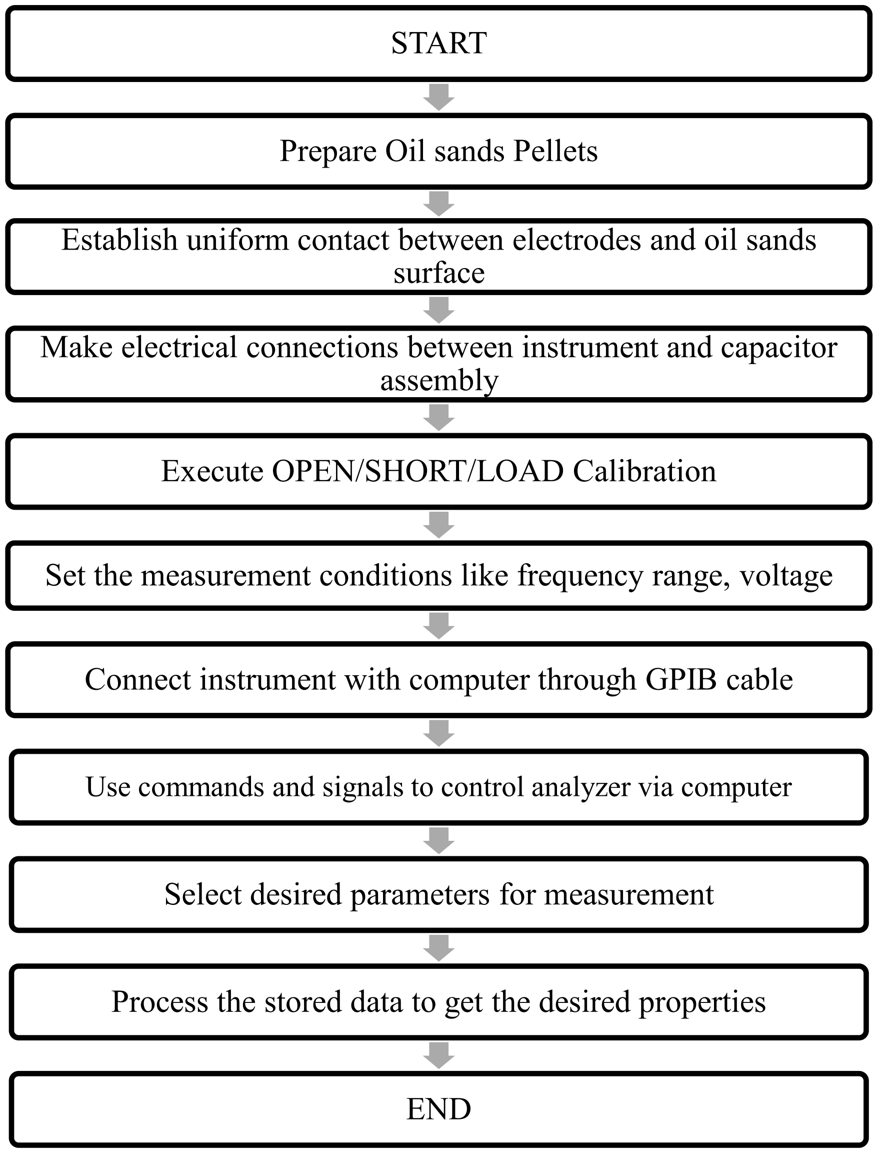
## 4.6 Experimental Procedure

The procedure for measuring the dielectric properties is explained below. The parameters obtained through measurements are capacitance ($C_P$), dissipation factor (D), and conductance (G).

1. Loose oil sand sample was taken and pellets of 1inch diameter and appropriate thickness were prepared by using the Hydraulic Press and Pressing Die assembly.
2. Pellets were prepared with uniform contact between the copper electrodes and uneven oil sands surface by using the assembly.
3. Electric connections were made between the electrodes and 4294A Impedance Analyzer.
4. As discussed previously, calibration process is followed to eliminate the errors due to noise, instrument inaccuracies and residuals caused by the fixture as well as cables attached to the instrument.
5. The measurement conditions were set then. The frequency range was set from 100 Hz to 1 MHz. The number of points for the measurement was set to 700. The sweeping time was fixed to 20 seconds.

6. Impedance Analyzer was connected to external computer through General Purpose Interface Bus (GPIB) cable.
7. The data acquisition software was operated through computer to control the commands and signals to Impedance Analyzer.
8. The desired parameters for measurements were selected using the interface between the instrument and computer. Capacitance ($C_P$), dissipation factor (D), conductance (G) and a few more pairs of parameters were selected.
9. The data stored in the computer was then processed to get the desired dielectric properties.

The flowchart for the experimental procedure is given below:


START
Prepare Oil sands Pellets
Establish uniform contact between electrodes and oil sands surface
Make electrical connections between instrument and capacitor assembly
Execute OPEN/SHORT/LOAD Calibration
Set the measurement conditions like frequency range, voltage
Connect instrument with computer through GPIB cable
Use commands and signals to control analyzer via computer
Select desired parameters for measurement
Process the stored data to get the desired properties
END

# Chapter 5
# Results and Discussion

This chapter includes the results and discussions on electric properties. The dependence of oil sands properties on frequency and temperature is also analysed in this section.

## 5.1 Impedance Analysis

When an external electric field is applied to the material, the effective electrical response contains two components. The component which is in phase with the applied voltage represents the loss, while the component which out of phase with the applied voltage represents the energy storage within a system (Knight, 1984). To understand the dielectric properties of oil sands, it is necessary to develop some understanding of the electrical response when an external field is applied. Loss and storage components can be represented either in terms of a series RC circuit or a parallel RC circuit or a combination of both. Impedance Z and θ are measured for medium-grade oil sand sample at room temperature for a frequency range of 100 Hz-1 MHz (see figure 5.1). The equivalent circuit for the system can be derived using the Nyquist plots of the real part of impedance vs imaginary part of impedance for different frequencies. Figure 5.2 represents the variation of imaginary part of impedance with the real part, which consists of semicircular arc and an inclined line.

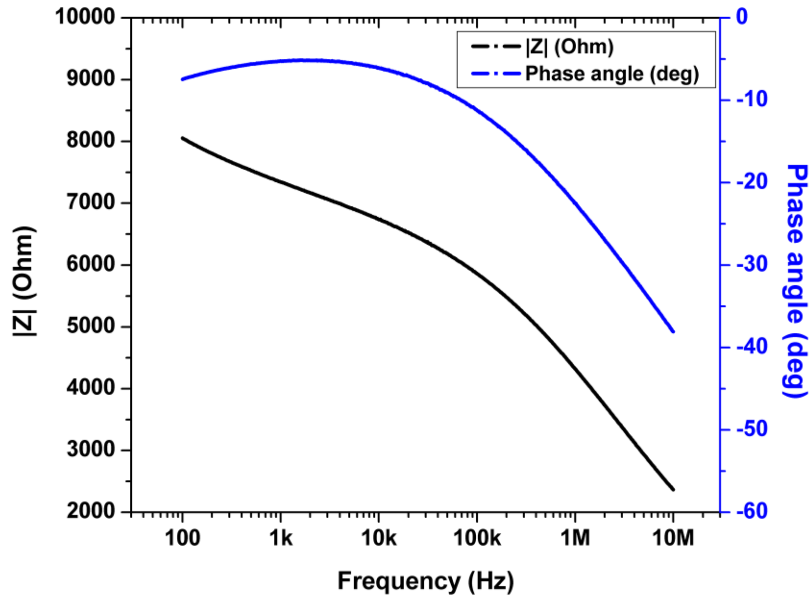



**Figure 5.1: Impedance variation with frequency for medium-grade oil sand sample**

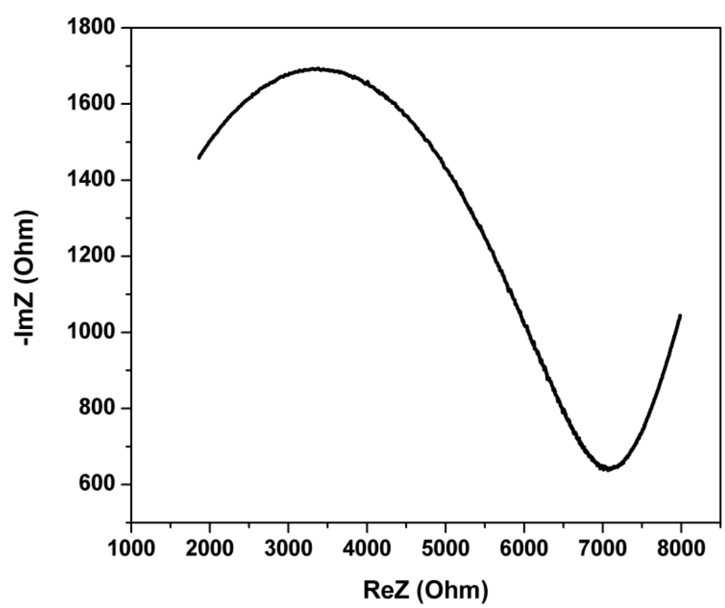



**Figure 5.2: Variation of imaginary part with real part of impedance**

The point at the end of inclined line is for the lowest frequency, as the frequency increases the graph travels to the left down line and along the semicircle in counter clockwise direction. The frequency at the lowest point which separates linear part from semicircular part is denoted by $f_0$. Significant work has been done by Knight (1984) to interpret and represent the frequency response of sandstone into the equivalent electrical circuit by using basic Debye circuit.

From the complex impedance analysis, it is evident that there are two regions in the frequency response. The lower frequency region corresponds to the linear response while the higher frequency region corresponds to semicircular arc in the figure 5.2. Using the Debye circuit as an approximate equivalent circuit, both lower as well as higher frequency responses can be modelled as electrical circuits with resistor and capacitors. If we consider higher frequency response, it can be modelled as a circuit with resistor and capacitor connected in parallel with each other.

The measurements for impedance parameters Z and θ for high-grade oil sand and low-grade oil sand sample at room temperature for the frequency range of 100 Hz-10 MHz are shown in Figures 5.3 and 5.4.

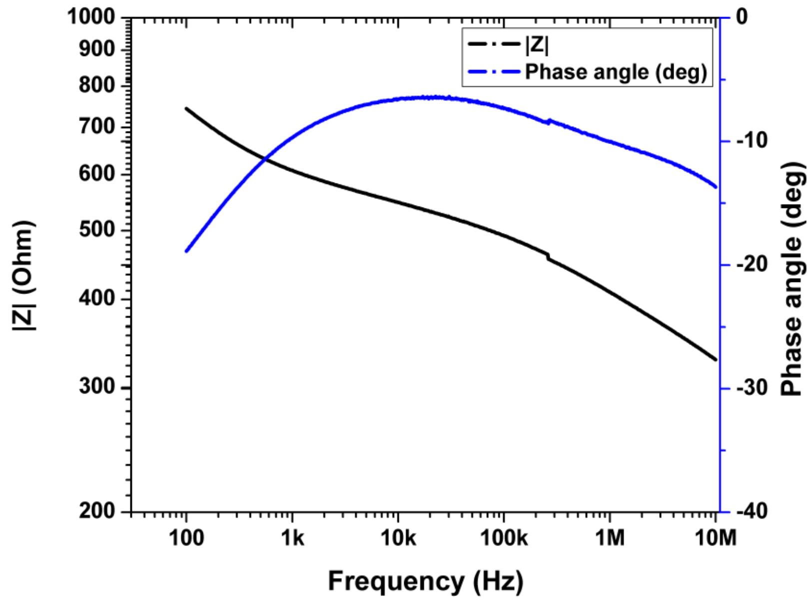



**Figure 5.3: Impedance variation for high-grade oil sand sample**

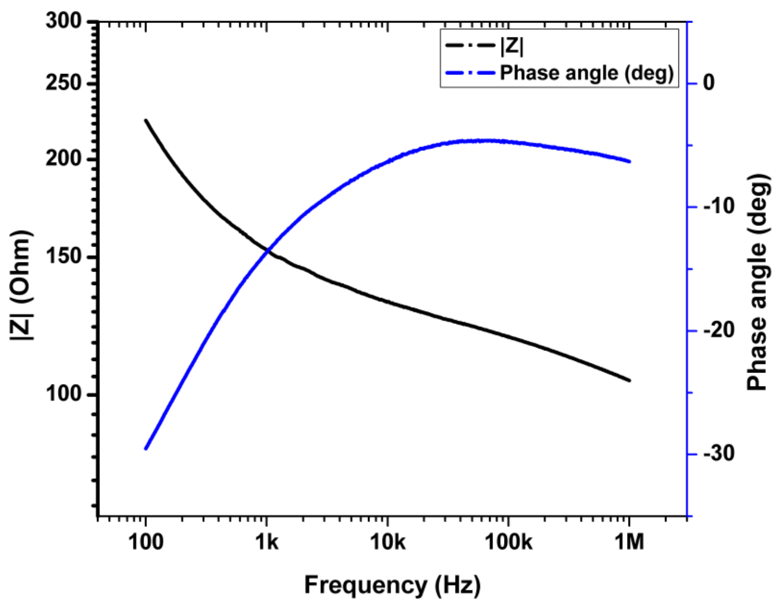



**Figure 5.4: Impedance and phase angle variation with frequency for low-grade**

Analysis of impedance parameters Z and θ will yield information about the electrical properties of oil sand system. The comparison between the measurements of impedance parameters Z and θ for high-grade oil sand, medium-grade and low-grade oil sand samples can be summarised as

| Oil sand | Phase angle | Impedance |
|---|---|---|
| High Grade | Varied from -25° to -10° | In the range of ohms |
| Medium Grade | Varied from -10° to -40° | In the range of kilo ohms |
| Low Grade | Varied from -30° to -5° | In the range of ohms |

**Table 5.1: Comparison of impedance parameters**

It can be seen that in the higher frequency range, high-grade and low-grade samples show resistive behaviour while medium-grade sample shows resistive to capacitive behaviour. The impedance data indicates that medium-grade oil sand sample exhibit high impedance while other samples have low impedance. It can be said that high-grade and low-grade oil sand samples have similar impedance behaviour while medium-grade behaves differently. Any conclusion regarding the direct dependence of electrical properties of oil sands can not be drawn. Presence of water, ions, and clays could be causing the oil sand medium to behave differently. But, as oil sand medium is complex mixture, it is difficult to conclude about the contribution of each component. The possible effect of different parameters on electrical properties is discussed in this section.

## 5.2 Factors affecting impedance parameters

Typical oil sands consist of a mixture of sand, clay, fine minerals, water, electrolytes and bitumen (Masliyah J. H., Czarnecki J., Xu Z. 2011). The grades of oil sands are determined by the percentage bitumen content in the oil sands. Associated components in the oil sands vary with the grade of oil sands. The possible effect of these factors on impedance is described.

### Water content

Water content in oil sands can vary from zero to around 7% in the oil sands. Water can be present as bound water as well as free water. Water content is highest in the low-grade oil sands ore while it is lowest in high-grade oil sands ore. The indigenous water in oil sands contains a variety of free ions such as sodium, calcium, magnesium, chloride and sulphate (Masliyah, Czarnecki et al. 2010). These ions increase the conductivity of oil sand medium, which plays a major role in the electric properties of oil sands. The low impedance in low-grade oil sands ore can be explained by the highest content of water in oil sands medium. Medium grade oil sands ore shows less impedance than low grade which falls in line with the idea. But low impedance shown by high-grade oil sands ore implies that the impedance behaviour is not dependent only on water content in the medium but on other factors too. A large variation in the ionic concentration of electrolytes occurs in oil sands ores might also effect the impedance characteristics of oil sands.

## Clays and fines

Mineral solids present in the form of clays and fines may affect the impedance properties of oil sands. Fines are mineral solids with dimension less than 44 μm. Clays are classified as mineral solids which are smaller than 2 μm. The clays are composed of tetrahedron and octahedron structures of silicon-oxygen and aluminum sheets. Clays are charged species and have the property to exchange ions with water. This might result in the availability of free ions in the system which may affect the impedance behaviour of oil sands system. Heavy metals found in mineral solids might also help in enhancing conduction and thus lowering the impedance.

## Geometrical arrangement

Another factor which may be affecting the electrical properties could be the geometrical arrangement of oil sands sample. Oil sands is a heterogeneous medium which does not have a particular packing or geometrical pattern. The oil sands samples used had different pore distributions and microstructures. The microscopic roughness at the interfaces will change the impedance characteristics of the material. Due to complex interactions between water-bitumen-mineral solids, it is difficult to interpret the effect of individual component on electrical properties as such. Thus, a qualitative analysis is done to understand the dependence of impedance parameters on frequency.

## 5.3 Temperature Dependence: Impedance

Further, we need to investigate the impedance properties of oil sands samples with temperature as it will prove to be useful in the electromagnetic heating studies going on in our group.

Experiments were carried out to study the electrical properties of oil sands as a function of temperature ranging from 25 °C to 250 °C. The experiments were carried over a range of frequency from 100 Hz to 1 MHz. The temperature analysis was done for three frequencies chosen from the spectrum i.e. 1 kHz, 200 kHz and 1 MHz. The impedance parameters used in the study are Z and θ. Figures 5.5, 5.7 and 5.9 illustrate the variation of impedance, Z, at different temperatures for high-grade, medium-grade and low-grade oil sands samples. Figures 5.6, 5.8 and 5.10 represent the compilation of phase angle curves at the specified frequencies with temperature variation.

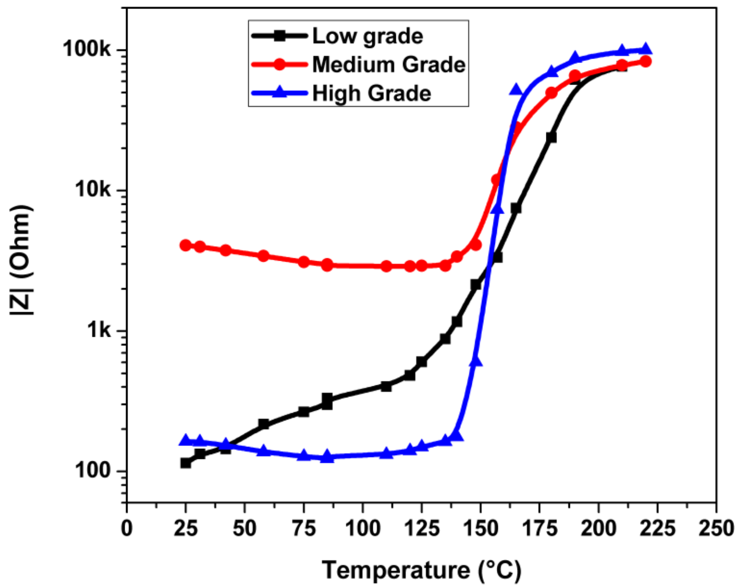



**Figure 5.5: Impedance variation with temperature (1 kHz)**

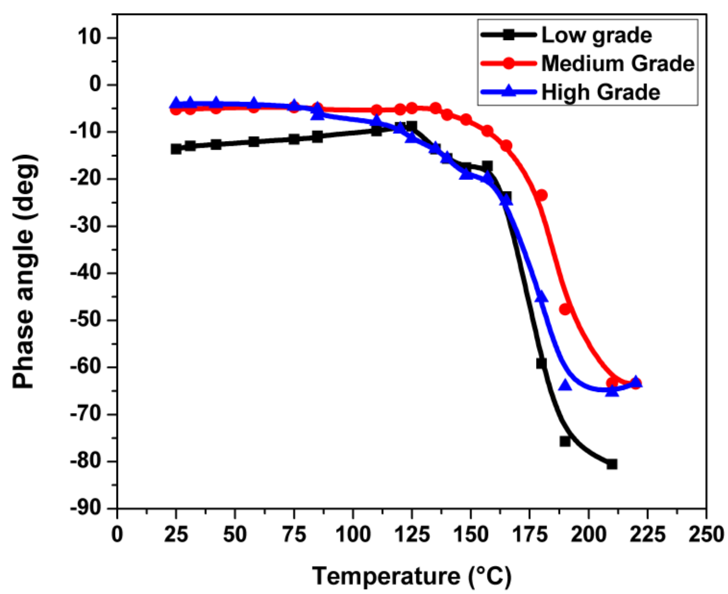



**Figure 5.6: Phase angle variation with temperature (1 kHz)**

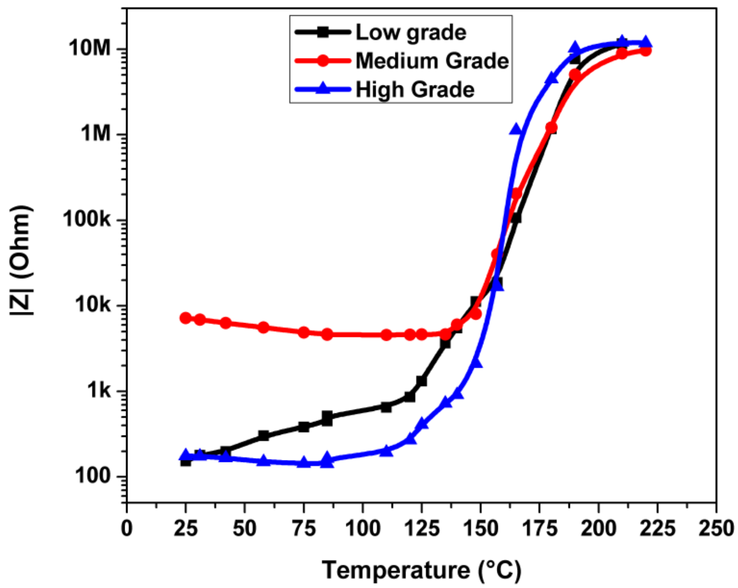



**Figure 5.7: Impedance variation with temperature (200 kHz)**

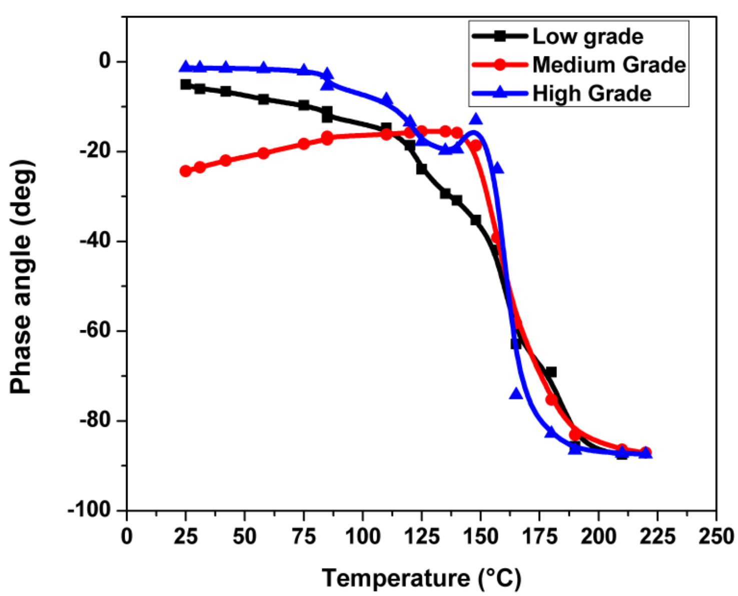



**Figure 5.8: Phase angle variation with temperature (200 kHz)**

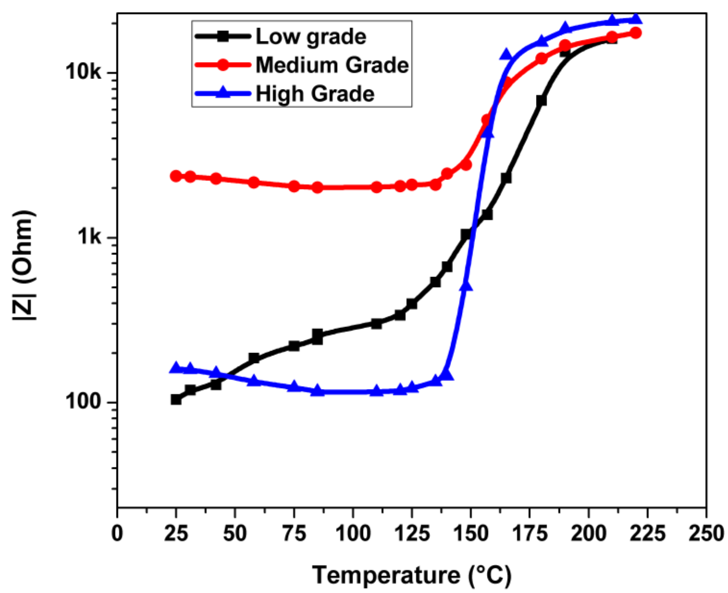



**Figure 5.9: Impedance variation with temperature (1 MHz)**

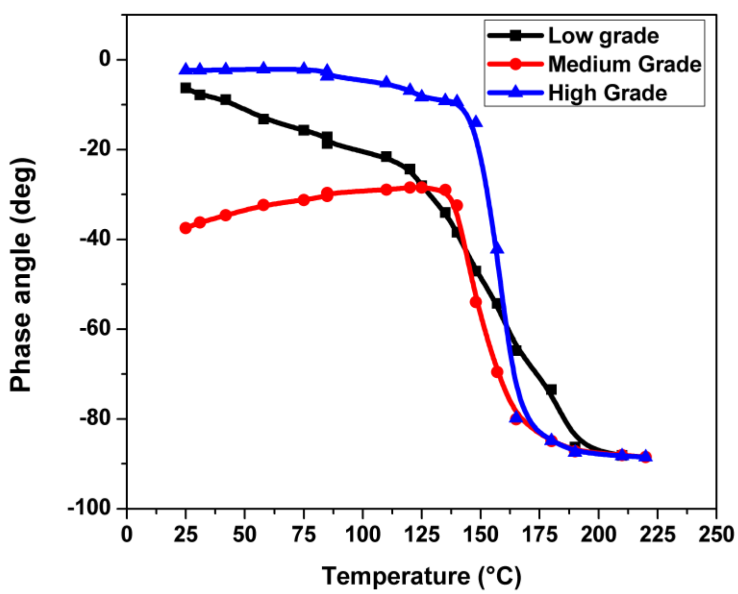



**Figure 5.10: Impedance variation with temperature (1 MHz)**

The comparison between the measurements of impedance parameters Z and θ for high-grade oil sand, medium-grade and low-grade oil sand samples can be summarised as

- At lower temperatures, impedance (Z) was found to be the highest in low-grade oil sands sample for all three frequencies.
- As the temperature was increased, impedance Z increased slowly till 140 °C in low-grade and high-grade oil sands samples while it showed a decreasing trend in medium-grade oil sands.
- It was found that the impedance for all three grades increased rapidly around the temperature of 140 °C. Phase angle θ decreased rapidly around the temperature of 140 °C for all three grades of oil sands. It shows that the system is changing its impedance behaviour rapidly around the temperature of 140 °C. Rapid decrease in phase angle indicates that the system is becoming more capacitive in nature.
- At temperatures higher than 180 °C, impedance (Z) was found to have very large values for all oil sands samples in for all three frequency studies. Increase in impedance at higher temperatures indicates the increase in resistance and thus decrease in conduction.
- At temperatures higher than 180 °C, phase angle (θ) was decreased and approached the value around – 80° for all frequency studies. As the phase angle decreases to –80 ° the system will be having capacitive nature predominating.

The sharp changes in impedance parameters Z and θ indicate that the electrical as well as physical properties of oil sand samples are changing around the temperature of 140 °C. It can be stated that the factors affecting the impedance properties might be the loss of water from oil sands matrix. The sharp change shows that the water evaporation from the system led to decrease in the conductivity through the sample. Other factor attributed to this change can be the increase in the fluidity of bitumen in the oil sands matrix. Bitumen is too viscous to flow at room temperature but as the temperature increases above 90 °C it starts flowing. At the temperatures above 120°C the viscosity reduction will be significant and it will lead to free flow of bitumen through oil sands matrix. The fluid bitumen may start flowing due to gravity towards bottom electrode resulting in increase in the impedance and capacitive behaviour. The temperature $< 170$°C represents to the boiling point of naptha components in the fractionation of bitumen (Yoon, Son et al. 2009). This shows naptha cut of the bitumen gets fractionated and cracking loss of naptha form oil sands matrix. This would lead to increase in the impedance and capacitive nature of oil sands.

The information about the changes in impedance properties with frequency and temperature will turn out to be useful in understanding the electrical behaviour of oil sands system. This study is further extended for analysis of two more electrical parameters known as dielectric constant and dissipation factor. As discussed in the previous chapter, analysis of these factors will turn out to be very helpful in understanding the electromagnetic heating of oil sands.

## 5.4 Dielectric property analysis

Previously, attempts have been made by (Erdogan, Akyel et al. 2011, Chute, Vermeulen et al. 1979, Vermeulen, Chute 1983) to find out the electrical properties for oil sands. The parallel plate capacitor method is used to measure the dielectric properties in this study. Impedance measurements were done over a frequency varying from 100 Hz to 1 MHz using an Impedance analyzer and parallel plate capacitor cell system. Dielectric properties were derived from the data obtained from impedance measurements. Dielectric constant and dissipation factor are measured for high-grade, medium-grade and low-grade oil sand samples at room temperature.

Dielectric constants and dissipation factor (DF) for three different grade oil sand samples measured were compiled in the figures 5.11 and 5.12 respectively.

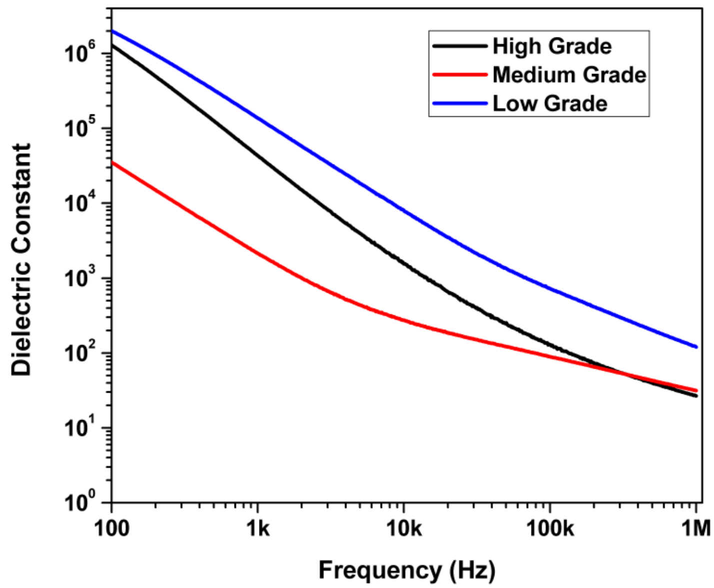



**Figure 5.11: Relative dielectric constants for three grades of oil sand samples**

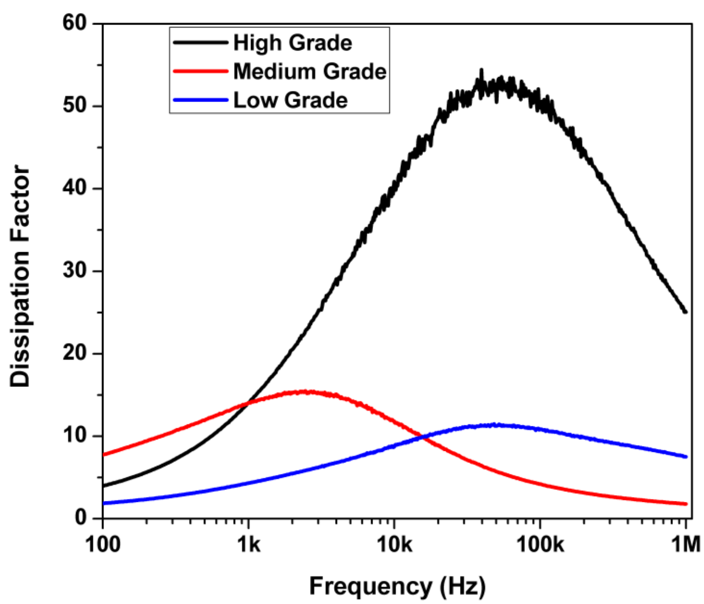



**Figure 5.12: Dissipation factor (DF) for three grades of oil sand samples**

The experiments were repeated with a number of samples for high-grade, medium-grade as well as low-grade oil sand samples. The results obtained for different samples for the same grade oil sands exhibit a similar trend but an inherent spread in the measured data was observed.

Dielectric properties of oil sands are dependent on the amount of pore water content in the samples as well as on the pore distribution throughout the sample (Chute, Vermeulen et al. 1979). Dielectric constant and dissipation factor are sensitive to the changes in packing and pore structure within the sample. The expected variation in the measured dielectric properties can be explained by the non-homogenous behaviour of oil sands. Although the samples were prepared from the same source for a particular grade of oil sand, there existed a sample to sample variation in the moisture content and bulk density. The pellets made by using hydraulic press had different packing and thus, had altogether different pore distributions and microstructures. Thus, it is impossible to duplicate the pore distribution in different samples. A variation of 20-25% has been observed in the properties of the oil sand samples. The results shown for dielectric properties relate to each individual sample of different grades of oil sands. The dielectric properties obtained from a particular sample might not be the representative of the type of oil sand under observation. This is due to the differences in moisture content as well as pore distribution within samples. However, the mean values of dielectric properties over a frequency range can be considered to characterize a particular type of oil sand.

The dependence of dielectric properties on the frequency is apparent from the figures 5.11 and 5.12. Relative dielectric constant decreases as the frequency is increased from 100 Hz to 1 MHz in all three different grade oil sands. The decrease in dielectric constant is two to three orders of magnitude for each type of oil sands. It is noted that the low-grade oil sand has the dielectric constant value around $10^6$, followed by medium-grade oil sand with value around $10^4$. High-grade oil sand exhibits the value for dielectric constant i.e. around $10^6$. High dielectric constant of oil sands medium can arise due to many factors (Chute, Vermeulen et al. 1979, SWIECH, TAYLOR et al. 2013, Sen, Chew 1983) .Dissipation factor follows similar trend for all three grades of oil sands. It attains a maximum value at a particular frequency and then decreases as the frequency is increased.

As discussed in chapter two, oil sand is a complex mixture of sand, clay, fines, connate water and bitumen. The dielectric constants for water, bitumen and sand derived from the impedance analyzer and capacitive cell system are shown in the figures 5.13, 5.14 and 5.15. It can be seen that the dielectric constant of bitumen as well as sand remains almost constant even with the variation in the frequency, while it decreases in case of water.

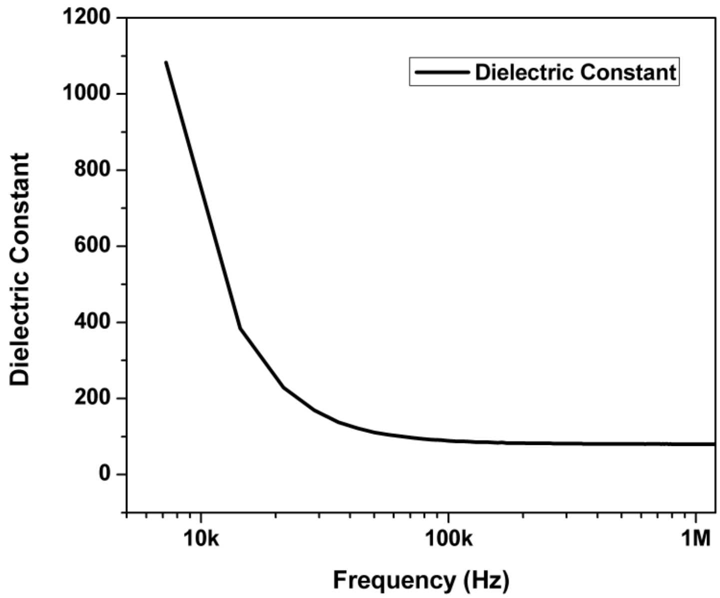



**Figure 5.13: Dielectric constant of water**

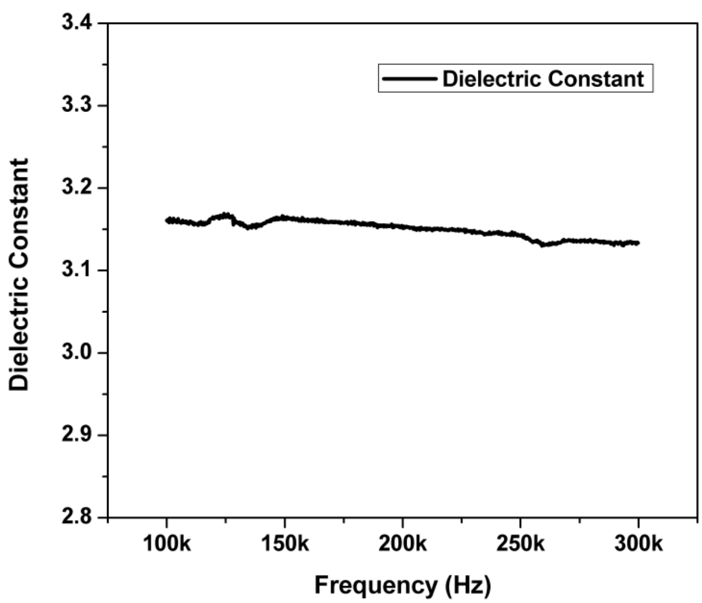



**Figure 5.14: Dielectric constant of bitumen**

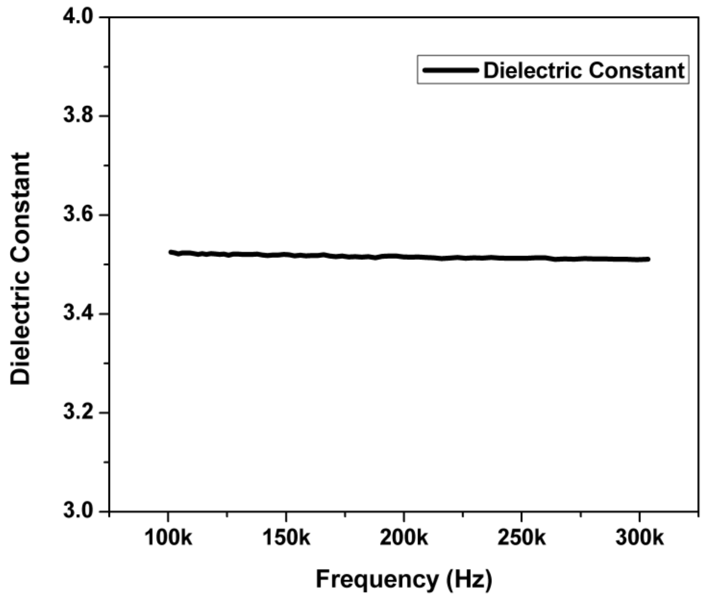



**Figure 5.15: Dielectric constant of sand**

At lower frequencies, dielectric constants for samples were found to be as high as $10^6$ as reported in Figure 5.1. Dielectric constant of sedimentary rocks is dependent on the salinity, frequency, water and clay content and textural effects. An attempt was made to summarize the dependence of dielectric properties on the parameters mentioned above. The polarization mechanism is considered to be the major contributing factor for frequency dependence of the dielectric constant. Different types of polarizations and its effect on dielectric constant is discussed further.

## 5.5 Frequency Dependence Mechanism

Figure 5.16 and 5.17 show the relative dielectric constant and loss factor variation with frequency ranging from 100 Hz to 1 MHz for medium-grade oil sand sample.

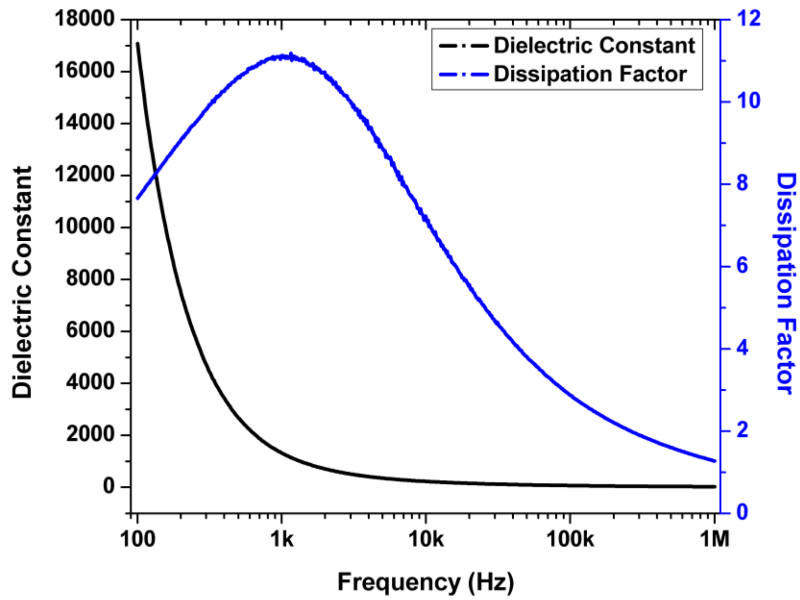



**Figure 5.16: Variation of dielectric constant and loss for medium-grade oil sand sample**

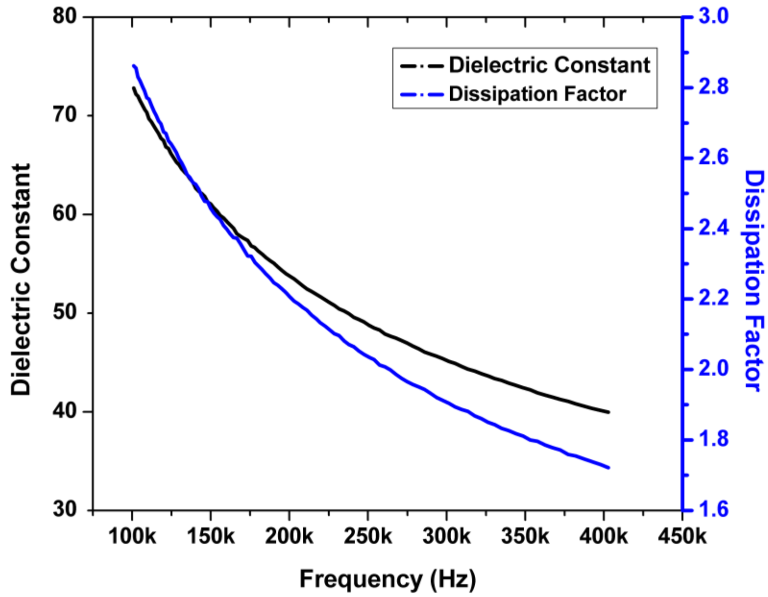



**Figure 5.17: Variation of dielectric properties for oil sand sample (100 kHz-400 kHz)**

## Polarization

The large values for dielectric constant can be explained by mechanisms such as interfacial polarization and electrochemical polarization (Chute, Vermeulen et al. 1979). Interfacial polarization is related to the accumulation of charge on the interface of two materials with different dielectric properties. Such polarization occurs at the sand-water interface in oil sands. Accumulation of charge on the interface leads to an increase in effective capacitance and in turn dielectric constant of the oil sand system. Another type of polarization is electrochemical polarization also known as Faradaic polarization (Grahame,1952). Electrochemical polarization comes into picture when charge crosses the interface between electrolyte and conducting material due to electrochemical reactions. These electrochemical reactions proceed only after a certain concentration of ions is reached through charge accumulation. This polarization is similar to interfacial polarization; the only difference is that the electrochemical polarization takes place within the bulk of the material and not on the electrode surfaces.

This accumulation of charge within the system results in increased dielectric constant of the system. These polarizations are dominant in lower frequency regime. As the frequency is increased charge accumulations via these mechanisms do not have enough time to build up across the interfaces. This results in decreased values of dielectric constant with an increase in frequency.

## Micrography and pore diffusion

Another source of frequency dependence is the geometrical arrangement of electrical elements in the material (Knight, 1984). Polarization characteristics are determined by the arrangement of accumulation of charges at the interfaces. The microscopic roughness at the interfaces changes the dielectric properties of the material.

The dielectric properties are also dependent on the diffusion of ions in the pores. Ion sieving polarization occurs whenever the diffusion and thus, mobility of ions through pores changes (Chute, Vermeulen et al. 1979). When the viscosity of water in the pores changes, the forces holding the water ions in the pores change too and this effectively reduces the mobility of ions. It leads to accumulation of charges at the interfaces and increase in the value of dielectric constant.

## Water Content

The presence of bound as well as free water also plays a major role in determining the dielectric properties of oil sands. Significant changes have been observed in the dielectric properties of oil sands with different water contents. Dielectric constant increases with increase in the water content in oil sand samples. It is noted that the low-grade oil sand has high water content and thus has large dielectric constant value. The dielectric constant for medium-grade oil sand samples has lower dielectric constant as the water content is low.

As discussed in chapter three, dissipation factor is defined as the ratio of energy lost to energy stored in the medium. Movement of ions to the interfaces in the oil sand samples is conduction which represents the energy loss in the system, whereas accumulation of charges at the interface leads to increase in the capacitance, which represents the energy storage in the system. Loss and storage components are inter-related. As more ions are conducted, more ions can be stored at the interfaces. Thus, it can be said that the storage is proportional to the energy loss in the system.

## 5.6 Medium-grade oil sand Analysis

### 5.6.1 Temperature Dependence: Dielectric Constant

Electromagnetic heating can prove to be an effective method for heating oil sands in the reservoirs. As the temperature of the system is increased, the dielectric properties of oil sand samples undergo some changes. A thorough understanding of the dielectric properties of oil sands will prove to be very useful in determining the electromagnetic heating behaviour. Experiments were carried out to study the dielectric properties of oil sands as a function of temperature over a frequency range 100 Hz – 1 MHz. The temperature was varied from 25$^{\circ}$C to 250$^{\circ}$C. Figure 5.18 illustrates the variation of dielectric constant of Athabasca oil sands with frequency at different temperatures.

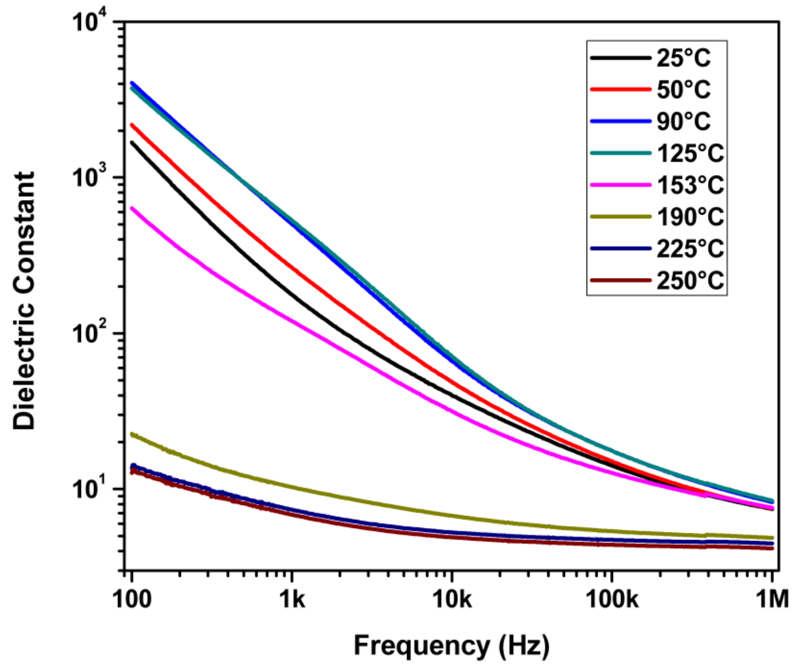



**Figure 5.18: Temperature dependence of dielectric constant of oil sands**

It can be seen that the frequency dependent curves at different temperatures show variation in the dielectric constant. The dielectric constants at a particular frequency increase from 25˚C to 125˚C and then decrease for the curve 153˚C to 250˚C. This suggests that the dielectric constant shows a maximum value for the curve of 125˚C. To investigate this property further the behaviour was studied for particular frequencies with the variation in temperatures from 25˚C to 250˚C.

Figure 5.19 represents the compilation of dielectric constant curves at frequencies i.e. 100 kHz, 200 kHz, 300 kHz and 1 MHz with temperature variation. Dielectric constant exhibits similar trend in all cases. A maximum for the dielectric constant was observed in the measured temperature range around 140°C.

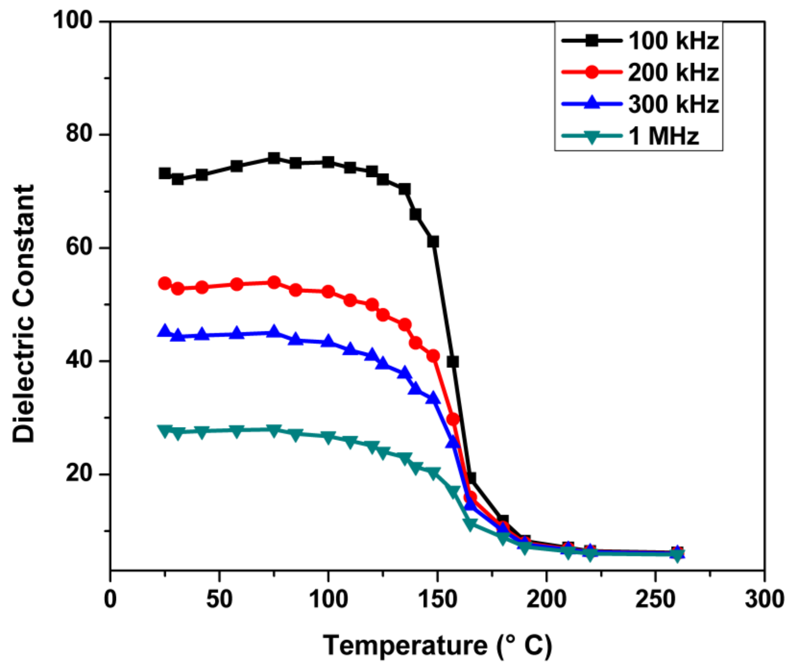



**Figure 5.19: Temperature dependence of dielectric constant**

The sharp increase in the dielectric constant at temperature T≅120℃ corresponds to the loss of water from the oil sand samples. The loss of water molecules from oil sand matrix eventually reduces the molecules which are free to orient along with the electric field. Thus, the dielectric constant is reduced after the loss of water from the oil sands samples.

### 5.6.2 Dissipation factor: Temperature Dependence

Figure 5.20 illustrates the variation of dissipation factor of medium-grade Athabasca oil sands with frequency at different temperatures. It can be seen that the frequency dependent curves at different temperatures show variation in the dielectric constant.

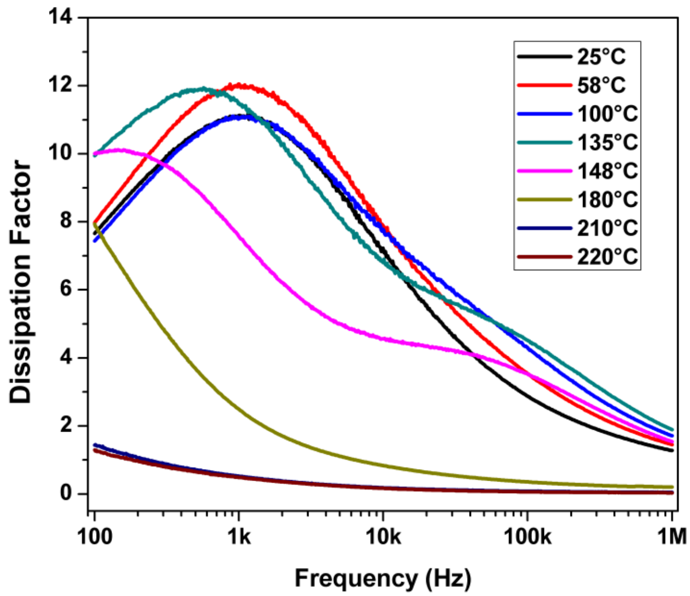



**Figure 5.20: Variation of dissipation factor at different temperatures**

It can be seen that the maximum dissipation factor shifts to a lower frequency region with the increase in the temperature. The curvature of the peak flattens as the peak shifts to lower frequency zone. To investigate the dependence of dissipation factor with temperature and frequency, experiments were performed for some particular frequencies. The temperature was varied from 25 °C to 250 °C and the dissipation factors were measured at frequencies 100 kHz, 200 kHz, 300 kHz and 1 MHz as shown in figure 5.21. It was found that the dissipation factor increased with an increase in temperature, and reached to a maximum value before decreasing. All the plots show the peaks in the dielectric constant values around 140 °C. The peaks in dissipation factors around temperature T≅130 °C corresponds to the loss of water from the oil sand samples. Dielectric constant as well as the dissipation factor exhibit the maxima in the values which correspond to the loss of water from the oil sands samples.

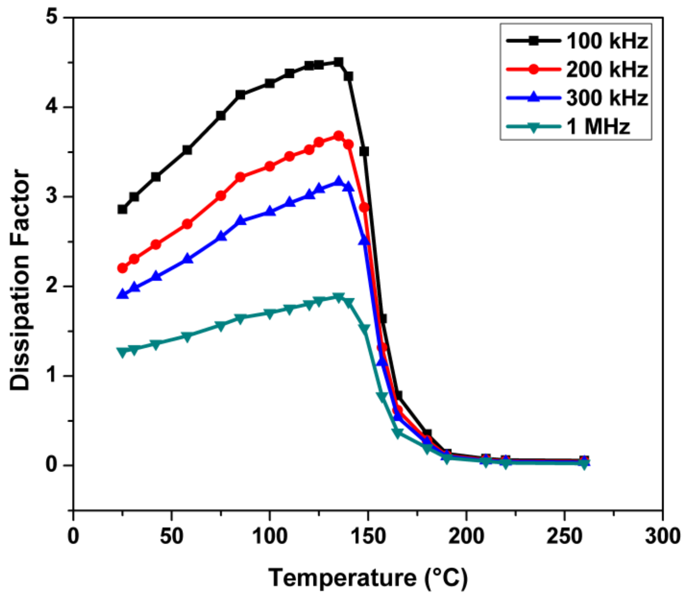



**Figure 5.21: Temperature dependence of dielectric constant**

## 5.7 High-grade oil sands Analysis

### 5.7.1 Frequency Dependence

Detailed discussion regarding the dielectric properties for high-grade oil sands is done in this section. The Dean Stark analysis of high-grade oil sand sample shows that it is comprised of 13-14% bitumen and 2-3% water by weight, with the remainder being fine-grained solids and fines. Figures 5.22 and 5.23 show the variation of dielectric constant and dissipation factor with frequency. The dielectric constant was found to be very high (around $10^6$ Hz) at lower frequencies which decreases at higher frequencies. The dissipation factor reached a maximum value around the frequency of 100 kHz. The frequency for maximum value of dissipation factor was found to be higher as compared to medium-grade oil sand sample.

The maximum frequency corresponds to maximum loss when an electric field is applied. Our electromagnetic system operates approximately around 200 kHz. This frequency is close to the maximum frequency, which may result in maximum heating of the oil sands medium. To get the numerical values experiments were conducted for the frequency ranging from 100 kHz to 400 kHz. The results for dielectric constant and for dissipation factors are compiled in the figure 5.22.

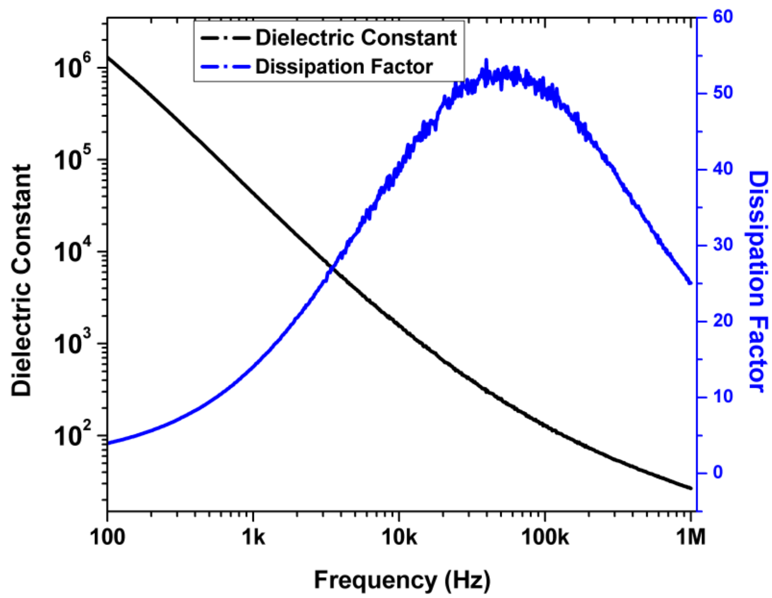



**Figure 5.22: Variation of dielectric constant and loss factor for high-grade oil sand**

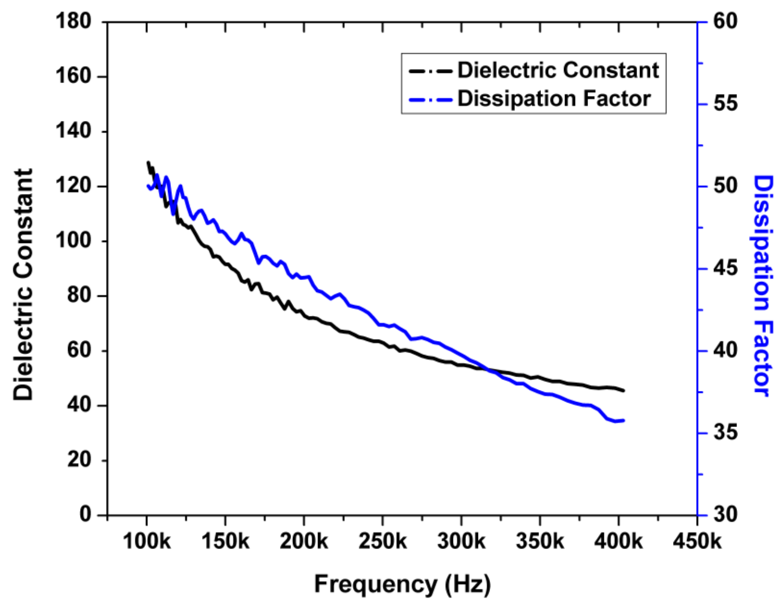



**Figure 5.23: Variation of dielectric constant and DF (100 kHz-400 kHz)**

### 5.7.2 Temperature Dependence

Experiments were conducted under similar conditions described in the previous section to study the temperature dependence of dielectric properties. Figure 5.24 represents the variation of dielectric constants with frequency at noted at different temperatures. In order to find out the variation of dielectric constant with temperature, different readings were taken for particular frequencies. The dielectric constant was found to have a maximum at temperature $T \cong 120^{\circ}C$ for all frequencies. It was found that the dielectric constant increase at lower frequencies was enormous. As the frequency was increased the value of dielectric constant was found to decrease as shown in figure 5.24.

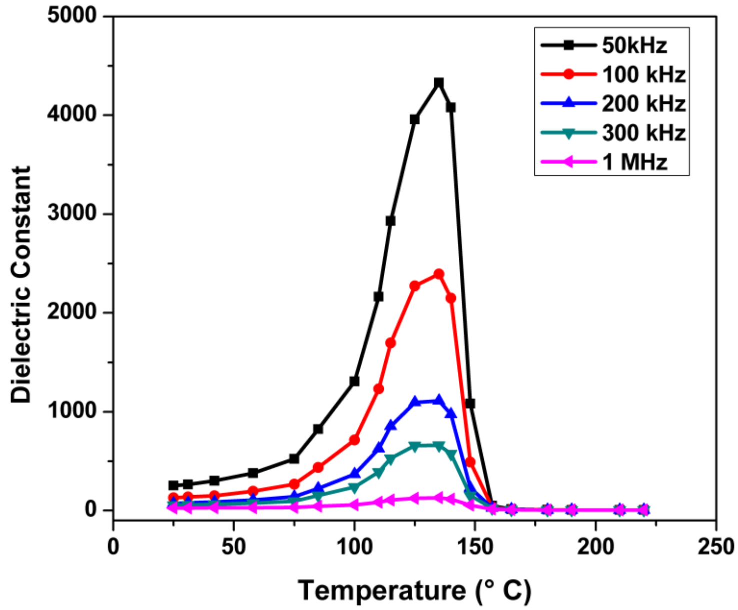



**Figure 5.24: Temperature dependence of dielectric constant**

The temperature dependence of dissipation factor is discussed in this section. Figure 5.25 shows the variation of dissipation factor with temperature for the frequency ranging from 100 Hz-1 MHz. As the temperature was increased from $25^0$C to $250^0$C, the dissipation factor decreased.

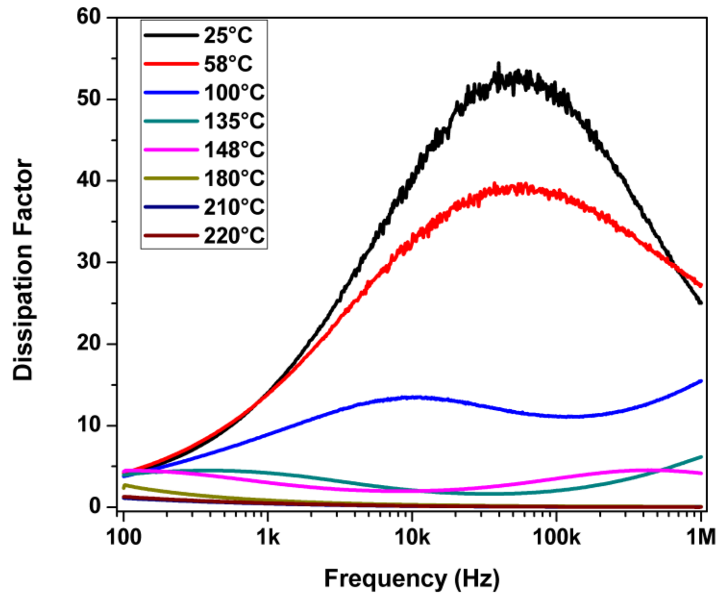



**Figure 5.25: Temperature dependence of dielectric constant of oil sands**

Figure 5.26 shows the compilation of the results obtained from experiments at higher frequency regions. The dissipation factor was found to have a decreasing trend with an increase in frequency.

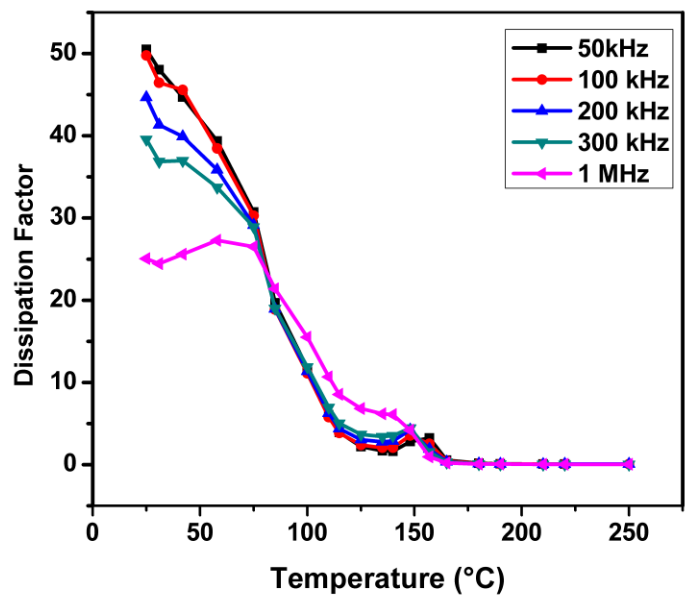



Figure 5.26: Temperature dependence of dissipation factor

## 5.8 Low-grade oil sands Analysis

### 5.8.1 Frequency Dependence

Dielectric properties for low-grade oil sand sample were obtained by impedance measurements over a frequency range 100 Hz-1 MHz. Representative data for dielectric constant and dissipation factor are shown in the figure 5.27. As discussed in the previous section, the dielectric constants had large values at lower frequencies and lower by order of 3-4 at higher frequencies. The maximum for dissipation factor was found around 100 kHz. The numerical values for dielectric constant and dissipation factor are found out by conducting experiments in the frequency region of 100 kHz- 400 kHz as shown in the figure 5.28.

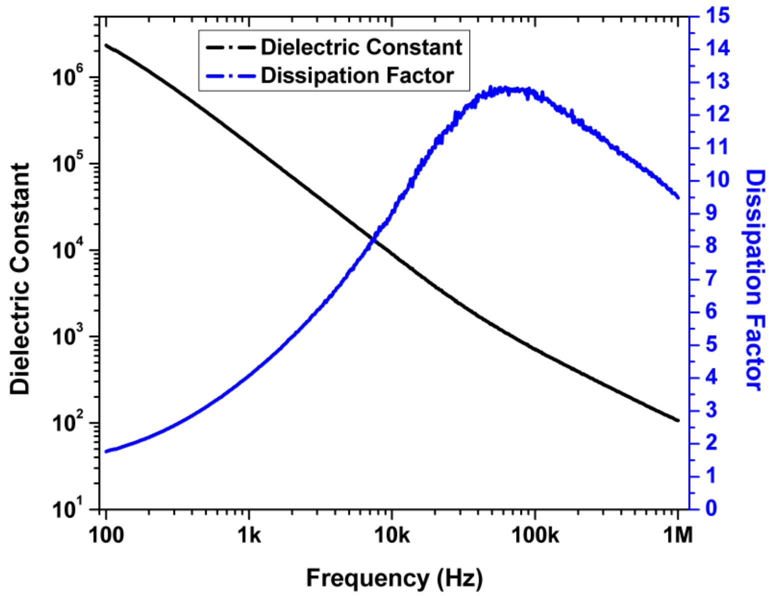



**Figure 5.27: Dielectric constant and loss factor variation with frequency**

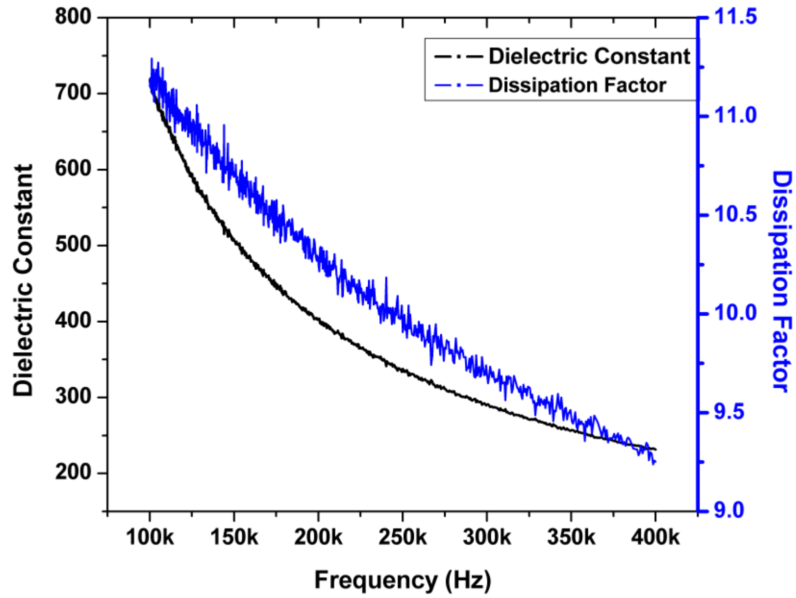



**Figure 5.28: Dielectric constant and loss factor variation (100 kHz-400 kHz)**

### 5.8.2 Temperature Dependence

Experiments were conducted under similar conditions described in the previous section to study the temperature dependence of dielectric properties. Figure 5.29 and 5.30 represent the variation of dielectric constants with frequency noted at different temperatures. In order to find out the variation of dielectric constant with temperature, different readings were taken for particular frequencies. The dielectric constant was found to have maximum value at different frequency.

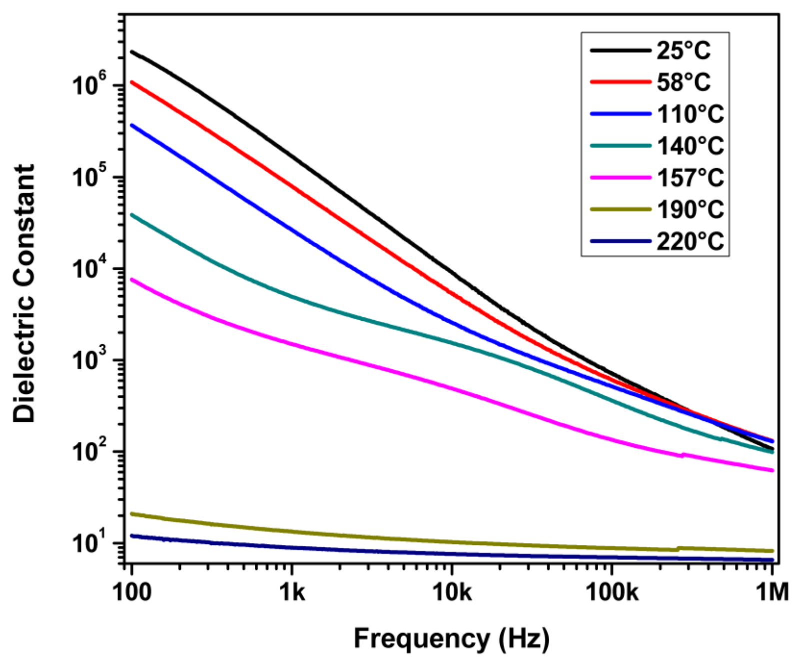



**Figure 5.29: Temperature dependence of dielectric constant of oil sands**

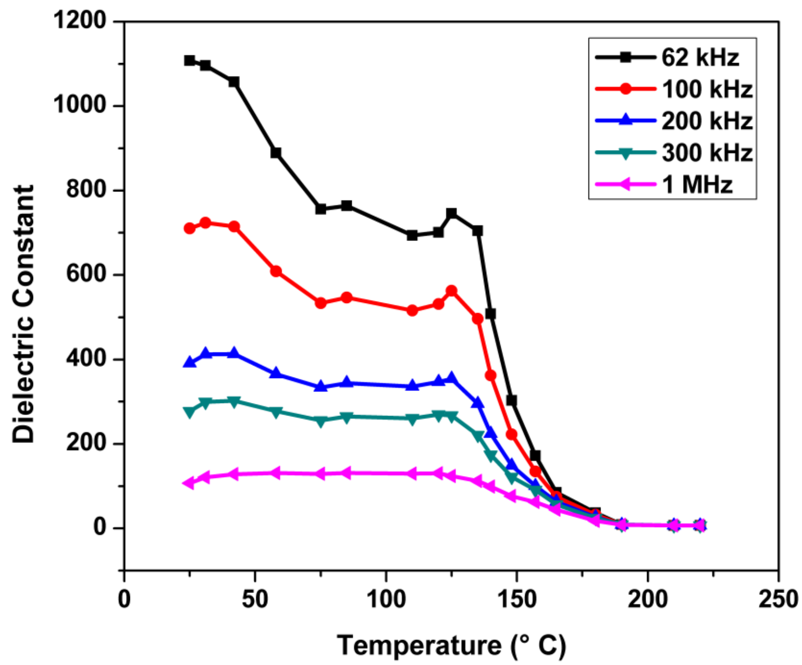



**Figure 5.30: Temperature dependence of dielectric constant (higher frequency)**

The experiments were conducted in the temperature range of $25^0$C-$250^0$C. The readings at the frequency (62 kHz) corresponding to the maximum dissipation factor were also taken. Dielectric constant curves at frequencies i.e. 62 kHz, 100 kHz, 200 kHz, 300 kHz and 1 MHz with temperature variation were also noted. Unlike high-grade and medium-grade oil sand samples, the dielectric constant was found to have a decreasing trend with increase in temperature. Similarly, the dissipation factor constant was found to have a decreasing trend with increase in temperature (See figure 5.31).

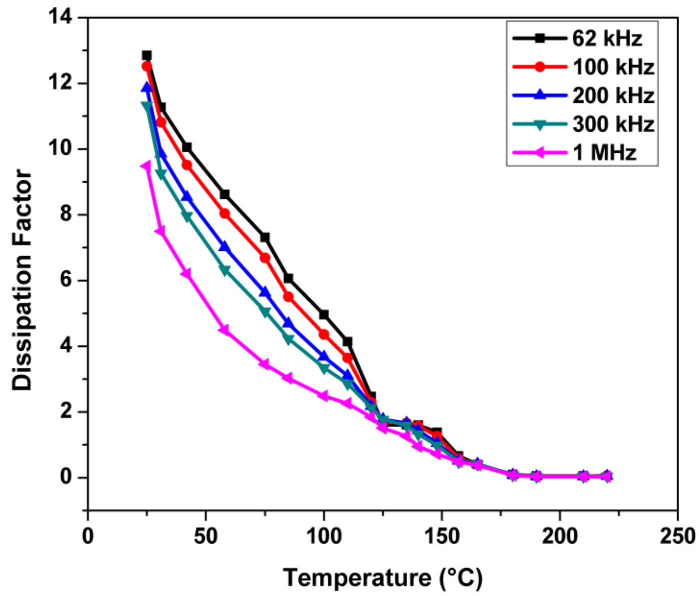



**Figure 5.31: Temperature dependence of dissipation factor**

The objective of this work is to characterize electrical properties of Athabasca oil Sands with variation in frequency as well as temperature. To find out dielectric properties, a set of experiments were performed for a frequency ranging from 100 Hz to 1 MHz with temperatures varying between $20^0$C - $250^0$C. The data obtained of oil sands will turn out to be useful in experimental as well as in modelling studies of electromagnetic heating.

# Chapter 6
# Conclusions and future work

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- Parallel plate capacitance method is used for measuring dielectric properties. Impedance parameters Z, θ, Cp, D were used to analyze the electrical properties of oil sands. The information extracted from the analysis will prove to be useful in understanding the electromagnetic heating system behavior for oil sands.
- Electrical properties of oil sands are dependent on various factors such as frequency, temperature, amount of pore water content, the pore distribution throughout the sample, interfacial polarization and the presence of ions in the sample.
- Variation of electrical properties with temperature has been studied. The behaviour of oil sands sample becomes more capacitive as temperature is increased.
- Dielectric constant for oil sand samples decrease with an increase in frequency. A maximum was observed for the dissipation factor for all three oil sands samples with an increase in the frequency.
- At lower frequencies, the dielectric constants for all three samples were found to be as high. High dielectric constants at low frequencies can be

explained with the induced polarization of the free ions at the interfaces of oil sands sample and electrodes. The parallel plate capacitor system was represented using the equivalent electrical circuit to understand the dielectric properties of oil sands, it is necessary to develop some understanding of the electrical response and represent the system using equivalent electrical circuit.

- The effect of temperature on the dielectric properties of oil sands was studied for different frequencies. Dissipation factor decreases as the temperature is increased and the position of the maxima also shifts to higher frequency.The peaks in dielectric constant as well as loss factor around temperature of $100^0$C correspond to the loss of water from the oil sand samples.

# Future work

- To find out the empirical relations for electric property estimations with variation in temperature as well as frequency.
- To come up with a mechanism for the dependence of dielectric properties of oil sands on interfacial polarization, salinity, the grain shape and composition for a better understanding of the reservoir rock behaviour.

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