2014

Development of impedance spectroscopy based in-situ, self-calibrating, on-board wireless sensor with inbuilt metamaterial inspired small antenna for constituent detection in multi-phase mixtures like soil

Gunjan Pandey
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Development of impedance spectroscopy based in-situ, self-calibrating, on-board sensor with inbuilt metamaterial inspired small antenna for constituent detection in multi-phase mixtures like soil

by

Gunjan Pandey

A dissertation submitted to the graduate faculty in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

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Iowa State University
Ames, Iowa
2014

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DEDICATION

I would like to dedicate this thesis to my wife Shambhavi without whose support I would not have been able to complete this work. I would also like to thank my friends and family for their loving guidance and support during the writing of this work.
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I would like to take this opportunity to express my thanks to those who helped me with various aspects of conducting research and the writing of this thesis. First and foremost, Dr. Ratnesh Kumar for his guidance, patience and support throughout this research and the writing of this thesis. His insights and words of encouragement have often inspired me and renewed my hopes for completing my graduate education. I would also like to thank my co-advisor Dr. Robert J. Weber for his efforts and contributions to this work. Work would not have been complete without his insights into the intricacies of RF, microwave and antenna design. Sincere thanks to all my dear friends in Ames and fellow graduate students at Iowa state university for their continuous encouragement and support.
ABSTRACT

Real time and accurate measurement of sub-surface soil moisture and nutrients is critical for agricultural and environmental studies. This work presents a novel on-board solution for a robust, accurate and self-calibrating soil moisture and nutrient sensor with inbuilt wireless transmission and reception capability that makes it ideally suited to act as a node in a network spread over a large area. The sensor works on the principle of soil impedance measurement by comparing the amplitude and phase of signals incident on and reflected from the soil in proximity of the sensor. The permittivity of the soil dielectric mixture which is calculated from these impedance measurements is used as input parameter to the dielectric mixing models which are used to estimate the ionic concentration in soil. The inbuilt wireless transceiver system is connected to a specially designed metamaterial inspired small antenna in order to reduce the sensor size while keeping the path losses to a minimum by using a low frequency. This composite right-left handed (CRLH) antenna for wireless transmission at 433 MHz doubles up as an underground, sensing element (external capacitor) and integrates with the on-board sensor for soil moisture and nutrient determination. The input impedance of the CRLH sensor, surrounded by the soil containing moisture and nutrient and other ions, is measured at multiple frequencies. It is shown that the change in moisture and ionic-concentration can be successfully detected using the sensor. The inbuilt self-calibrating mechanism makes the sensor reliable at different environmental conditions and also useful for remote, underground and hand-held applications. A multi-power mode transceiver system has been designed to support the implementation of an energy efficient medium-access-control.
CHAPTER 1. OVERVIEW

Dielectric spectroscopy is the measurement of the dielectric properties of a medium as a function of frequency. The change in dielectric properties like resistivity, conductivity, real and imaginary permittivity indicate the change in concentration of constituents present in a mixture of dielectric materials. Hence, an accurate measurement of these properties is very much desirable in order to detect the presence as well as to estimate the concentration of different constituents of a dielectric mixture. The dielectric properties, however, are not directly measurable. For instance, permittivity of a material itself cannot be measured but a measurement of the capacitance of an ideal parallel plate capacitor with this material as the dielectric between the plates can be easily measured. Similarity, conductivity can be derived from conductance and resistivity from resistance. Other more complex methods may involve measuring the propagation constant and attenuation factor of an electromagnetic wave propagating through a medium and using these values to derive the real and imaginary parts of the permittivity of the medium. In general, impedance measurement can provide information about real and imaginary parts of the permittivity of a medium. For example, for an ideal parallel plate lossless capacitor, impedance will provide information about the capacitance which can be used to derive the real permittivity of the dielectric between the plates. Similarly, if the dielectric between the plates is made up of lossy material, it will have a finite conductance apart from capacitance which can be used to derive the imaginary part of the permittivity of that dielectric material.

Detection and estimation of individual constituents of a dielectric mixture has immense applications. In precision agriculture, a major impediment is the ability to adequately describe the spatial/temporal variation of important physical parameters such as moisture and nutrients. There is a critical need for the development of technologies in production agriculture that
allow automated collection of real-time spatial soil and crop data while advancing a deeper understanding of the fertilizer inputs and Nitrogen (N) cycling. Such detection of nitrates will prove beneficial for the environment by helping to reduce the amount of nitrate leaching to the underground water table. Ability to detect multiple ions will help regulate other harmful ions such as chlorides, potassium etc. as well.

Apart from ionic detection in agriculture, spectral dielectric sensing also has the potential to detect pathogens and undesired microbial growth whose presence changes the electric properties of the sample. Such spectroscopic dielectric sensing has been shown to be useful in detecting the presence of bacteria such as E-coli after incubation with appropriate anti-microbial peptides. Micro-capacitive sensors based on similar principle have shown direct proportionality between impedance changes due to change in the dielectric properties and the extent of analyte binding occurring on the surface of an electrode.

Above examples are some of the many applications of dielectric spectroscopy. Although dielectric spectroscopy has been known and applied to various areas in the past, in-situ sensors to detect as well as estimate the concentration of constituents in a dielectric mixture in real time have not yet been realized.

From the previous discussion we can infer three main directions in the development of sensors for dielectric spectroscopy. One, development of a sensor architecture that can measure the impedance of the sensor inserted in the medium surrounding the sensor at multiple frequencies; two, a method to determine the permittivity of the medium given the impedance value and three, a model that can use these permittivity values at multiple frequencies and estimate the concentration of the constituents of the dielectric mixture.

This research focuses on estimating moisture and ionic concentrations in soil using dielectric spectroscopy. An on-board sensor architecture for measurement of impedance has been developed and fabricated. The sensor has shown accurate results that are comparable to that of a bench-top Network Analyzer for low radio-frequencies. We have also performed experiments which indicate that nitrate detection in soil is possible using multi-frequency impedance measurement combined with dielectric mixing models that relate the permittivity of a dielectric mixture to permittivity and concentration on individual constituents.
To make the sensors more useful for use in underground precision agriculture, it has been endowed with certain additional features to make it more useful for underground and remote operations. Accuracy of measurements is enhanced by considering a distributed transmission line model for the on-board connections. Presence of an inbuilt self-calibrating mechanism which operates on the standard short-open-load (SOL) technique makes the sensor independent of inaccuracies that may occur due to variations in temperature and surroundings. Moreover, to minimize errors, the parasitic impedances of the board are taken into account in the measurements. Measurements of both real and imaginary parts of soil impedance at multiple frequencies gives the sensor an ability to detect variations in ionic concentrations other than soil moisture content. A switch-controlled multiple power mode transmission and reception is provided to support highly energy efficient medium access control. The sensor has an inbuilt transmission system connected to an antenna which can transmit data to another node in a large network of similar sensors. Design of such antenna which can efficiently work underground and is small in size is another aspect of this research and has been discussed in later sections.

To estimate the soil ionic concentration, multi-frequency impedance measurements have been combined with the quasi-static dielectric mixing models to infer the various ionic concentrations. In our approach, the permittivity of the soil dielectric mixture is measured using impedance spectroscopy and the results are used as input parameters to dielectric mixing models, combined with the debye-type dielectric relaxation models. We observe that the dielectric mixing models work well for low RF (radio-frequency) range and help in determining the individual ionic concentration in a multi-component soil mixture. Using the fact that the permittivity of a dielectric mixture is proportional to its impedance, we validated our approach by making multi-frequency impedance measurements of a soil mixture at different concentrations of various components. The method provides a good estimate of individual components such as air, water and ions like nitrates. While the research work has been done with the perspective of soil constituent concentration determination, the underlying principle of determining individual component concentration using multi-frequency impedance measurement is applicable more generally in areas such as characterizing biological systems like pathogens, quality control of pharmaceuticals etc.
1.1 Introduction

Efficient management of agricultural resources for increased productivity and minimum environmental impact forms the basis of precision agriculture. In a generic precision agriculture layout (see Figure 1.1), intra- and inter-field variabilities are characterized using a network of sensor nodes spread over a large area. Each sensor node sends local information about the properties of the soil surrounding it. All the information collected is sent to a central node which processes the information and takes necessary measures like irrigation and fertilization. Thus, each node in this sensor network not only measures the soil content accurately in real time, it also communicates efficiently with other nodes in the network in order to transmit the collected information.

Our work aims at developing an on-board solution for a sensor which can measure local soil conditions like water as well as ion content with high accuracy. It will have the capability to sweep through a frequency range to improve the data reliability of moisture and ionic content measurements. To achieve high accuracy and robustness of measurements, the sensor has a built-in self-calibrating system which is based on embedded measurements of open, short and matched load conditions. For efficient transmission, a built-in multi-power mode is introduced in transmission and reception. Such system has been shown to support more energy-efficient medium-access-control (MAC) protocol Sahota et al. (2011). The main contributions of this research can be summarized as:

1. The sensor has the capability to measure impedance at multiple frequencies. This improves accuracy and also gives the sensor the ability to detect multiple ions Chighladze et al. (2011).
2. The sensor has a built-in self-calibration system which makes them impervious to the variations in soil temperature and climatic conditions like hail, drought, rain etc.
3. The sensor is designed for in-situ underground operation through an in-built antenna and wireless transceiver, so as not to interfere with the above ground operations. To economize on size, the sensor probes are diplexed to act as antenna.
4. The built-in transceiver has multiple power modes for transmitter/receiver to support a highly energy efficient MAC (Medium Access Control) Sahota et al. (2011).
A model for accurate determination of nitrate detection in soil is developed to extract the nitrate content information from the permittivity measurements.

Spectral dielectric sensing also has the potential to detect pathogens and undesired microbial growth whose presence changes the electric properties of the sample. Such spectroscopic dielectric sensing has been shown to be useful in detecting the presence of bacteria such as E-coli Mannoor et al. (2010) after incubation with appropriate anti-microbial peptides (AMP). Microcapacitive sensors based on similar principle have shown direct proportionality between impedance changes due to change in the dielectric properties and the extent of analyte binding occurring on the surface of an electrode Berggren et al. (2001).

1.1.1 Soil Moisture and Impedance Sensors

Many attempts have been made to accurately measure varying moisture levels in the soil. Notable approaches found in literature for soil-moisture measurement include thermal sensors Yamamoto and Ogawa (2002), neutron probe sensors Watson et al. (1996), granular matrix/gypsum block sensors Irmak (2006), TDR/FDR based impedance measuring electronic sensors Gaskin and Miller (1996), Will and Gerding (2009), Robinson et al. (2003), Majid et al. (). Some of them are discussed here.

Thermal sensors determine the properties of the soil using the thermal properties like con-
duction and radiation. They are based on the principle that soil thermal properties vary with change in the moisture level and hence a measure of moisture content can be obtained by observing the change in these properties. Thermal Sensors Yamamoto and Ogawa (2002) determine moisture content using these two methods (heat conduction and heat radiation). The heat conduction method uses a heater and temperature sensor separated by a distance and the temperature change in the soil is measured to determine the amount of moisture present. Heat radiation method uses a heater and two thermo couples-one of which is attached to the radiation plate connected to the heater and second is placed on a patch attached to an aluminum plate kept at a distance from the heater. The temperature curves of thermocouples are then observed to see the thermal characteristic of the medium. Another thermal pulse sensor Valente et al. (2010) generates heat pulse whose durations and magnitude is controlled by a microprocessor. This heat pulse changes the voltage across the thermistor. The change in voltage across the thermistor is related to the moisture content by using the empirical relations Kamai et al. (2009). One inherent disadvantage of using a thermal sensor is that the installed heater can affect the moisture concentration around the sensor node causing evaporation and hence accurate measure of soil content may not be determined. Also, due to the agricultural activities carried out in the soil, the thermal properties might change due to factors other than moisture. Some examples can be addition of fertilizers and loosening of soil due to ploughing. Hence a periodic calibration is needed.

Another approach used to measure soil moisture is to use a satellite system which can remotely measure the moisture content by GNSS-R (Global navigation satellite system reflection) signal Songhua et al. (2010). Each sensor node has two antennae one of which points to the ground and other to the satellite. The sensor measures the reflectivity of the soil, which primarily depends on the moisture content. This information is sent to the satellite and a global data can be collected through a series of such sensor nodes. Since the nodes are placed above the ground and the depth to which a wave can travel is limited by the losses in the soil, this method does not necessarily provide actual moisture level which is available to the roots of the crops. GPR (ground penetration radar) based method is also used for soil sensing Benedetto and Benedetto (2011). Using the Debye model for relaxation time of water molecules it is
argued that the amount of water will affect the scattering characteristics of the signal reflected from the ground. Hence, the frequency spectrum of the received signal will give an indication of the water content present in the soil. Satellite based sensors can provide good accuracy, but spatial resolution is limited.

A granular matrix based approach to the soil moisture sensing has been described in literature Irmak (2006). The sensor operates on the electrical resistance principle and is made of porous ceramic material as external shell. Two electrodes are inserted in the internal matrix. As water content in the soil varies, the amount of water seeping in through the porous shell also changes. This changes the electrical resistance between two electrodes which can be monitored. Sensor contains a wafer of gypsum to protect the electrodes against salinity in the soil. An NIR (Near Infra-Red) spectrometer Mouazena et al. (2005) is another type of sensor. The spectrophotometer is attached to the back of the subsoiler chisel to perform light reflectance measurement from the soil surface. The calibration is done under laboratory condition. This method is highly expensive due to sophisticated components involved. Also the measurement is done only while using the chisel which may not be required at all times.

A neutron probe based soil sensor has also been developed Watson et al. (1996). The authors provide computer modeling and testing for the neutron sensor which will work for top 15 cm of soil. In a neutron probe sensor, fast neutrons are emitted from a decaying source into the soil where they bounce around and gradually slow down in the process mainly due to collisions with the hydrogen nuclei in the surroundings. Thermal and epithermal probes detect a fraction of these moderated neutrons depending on the concentration of hydrogen nuclei in the surroundings. In most soils, the only source of hydrogen would be water which means that the slowing down of fast neutrons would be due to water. These sensors, although very accurate, are more suited for reactor sites where radioactivity is not a problem. These are expensive due to cost of neutron probe and detector.

Impedance Sensors are a common type of sensors which have the potential to get rid of most of the inherent disadvantages which are present in thermal, granular matrix, neutron probes and remote no-contact sensors. Some of the advantages offered by impedance sensors are their ability to make measurements in real time, ease of sensor calibration, no effect of sensor on
surrounding soil properties, no interference of sensor in agricultural processes, accuracy and ease of measurement. Moreover, impedance sensors can naturally form a part of a circuit which can include a transceiver and hence they can easily be embedded into a network. This further means that they are controllable from a remote station. These sensors can be calibrated to work for different types of soil and they work for large range of moisture content. The downside of impedance sensors is increased complexity. Moreover, all electrical circuits come with a certain amount of noise which affects accuracy. Many different types of impedance sensors are present in the literature. Fringing electric fields have been used Manut and Firdaus (2009) to determine the permittivity of soil under observation. The experimental setup discussed uses a fringing capacitance followed by the FEM (finite element method) analysis to relate capacitance with permittivity. Variations in moisture content changes the permittivity which in turn changes the capacitance that can be detected. The setup lacks a self-calibration algorithm and requires measuring capacitance and permittivity for known moisture content from time to time. This is not practical specially for a large network of buried autonomous sensor nodes. The study has not been extended to analyzing nutrients besides moisture.

Another frequency domain approach has been suggested based on capacitive sensing Majid et al. (). Like in previous discussion Manut and Firdaus (2009), a fringing field capacitance is used to project the sensing electric field into the surrounding material. An AC (Alternating Current) signal applied to this capacitor will shift its phase depending on equivalent RC model of capacitance. The phase shift has been shown to be proportional to $\sin^{-1}$ of the capacitance value. The driving signals follows two paths- one direct and another through soil container- to the phase detector. It is assumed that the only phase shift will occur in the soil containing capacitor which is a fair assumption given proper fabrication is done. Thus phase shift can be used to find capacitance which in turn can be used to determine dielectric constant of soil contained in the capacitive cell. Since, different frequencies will result in different phase change across the capacitor, calibration is needed every time the frequency is changed. Hence a multi-frequency implementation of this work becomes more complicated. FDR approach has also been used Balendonck and Hilhorst (2001) to develop an ASIC (Application Specific Integrated Circuit) to measure the capacitance which has been mapped to permittivity and
soil moisture. The sensor shows good accuracy and permittivity resolution.

Time Domain Reflectometry (TDR) has inspired many past and ongoing sensor designs. The basic idea is to direct a square pulse towards a soil sample and calculate the coefficient of reflection at the surface from which the wave gets reflected. The reflection coefficient along with the characteristic impedance of the line gives the impedance of the surface at which reflection takes place. Creation of the dielectric profile for the soil using TDR has been discussed in literature Will and Gerding (2009). For the measurement of spatially resolved dielectric profiles by using delay time measurements, a transmission line is used. The delay time of an electromagnetic pulse along the transmission line is measured with the help of an industrial TDR system operating in the baseband up to 3 GHz with a pulse width of 300 ps. A phase-shift based approach for finding unknown impedance using TDR has also been discussed Xing et al. (2005). Phase shift in traveling wave along the length of transmission is proportional to the square root of permittivity. Thus knowing the phase shift gives the value of permittivity of the medium. In situ application of this method requires a high cost of TDR system. TDR method has additional drawbacks such as problems with extracting accurate parameters from the received waveforms, difficulties in detecting the reflected signal in saline soils, and dependence of measurement on the coaxial cable and probe lengths Benedetto and Benedetto (2011).

To summarize, there is a definitive need for a sensor system which gives accurate results at multiple frequencies, is self-calibrating, can be part of a network, consumes small amount of power and is relatively less expensive. Our approach involves reflection as in TDR systems, but instead of measuring the time delay or phase shift we measure the amplitude and phase of incident and reflected waves. This is beneficial for real time sensing as the surface under observation can be outside of the system unlike capacitive sensorsMajid et al. () which needs waves to travel through the conductor contained in the line. Our system has the capability to make measurements at multiple frequencies and is self-calibrating which makes it more robust, more accurate and low maintenance. Like capacitive measurement schemes used in Manut and Firdaus (2009) and Majid et al. (), our system measures unknown impedance. Its measurements are based on reflectometry allowing us to make a direct correlation between impedance and permittivity. The use of transmission line model allows us to include line losses and calibrate the
system periodically. Due to multi-frequency approach, our sensor has the capability to detect and transmit information about soil moisture as well as ionic concentration Chighladze et al. (2011). Using de-embedding techniques, some of the disadvantages related to TDR sensors like inaccurate measurements due to probe length have been removed.

1.1.2 Soil moisture and nitrate detection

Determination of accurate ionic concentrations in a dielectric mixture is a long standing research problem. One of the most important applications of ionic concentration determination is the estimation of soil nitrate concentration in an agricultural setting. Accurate estimation of nitrates can provide great improvements in agricultural production by helping to regulate nitrate management. It can hugely benefit the environment by minimizing the amount of nitrate leeching to the underground water table.

In general, soil is comprised of a number of components. Main amongst them are Soil bulk, moisture, ions and trapped air molecules. Moisture itself is further divided into two parts - Bound and Bulk moisture Santamarina et al. (2001) deLooor (1968). Bound moisture is the moisture which is closest to the soil particles and forms a layer around them called the Stern Layer. In the Stern layer, water is not free to move and its diffusivity and polarizability are not same as those of free water. At some distance from the soil particles, water is present as free flowing liquid. This layer is known as Gouy layer and possesses properties of normal liquid water. Due to the presence of water, salts tend to ionize and hence conductivity of the soil is increased. The conductivity is directly proportional to the dissociation constant of salt-water equilibrium reaction. Conductivity increases with valency of cation and anion and with the size of the ion. Another important property of these ionized salts is that both the cation and anion tend to hydrate themselves. This means that the total ionic radius includes the size of the ion along with the size of water molecules which are attached to this free ion. Ions with larger hydrated radius have lesser mobility and result in lesser contribution towards overall conductivity.

Dielectric mixing models are used to determine the permittivity of a mixture in terms of the permittivity and concentration of its constituents. Many dielectric mixing models such as
Maxwell-Garnett, Bruggeman Sihvola (1999) and deLoor’s mixing rule Dobson and Hallikainen (1985) have been shown to be efficient in detecting changes in soil moisture content given that the properties of other constituents like soil bulk, trapped air and bound and bulk water are known. Usually bulk water is treated as free water, while bound water can be assumed to have the electrical properties of snow. This assumption has been validated by making accurate measurements of soil moisture using the dielectric measurements Dobson and Hallikainen (1985). Empirical model have also been developed Wang and Schmugge (1980) to relate the dielectric constant with varying moisture content in the soil. It has been demonstrated in this study that a combination of dielectric properties of ice, water, air and rock can describe the dielectric behavior of soil-water mixture at 1.4 GHz and 5 GHz. Models for moist soils based on variations in soil complex refractive index with moisture have also been developed Mironov et al. (2004). In a more recent work, the dielectric properties of saline deposits are used for mapping of moisture content Lasne (2007). Most work in soil research has been focussed on soil moisture determination and little has been done so far to determine the concentration of ions such as nitrates, chlorides etc.

Time domain reflectometry (TDR) is one of the main approaches for making multi frequency impedance measurements. In this method a square signal pulse of known properties is reflected from an unknown surface and time difference between the transmitted and received waves is used to determine the surface impedance. TDR approach has been extended to detect nitrate and chloride ions in soil as well. In one such study Krishnapillaia and Ranjanb (2009), authors present an in-laboratory method to detect nitrate concentration. A potassium nitrate solution of known concentration of 500ppm was flown through the TDR sensor contained in the soil inside a plexiglass cell. Nitrate concentration was changed with time and the TDR probes were placed at regular intervals along the flow cell. The bulk soil electrical conductivity and the water content values extracted from the TDR wave forms were used to predict the nitrate concentrations at different locations. The nitrate concentration values predicted from the TDR-measured bulk electrical conductivity and water content data were observed to correlate with the nitrate concentrations obtained by soil solution sampling method. In another study Chighladze et al. (2011), the variation of soil permittivity and soil conductivity within
the frequency range 200 Hz to 13 MHz is presented. It is shown that the TDR approach can be used to effectively detect nitrate concentration by measuring the soil permittivity and conductivity. The potential of using TDR to simultaneously estimate volumetric soil water content, soil solution electrical conductivity, and soil nitrate/nitrogen concentration in an irrigated peppermint field has also been demonstrated Das et al. (1999). This work compares TDR-estimated nitrate/nitrogen concentration with the estimates obtained from direct soil measurements (soil cores and soil solution samples). It was observed that the estimates from all methods were comparable and had similar pattern, magnitude and variance. The work done on feasibility of using TDR to monitor changes in nitrate and nitrogen concentration in an irrigated agricultural soil Payero et al. (2006) concluded that the TDR probes could be used to measure nitrate/nitrogen concentration in non-saline soils and water after the proper calibration over a long enough period of time. Calibration is needed to include the expected variations in volumetric water content (VWC), temperature and nitrate/nitrogen concentration. In another study Neve et al. (2000) Neve et al. (2003), the effect of temperature on the extent of nitrogen mineralization in the soil due to various processes like microbial activity has been studied. This work also determines the feasibility of employing TDR based conductivity measurement to make these observations. It was observed that the mineralization of nitrogen results in a measurable increase in the electrical conductivity and hence nitrogen concentration could be monitored using the TDR probe.

We determine the concentration of ions in a multi-component mixture like soil using impedance spectroscopy. The need for multiple frequency measurements arises because from a single frequency measurement, concentration of more than one unknown ion cannot be determined, regardless of the accuracy of the measurements. Since all parameters of each component are known except the concentrations, for a host with $n$-components, measurements at atleast $n$ frequencies give adequate information to infer the $n$ unknown concentrations. If there are more than $n$ measurements, then least square methods are used to find the “best” fitting set of unknown concentrations that minimize the squared-error of the fit.
CHAPTER 2. Design and Implementation of Impedance Measurement System

The problem of measuring soil moisture content can be broken down broadly into three steps:
1. Measuring the unknown impedance of sensor which is embedded in the soil whose properties are to be determined;
2. Calculating the permittivity of soil using the measured impedance value;
3. Determining the soil moisture content from the permittivity values.

If a transmission line with characteristic impedance $Z_0$ is terminated in a load impedance $Z_L$, then the coefficient of reflection is defined as Weber (2001):

$$\Gamma_L = \frac{V_r}{V_i} = \frac{Z_L - Z_0}{Z_L + Z_0}. \quad (2.1)$$

$V_r$ and $V_i$ are signals reflected from and incident upon the load impedance respectively (see Figure 2.1). For a micro-strip transmission line, $Z_0$ is a constant and depends on the width of the transmission line and permittivity of the pcb substrate. We have designed $Z_0$ to be 50 ohms. Thus, if we make accurate measurements of $V_r$ and $V_i$, we can calculate the value of $Z_L$. To measure $V_r$ and $V_i$, we need to measure amplitudes and phases of $V_r$ and $V_i$. To make these measurements, incident and reflected signals, which are on the same transmission line, have to be separated. This can be done efficiently using a pair of directional couplers. A directional coupler is a device that can be used to couple a small fraction of the signal flowing in a particular direction on a transmission line to its output port. The signal flowing in other direction is not coupled. Two directional couplers are connected to the main line as shown in Figure 2.1. One coupler couples the incident signal, while the other couples the reflected signal to its output port.
Figure 2.1  Directional Coupling of incident and reflected signals.

Since on-board couplers are not point objects, there always is a finite distance between point at which load is connected to the line and the point where signal coupling takes place. This means that the coupled signals at ports 3 and 4 are gain/phase shifted relative to the incident signal, $V_i$ and reflected signal, $V_r$ respectively. Since this shift depends on the frequency and the length of the transmission line between the coupler and the load impedance, this shift can be accounted for by proper calibration. In order to find a relationship between the ratio of reflected and incident signals ($\Gamma_L = \frac{V_r}{V_i}$) at the load and the ratio of coupled reflected and incident signals at the ports 4 versus 3 ($\Gamma_m = \frac{V_{o4}}{V_{o3}}$), we treat the combined system of two couplers and the transmission line as a four port network, with the ports numbered as shown in Figure 2.1, where the source is port 1 and the load is port 2; port 3 couples with the incident signal and port 4 couples with the reflected signal. Owing to the fact that the outputs and inputs of such a 4-port network are linearly related, it has been shown Weber (2001) that the measured value of reflection coefficient, $\Gamma_m$ and the reflection coefficient at the load, $\Gamma_L$ are related by a bilinear transformation:

$$\Gamma_m = \frac{V_{o4}}{V_{o3}} = \frac{a \Gamma_L + b}{c \Gamma_L + 1} \quad (2.2)$$

where $a$, $b$ and $c$ are constants for this 4-port network while $V_{o4}$ and $V_{o3}$ are output voltage signals from port 4 and port 3 respectively. This implies that 3 measurements of $\Gamma_m$ at three different loads (or equivalently three different $\Gamma_L$’s) will give us 3 equations in 3 unknown calibration constants $a$, $b$ and $c$. For ease of calculation we can chose these three known load impedances.
Figure 2.2 Quadrature Demodulator is used to calculate Signal Amplitude and Phase.

to be $\infty$ (open-circuit), 0 (short-circuit) and $Z_0$ (matched load), with the corresponding load reflection coefficients $\Gamma_L$ being 1,-1 and 0 respectively. The equations in 3 unknowns can be solved by solving the matrix:

$$
\begin{bmatrix}
\Gamma_{L1} & 1 & -\Gamma_{L1}\Gamma_{m1} \\
\Gamma_{L2} & 1 & -\Gamma_{L2}\Gamma_{m2} \\
\Gamma_{L3} & 1 & -\Gamma_{L3}\Gamma_{m3}
\end{bmatrix}
\begin{bmatrix}
a \\
b \\
c
\end{bmatrix}
= 
\begin{bmatrix}
\Gamma_{m1} \\
\Gamma_{m2} \\
\Gamma_{m3}
\end{bmatrix}.
$$

The constants $a, b, c$ can then be calculated using:

$$
\begin{bmatrix}
a \\
b \\
c
\end{bmatrix}
= 
\begin{bmatrix}
\Gamma_{L1} & 1 & -\Gamma_{L1}\Gamma_{m1} \\
\Gamma_{L2} & 1 & -\Gamma_{L2}\Gamma_{m2} \\
\Gamma_{L3} & 1 & -\Gamma_{L3}\Gamma_{m3}
\end{bmatrix}^{-1}
\begin{bmatrix}
\Gamma_{m1} \\
\Gamma_{m2} \\
\Gamma_{m3}
\end{bmatrix}.
$$

Once we know the values $a, b$ and $c$, we can use these values and the measurement of $\Gamma_m$ to infer

$$
\Gamma_L = \frac{b - \Gamma_m}{c\Gamma_m - a}, \quad (2.3)
$$

when an unknown impedance is presented as the load. This value of $\Gamma_L$ can then be used to calculate the unknown load $Z_L$, by applying Equation 2.1.

### 2.1 Design of Amplitude and Phase Measurement System

For accurate measurement of $\Gamma_m$, it is important to make an accurate noise free measurement of $V_4^o$ and $V_3^o$. $V_4^o$ and $V_3^o$ are signals of a certain pre-determined frequency, $\omega$. These
signals can be sent as input to a Quadrature Demodulator. A quadrature demodulator is essentially a pair of mixers which multiplies its input by a pair of sinusoids that are identical except their phases are 90 degrees apart, $S_1 = S_0 \cos(\omega t)$ and $S_2 = S_0 \sin(\omega t)$. Letting $V_{k0}^o = V_{k0}^o \cos(\omega t + \phi_k)$ denote the port-$k$ output ($k = 3, 4$) that is fed as input to the quadrature demodulator (see Figure 2.2), we have:

$$S_1 \ast V_{k0}^o = \frac{S_0 V_{k0}^o}{2} \left( \cos(\phi_k) + \cos(2\omega t + \phi_k) \right),$$

$$S_2 \ast V_{k0}^o = \frac{S_0 V_{k0}^o}{2} \left( \sin(\phi_k) + \sin(2\omega t + \phi_k) \right).$$

On low pass filtering these two mixer outputs, high frequency terms are rejected and the two outputs are the in-phase component:

$$I_k = \frac{S_0 V_{k0}^o}{2} \left( \cos(\phi_k) \right),$$

and the quadrature-phase component,

$$Q_k = \frac{S_0 V_{k0}^o}{2} \left( \sin(\phi_k) \right).$$

Then the ratio of amplitudes of $V_4^o$ and $V_3^o$ is calculated using:

$$\frac{V_4^o}{V_3^o} = \sqrt{\frac{I_4^2 + Q_4^2}{I_3^2 + Q_3^2}}.$$  

(2.6)

The phase difference $\phi_4 - \phi_3$ is given by:

$$\phi_4 - \phi_3 = \tan^{-1} \left( \frac{Q_4}{I_4} \right) - \tan^{-1} \left( \frac{Q_3}{I_3} \right).$$

(2.7)

The outputs of the quadrature demodulator are received by the microprocessor through an inbuilt Analog to Digital Converter (ADC). The microprocessor performs the calculations stated above to accurately determine $\Gamma_m = \frac{V_4^o}{V_3^o}$. While calibrating, it uses these values to calculate the coefficients $a, b$ and $c$ using the matrix-based computation discussed in previous section. While measuring the unknown load, it uses the $a, b, c$ coefficients to find out the reflection coefficient $\Gamma_L$ for the unknown load using Equation 2.3. The $\Gamma_L$ value is then used to determine the unknown load impedance $Z_L$ using Equation 2.1. For quadrature demodulation, AD8333 from analog devices has been used.
Figure 2.3  Self-Calibration using SP4T switch.

Figure 2.4  Implementation of different power modes in transmission and reception.
2.2 Design of Self-Calibration system

As seen in previous section, for sensor calibration we need the measurements on a set of 3 known impedances in order to account for the non-colocation of the load impedance and the coupler output ports. In order to make the sensor a self-calibrating system, we have designed a self-calibrating mechanism using a Single-Pole-4-Throw (SP4T) switch. An SP4T switch has a 2 bit control signal which controls the connection of input RF port to one of the 4 output ports (see Figure 2.3). The control signal is programmed by the microprocessor at the beginning of each sensing event to cycle through all the four values. Once the control signal has swept through the values 00 (open-circuit), 01 (short-circuit) and 10 (matched-load), we calculate the calibration constants $a$, $b$ and $c$. For the measurement of unknown impedance, the control signal is changed to 11. Using the $\Gamma_m$ for this measurement and calculated $a, b, c$ values, we calculate $\Gamma_L$ which is then used to calculate $Z_L$.

2.3 Multi-Power modes in transceiver

An energy-efficient MAC Sahota et al. (2011) can be implemented if an additional higher power transmission mode, called ping, and an additional lower power receiver sensitivity mode, called drowsy is included (besides the usual normal and sleep modes). This can be implemented by using a system shown in Figure 2.4, where a Single-Pole-2-Throw (SP2T) switch is placed in between the transceiver and the antenna. During a transmission in the ping mode, the SP2T connects the transmitter to a power amplifier which amplifies the signal by 15 dB before feeding it to the antenna. During reception, the SP2T feeds the signal directly to the receiver, bypassing the power amplifier. Besides, the off-the-shelf transceiver CC1110 from Texas Instruments that we use already has an in-built receiver with the additional drowsy mode for a “low-powered listening” during wake-up synchronization as initiated by the generation of a “high-powered ping” by a neighboring node Sahota et al. (2011).
Once the microprocessor has the soil impedance measurement, it needs to transmit it to the receiver. For this purpose, and to reduce the size of the sensor node, we have designed the sensor electrodes to double up and act as an antenna with the help of a diplexer. A quarter wavelength monopole antenna at 433 MHz frequency is approximately 17 cm in length and has been mounted as a center prong on a copper ground plane. Apart from this center prong that acts as a monopole antenna mounted on a copper ground plane, there are 4 more prongs surrounding the central prong (see Figure 2.5) which act as the ground pins for the antenna as well as the sensor electrode. In this 5 prong sensor, the center prong acts as the antenna at the transmission frequency of 433 MHz and as the positive electrode at the sensing frequencies.
of 1MHz to 30 MHz. The remaining 4 prongs act as the ground in the antenna and as the negative electrode in the sensor measurements. Since the transmission and the sensing occur at two different frequencies, 433MHz versus 1-30MHz respectively, it is possible to separate the two frequency paths using a diplexer (see Figure 2.6). A diplexer has a low pass path with the transmission frequency in its pass-band and the minimum sensing frequency in its stop-band. It also has a high pass path with the transmission frequency in its stop band and the minimum sensing frequency in its pass band.

2.5 Complete Sensor Architecture

The complete sensor architecture consisting of microprocessor, transceiver, phase-lock-loop (PLL) for sinusoid generation, directional coupler, quadrature demodulator, SP4T and SP2T switches, power amplifier, low-pass-filter and diplexer is as shown in Figure 2.7. When the system starts, the first step by the microprocessor is to program the PLL to the desired frequency of operation. The microprocessor, that also has an inbuilt transceiver, is CC1110 from Texas Instruments. In the first step, the $I^2C$ interface of the programmable PLL (CDC903 from Texas Instruments) is programmed to generate 2 frequencies $\omega_1$ and $\omega_2$ with $\omega_2 = 4\omega_1$. The $\omega_1$ signal is sent through the transmission line towards the SP4T switch. The $\omega_2$ ($= 4\omega_1$) signal is sent to the quadrature demodulator which internally converts these signals to 2 signals $S_1$ and $S_2$ that are at $90^\circ$ phase difference from each other. These signals are used by the quadrature demodulator to convert the outputs of the couplers into in-phase and quadrature-phase components as explained in Section III.

The SP4T switch which is used for calibration gets its control bits from the microprocessor. In the calibration mode, a sequence of 00, 01 and 10 is sent to the SP4T switch. Thus, the transmission line is connected to open, short and matched load in the consecutive cycles of SP4T switching control signal. Once calibration is completed the control signal is set to 11 which sets the connection to the unknown load which in our application is soil. The incident and reflected waves are coupled to the output ports of the directional couplers located along the transmission line. The output signals are passed on to the inputs of a quadrature demodulator. The quadrature demodulator performs the mixing with in-phase and quadrature-phase
signals, and the result is low pass filtered to get the DC outputs consisting of the in-phase and quadrature-phase components. The outputs of the low-pass filter are received by the microprocessor through an in-built 12-bit ADC (Effective number of bits is 10.8). The microprocessor calculates Amplitude and Phase of the incident and reflected waves. In the calibration mode, these values are used to calculate the coefficients $a$, $b$ and $c$ using the matrix method discussed in the previous sections. In the measurement mode, it uses these coefficient values to find out the reflection coefficient $\Gamma_L$ for an unknown load. The $\Gamma_L$ value is then used to determine the unknown impedance $Z_L$ using Equation 2.1.
2.6 Experimental Validation of Impedance Measurement system

A cylindrical fixture was constructed using acrylic material to hold the soil with the 5-prong sensor embedded into the soil (see Figure 2.8). To connect the sensor to the on-board circuit, the fixture was fitted with an SMA (Sub-Miniature version A) port at the top whose one end was connected to the SP4T switch mounted on the the pc board and the other end, which is interior to the cylindrical fixture, was connected to the 5-prong sensor/antenna. The cylindrical fixture with 5-prong sensor inside was filled with the clarion loam soil that was collected from the top 0.50 m layer at the Iowa State University Agronomy Research Farm situated in Boone County, Iowa. The soil impedance was measured by the on-board sensor that we designed and
for comparison of its accuracy also by a Network Analyzer (HP8714ES). This 5-prong sensor
with soil contained between the prongs, acts as the unknown load as discussed in Section III.
The data recorded by the on-board circuit is transmitted to a receiver which first calculates
the $a$, $b$ and $c$ calibration constants and then calculates the soil impedance using Equations 2.1
and 2.2. The real and imaginary parts of impedance measured using the on-board sensor
showed a good match with those measured using the network analyzer in the range 1-30 MHz
(see Figures 2.9 and 2.10).
2.7 Experimental Validation of Multi-Frequency Nitrate determination

A dielectric mixture comprises of a host material of certain dielectric constant value $\epsilon_{\text{host}}$ into which different scatterers, each with permittivity $\epsilon_i$ are embedded. Although such mixtures are microscopically heterogeneous, we can treat them as a macroscopically homogeneous mixture under certain assumptions about shape, concentration, conductivity and orientation of these inclusions. Many models have been developed in the past to explain the dielectric behavior of a mixture using dielectric constant of components and their concentrations. Maxwell Garnett mixing rule is one of the most well known model which assumes inclusions to be spherical and uniformly distributed in a host medium. The permittivity of the mixture $\epsilon_{\text{eff}}$ is given by:

$$\frac{\epsilon_{\text{eff}} - \epsilon_{\text{host}}}{\epsilon_{\text{eff}} + 2\epsilon_{\text{host}}} = f_i \frac{\epsilon_i - \epsilon_{\text{host}}}{\epsilon_i + 2\epsilon_{\text{host}}},$$

(2.8)

For multiple inclusions, this formula extends to:

$$\frac{\epsilon_{\text{eff}} - \epsilon_{\text{host}}}{\epsilon_{\text{eff}} + 2\epsilon_{\text{host}}} = \sum f_i \frac{\epsilon_i - \epsilon_{\text{host}}}{\epsilon_i + 2\epsilon_{\text{host}}}. \quad (2.9)$$

The Maxwell Garnett formula has been shown to work well for low concentrations of inclusions. However, when inclusion concentration becomes almost equal to the host then a more symmetrical rule is needed. Such a mixing rule has been proposed by Bruggeman:

$$\sum f_i \frac{\epsilon_i - \epsilon_{\text{eff}}}{\epsilon_i + 2\epsilon_{\text{eff}}} = 0. \quad (2.10)$$

A more generalized model Sihvola and Kong (1988) considers a mixture of $n$ different types of ellipsoidal particles with different concentration, orientation and distribution that are embedded in a host with permittivity $\epsilon_{\text{host}}$. The proposed equation for effective mixture permittivity, $\epsilon_{\text{eff}}$, in this model is:

$$\epsilon_{\text{eff}} = \epsilon_{\text{host}} + \frac{\frac{1}{3} \sum_{j=1}^{n} f_j (\epsilon_j - \epsilon_{\text{host}}) \sum_{i=1}^{3} \frac{\epsilon_{\text{host}}}{\epsilon_{\text{host}} + N_{ji}(\epsilon_j - \epsilon_{\text{host}})}}{1 - \frac{1}{3} \sum_{j=1}^{n} f_j (\epsilon_j - \epsilon_{\text{host}}) \sum_{i=1}^{3} \frac{N_{ji}}{\epsilon_{\text{host}} + N_{ji}(\epsilon_j - \epsilon_{\text{host}})}}, \quad (2.11)$$

where $f_j$ is the volume fraction of $j^{th}$ inclusion, $N_{ji}$’s are the depolarization factors of $j^{th}$ component along $i^{th}$ coordinate (value of depolarization factor depend on the shape of the inclusion) and $\epsilon_j$ is the permittivity of $j^{th}$ component. All the above dielectric mixing models
are Quasi-Static in nature, meaning that they hold well only for an Electric field that is not time dependent. However, for sufficiently low frequencies at which the particle is much smaller than the wavelength, the static approximation can still be used Sihvola (1999). This implies that for a certain range of frequencies, the static models for effective permittivity will hold well.

In general permittivity can be measured using the impedance measurements. Hence, accurate multi-frequency measurements on soil impedance can provide information about components of soil with known permittivity. To use impedance measurements to obtain ionic concentrations, we treat the soil as a homogeneous medium and various ions as inclusions embedded into this medium. Such homogenizing methods have been used in the past to obtain dielectric mixture models for soil Sihvola and Kong (1988). From the above models, if the properties of individual constituents are known and an accurate measurement of permittivity of the mixture is made, it is possible to calculate individual ionic concentrations. For a host mixed with n additional components, we need at least n equations in n unknown ionic concentrations. These n equations can be obtained by making n number of measurements of $\epsilon_{eff}$ at a fixed or multiple frequencies. For different frequencies, permittivity of individual ions also varies due to dielectric relaxation. Many such relaxation models like debye relaxation, Havriliak-Negami relaxation etc are present in literature Santamarina et al. (2001). Debye relaxation model is given by the equation:

$$
\epsilon = \epsilon' - j\epsilon'' = \epsilon_\infty + \frac{\epsilon_S - \epsilon_\infty}{1 + j\omega\tau},
$$

(2.12)

where $\epsilon_S$ the permittivity of the molecule at very low frequencies, $\epsilon_\infty$ is the permittivity at very high frequencies and $\tau$ is the relaxation time which is defined as the time required by the molecular dipole to reach new equilibrium when a time varying external Electric field is applied.

2.8 Experimental Setup and Measurements

A steel metallic cylindrical fixture partitioned into three cylindrical blocks, acting as outer conductor, and a hollow core, acting as central conductor (see Figure 1) was machined. The space between the outer and inner cylinders of the middle block is used to hold soil sample
Figure 2.11  Cylindrical fixture to measure soil impedance

Whereas the same space between the top and middle blocks was filled with an acrylic insulator. The two ends of the fixture were fitted with SMA connectors (SubMiniature version A) which can be connected to a Network Analyzer for impedance measurement. Soil sample was collected from the top 0.50 m layer at the Iowa State University Agronomy Research Farm in Boone County, Iowa. Impedance of the soil was measured at different concentrations of components over a frequency range of 300 kHz - 75 MHz using the Network Analyzer (HP8714ES).

2.9 Capacitance of dry soil

Dry soil is a dielectric mixture of 2 components - powdered rock (soil bulk) and air particles which are trapped in between the soil. Depending on the type of soil, the air fraction may change. Since air fraction can be a considerable amount of total volume, it has to be taken into account. Moreover, an estimate of permittivity of soil bulk is needed in order to use the dielectric mixing formulas discussed in previous sections. 500 ml of the soil sample was dried at 450°F for an hour to remove all the water trapped between soil cavities. Another set of measurement was done with just the air. Since the capacitance is proportional to permittivity,
the permittivity in above equation can be replaced by capacitance determined using Equation 3.8. The variations in permittivity of air and soil-air mixture is shown in Figure 2.12.

2.10 Variations in capacitance and conductance with varying nitrate concentration

To the dry soil sample a 100 mM Sodium Nitrate solution was added in steps of 25 ml (5% by volume). The aim was to capture the variations in capacitance and conductance with changing amount of solution and trying to explain the results in terms of dielectric mixing models discussed in previous parts. After adding the solution to soil, the mixture was allowed to rest for 30 minutes so that the solution is absorbed well and the mixture becomes as uniform as possible within the limits of the experiment. Due to high dissociation constant of sodium nitrate (NaNO$_3$), we assume that all of the salt exists in ionic form Na$^+$ and NO$_3^-$.

Since each 25 ml step of 100 mM NaNO$_3$ increases the concentration of both the ions and water present in soil, the capacitance and conductance should both increase proportionately. But due to factors such as non-uniform distribution of solution in soil, interfacial polarization, loss due to scattering and presence of magnetic impurities and ionic salts, the real behavior is different from ideal.

Also, lack in continuity of solution affects the conductivity. Hence, a 25 ml addition of sodium nitrate may increase the conductance if a direct connection between inner and outer
The measurements were repeated three times and the average value was calculated at each frequency in order to minimize the effect of random noise present in the system. The measured capacitance is shown in the Figure 2.13. It can be observed that for low concentrations of sodium nitrate solution, the capacitance is almost same as that of dry soil. Apart from experimental errors, this might be because initial water added to dry soil is used to form the bound water layer which does not polarize as fast as free water in presence of an external electric field. Also concentration of sodium and nitrate ions is too small to add significantly to the capacitance.
The conductance values shown in Figure 2.14 show an increasing conductance with increase in concentration of sodium nitrate solution.

2.11 Analysis of impedance data to extract water and nitrate content information

2.11.0.1 Determination of permittivity from impedance

Recall that the cylindrical fixture has 3 cylindrical blocks assembled in 3 layers. Top and bottom cylinders are identical and are connected to SMA (Sub-miniature version A) connectors which are used to connect these ports to the network analyzer. A central cylindrical rod connects the two ends of the 3 layered cylindrical system. The outer cylinder acts as ground for this coaxial assembly. Soil is filled in central cylinder while top and bottom cylinder are filled with acrylic and hence have fixed permittivity. For this coaxial cylinder the distributed RLGC parameters are given by the equations Eisenstadt and Eo (1992):

\[ R = \text{Re}(\gamma Z), L = \text{Im}(\gamma Z)/\omega, G = \text{Re}(\gamma/Z), C = \text{Im}(\gamma/Z)/\omega, \]  
\[ (2.13) \]

where \( \gamma \) is the propagation constant for the coaxial line and \( Z \) is its characteristic impedance. \( \gamma \) and \( Z \) for a cylindrical coaxial conductor of length \( L \) can be calculated from the \( S \)-parameters by the equations:

\[ e^{-\gamma L} = \left\{ \frac{1 - S_{11}^2 + S_{21}^2}{2S_{21}} \pm \left\{ \frac{(S_{11}^2 - S_{21}^2 + 1)^2 - 4S_{11}^2}{(2S_{21})^2} \right\}^{1/2} \right\}^{-1}, \]  
\[ (2.14) \]

\[ Z^2 = \frac{Z_0^2(1 + S_{11}^2)^2 - S_{21}^2}{(1 - S_{11}^2)^2 - S_{21}^2}. \]  
\[ (2.15) \]

\( S \)-parameter matrix maps the incident waves at the two ports to the reflected waves at the same two ports. From electromagnetic analysis the capacitance of coaxial cylinder is proportional to the real part of permittivity while the conductance is directly proportional to the imaginary part Shi et al. (2011). Since distributed parameters when multiplied by length approximate the overall lumped parameters, the \( C \) and \( G \) measured using \( S \) parameters give an estimate of the medium permittivity. However, care has to be taken to keep the frequency low enough to minimize skin and proximity effects. In particular, \( C \propto \text{Re}(\epsilon) \) and \( G \propto \text{Imag}(\epsilon) \). \( R \) and
$L$ parameters are affected by skin and proximity effects at high frequencies and depend on permeability of the medium.

### 2.11.0.2 Port De-embedding

The upper and lower cylindrical blocks along with the SMA ports need to be de-embedded so that the measurements reflect only the capacitance and conductance parameters of the center cylinder which contains the soil under test. If $T_0$ matrix represents the $T$-parameter matrix for center cylinder and $T_1$ and $T_2$ are the $T$ parameter matrix for upper and lower blocks respectively, then:

$$M = T_1T_0T_2$$

(2.16)

represents the $T$-parameter matrix for the combined system. $T$-parameter matrix maps the incident and reflected waves at the output port to those at the input port, and can be related to $S$-parameter matrix (see below). $T_1$ and $T_2$ are assumed to be same due to symmetry of construction. A measurement with upper and lower blocks was taken by removing the center cylinder from between. This gives us:

$$M' = T_1T_2 = T_1^2 = T_2^2.$$  

(2.17)

This means that $\sqrt{M'}$ gives us the matrix $T_1 = T_2$. For a complex-valued square matrix there can be more than one square root. In general, for a 2x2 matrix, like in our case, there can be 4 distinct square roots. To choose the correct square root we observe that a small lossless transmission line has $T$-parameter matrix of the form:

$$
\begin{bmatrix}
  e^{-j\beta L} & 0 \\
  0 & e^{j\beta L}
\end{bmatrix},
$$

where $L$ is the length of the transmission line and the propagation constant $\beta$ is a positive number. For a small transmission line ($L << \lambda$), this is close to an identity matrix with a small imaginary part. This gives us an indication of correct square root to choose. Once we know $T_1$ and $T_2$, we can invert them to determine $T_1^{-1}$ and $T_2^{-1}$. To determine the $T$-parameter matrix for the central cylindrical block we can now use the equation:

$$T_0 = T_1^{-1}MT_2^{-1}.$$  

(2.18)
Since network analyzer measurements provide $S$-parameters for 2-port cylindrical system, we need to convert $S$-parameters to $T$-parameters in order to de-embed upper and lower cylindrical blocks. This can be done using following relation between $S$ and $T$-parameter matrices Weber (2001):

$$
\begin{pmatrix}
T_{11} & T_{12} \\
T_{21} & T_{22}
\end{pmatrix} = \frac{1}{S_{21}} \begin{pmatrix}
-det(S) & S_{11} \\
-S_{22} & 1
\end{pmatrix}.
$$

(2.19)

Once we have de-embedded $T$-parameter matrix for central cylinder containing soil, we need to convert the $T$-parameter back to $S$-parameter in order to use Equation 3.8 to derive capacitance and conductance parameters. The conversion from $T$-parameter to $S$-parameter matrix can be done using Weber (2001):

$$
\begin{pmatrix}
S_{11} & S_{12} \\
S_{21} & S_{22}
\end{pmatrix} = \frac{1}{T_{22}} \begin{pmatrix}
T_{12} & det(T) \\
1 & -T_{21}
\end{pmatrix}.
$$

(2.20)

### 2.11.0.3 Estimation of air fraction in dry soil

Soil particles come in various shapes. The fraction of air in soil may vary depending on the type of soil and its particle size distribution. Air fraction is generally estimated by comparing the density of bulk soil with soil-air mixture. It is given by the equation:

$$V_{bulk} = \frac{\rho_{bulk}}{\rho_{soil}}.
$$

(2.21)

Since the aim of this work is to measure the soil constituents in-situ, we treat soil as a
mixture of soil particles and air. Since air fraction can be considerably large, it is difficult to consider either soil bulk or air as the background material (and other as inclusion). Hence, we used a symmetrical Bruggeman mixture model. According to Bruggeman’s model for spherical particles:

\[
f_{\text{soil}} \frac{\epsilon_{\text{soil}} - \epsilon_{\text{eff}}}{\epsilon_{\text{soil}} + 2\epsilon_{\text{eff}}} + f_{\text{air}} \frac{\epsilon_{\text{air}} - \epsilon_{\text{eff}}}{\epsilon_{\text{soil}} + 2\epsilon_{\text{eff}}} = 0, \tag{2.22}
\]

where,

\[f_{\text{air}} = 1 - f_{\text{soil}}.\]

The estimated value of dry soil capacitance is shown in Figure 2.15, which shows that the soil bulk has capacitance (hence permittivity) higher than both air and dry soil which is expected. The estimated air fraction was close to 50.2%.

### 2.11.0.4 Estimation of Saline water content

Once Sodium nitrate solution is added to the soil, the permittivity of the soil starts to increase. This results in increasing capacitance and conductance of the soil dielectric mixture. We assume that the fraction of soil bulk does not change during the course of the experiment (i.e. any swelling has negligible effect on soil-bulk fraction). Again we use the Bruggeman’s symmetrical mixing rule:

\[
f_{\text{soil}} \frac{\epsilon_{\text{soil}} - \epsilon_{\text{eff}}}{\epsilon_{\text{soil}} + 2\epsilon_{\text{eff}}} + f_{\text{air}} \frac{\epsilon_{\text{air}} - \epsilon_{\text{eff}}}{\epsilon_{\text{soil}} + 2\epsilon_{\text{eff}}} + f_{\text{sol}} \frac{\epsilon_{\text{sol}} - \epsilon_{\text{eff}}}{\epsilon_{\text{sol}} + 2\epsilon_{\text{eff}}} = 0. \tag{2.23}
\]

where,

\[f_{\text{air}} + f_{\text{soil}} + f_{\text{sol}} = 1,
\]

and \(f_{\text{sol}}\) is the fractional volume of solution added. Note again that each \(\epsilon\) term is a function of frequency and is assumed to follow the Debye relation model mentioned in Equation 5.2. According to the discussion in Sihvola (1999) on lossy inclusions in lossless background dielectric mixtures, if the imaginary part of the permittivity of inclusion is much smaller than the real part, then the effective permittivity is indistinguishable from the corresponding material with equivalent real permittivity but no imaginary permittivity. This essentially implies that for
Figure 2.16  Estimation of Saline water fractional volume

Accordingly, the estimated fraction of solution is shown in Figure 2.16 (dashed curve) and compared with the actual amount of solution added (solid curve). High inaccuracy is observed for very low and very high concentrations (less than 8% and more than 40%). In the mid-range the maximum error was found when solution volumetric content was around 15%. Upon linearization, the estimated curve gets quite close to the actual line. The remaining error can be explained in terms of spherical particle assumption, lossy nature of dielectric and experimental errors like inaccurate calibration of network analyzer and presence of bound water which restricts the polarizations in molecules of the solution.

2.11.0.5  Estimation of nitrate content

The conductivity of a dielectric mixture has been shown to increase with increasing concentration of saline solution. The conductance of cylindrical fixture is proportional to the imaginary part of dielectric permittivity. For a dielectric mixture with saline solution as a component, all of the conductivity is provided by the moving ions in the saline solution. The
other components, air and soil bulk are non conducting. Hence, once we have an estimate of fractional volume of saline solution, we can predict the amount of ions present by relating the conductivity at a single frequency with the molar concentration of ions present in the overall volume. The increase in conductivity at 300 kHz with increasing molar fraction on sodium nitrate is shown in Figure 2.17. At concentrations above 20 µM, the conductivity is linear. Since a 100 mM solution of sodium nitrate was used for the experiment, the lower concentration of nitrate implies a lower concentration of fractional water volume which in turn means a larger fraction of water being in the form of bound water. Moreover, less water fraction implies lesser connectivity between inner and outer cylinder which results in reduced conductivity.

2.11.0.6 Incorporating bound water in the mixing model

Molecules of water that are close to the surface of soil particles are not as free to move as water molecules which are at a distance. Such molecules constitute what is know as bound water. Bound water fraction is different for different types of soils. Typical values of bound water fraction vary from 0.9% to 5% depending on soil type, particle size, temperature etc Yajing Wanga and Lia (2011). On considering bound water as a separate component, we obtain the extended mixing model:

\[
\frac{f_{\text{soil}}}{\epsilon_{\text{soil}}} - \epsilon_{\text{eff}} + \frac{f_{\text{air}}}{\epsilon_{\text{air}}} - \epsilon_{\text{eff}} + \frac{f_{\text{bw}}}{\epsilon_{\text{bw}}} - \epsilon_{\text{eff}} + \frac{f_{\text{sol}}}{\epsilon_{\text{sol}}} - \epsilon_{\text{eff}} = 0. \tag{2.24}
\]
Figure 2.18  Estimation of Saline water fractional volume based on 3% bound water fractional volume assumption

Figure 2.19  Estimation of Saline water fractional volume based on apparent permittivity assumption in the vicinity of particles
The permittivity of bound water has been empirically shown to be close to that of ice (Dobson and Hallikainen (1985)). With this assumption and 3% volume fraction assumption on bound water fraction, we observe 1% improvement in the estimate for water fraction, as can be seen in Figure 2.18

2.11.0.7 Incorporating apparent permittivity near the particles

The original Bruggeman’s model is based on assumption that the permittivity near the assumed spherical particles is the effective permittivity. This assumption, however, may not be perfect specially when the concentration of the inclusion is small. In that case, the permittivity near the particles becomes closer to the permittivity of the host which is soil-particle in our case. A parametrized linear expression for apparent permittivity based on soil-particle and effective permittivites is given by Sihvola and Kong (1988):

\[ \epsilon_{\text{apparent}} = \epsilon_{\text{soil}} + \lambda(\epsilon_{\text{eff}} - \epsilon_{\text{soil}}) \] (2.25)

Different values of \( \lambda \) were tested to determine the closest estimate of saline water fraction. An estimated curve for \( \lambda = 0.96 \) is shown in Figure 2.19. It was observed that the estimates got closer to the actual value specially for lower concentrations. For higher concentrations, the error was still present, which can be attributed to the fact that permittivity in the vicinity of particles cannot be the same for all concentrations.
CHAPTER 3. Soil Ionic Concentration Measurement by Dielectric Spectroscopy of a Buried Microstrip Patch

An approach to infer the moisture and nitrate concentration in soil from in-situ, multi-frequency measurements of the input impedance of a microstrip patch sensing element buried underground is presented. The permittivity of soil dielectric mixtures is determined using impedance spectroscopy and solving the Bruggeman’s dielectric mixing model. Under the quasi static assumption, the approach predicts nitrate concentration with 90% accuracy for a 50 mM sodium nitrate concentration of less than 15% by volume. It was observed that soil conductivity is linear to the inferred nitrate concentration for a given saline water fraction in soil. The model has been validated by making multi-frequency impedance measurements of a soil mixture at different concentrations comprising of various constituents like soil, air, water and ions. To reduce the sensor size, a diplexer has been designed to allow the microstrip patch sensing element to dual as an antenna. A prototype that uses this method to determine real time moisture and nitrate concentration in soil is also presented.

3.1 Introduction

Determination of accurate ionic concentrations in a dielectric mixture is a long standing research problem. One of the most important applications of ionic concentration determination is the estimation of soil nitrate concentration in an agricultural setting. Accurate estimation of nitrates can provide great improvements in agricultural production by helping to regulate nitrate management. It can hugely benefit the environment by minimizing the amount of nitrate leeching to the watershed.

To understand the nature of the soil mixture, an understanding of its different constituents
is needed. In general, soil is comprised of a number of constituents like soil bulk, moisture, ions and trapped air molecules. Moisture itself is further divided into two parts—bound versus free moisture Santamarina et al. (2001); deLoor (1968). Bound moisture is the moisture which is closest to the soil particles and forms a layer around them called the Stern Layer. In the Stern layer, water is not free to move and its diffusivity and polarizability are not the same as those of the free water. At some distance from the soil particles, water is present as free flowing liquid. This layer is known as Gouy layer and possesses properties of normal liquid water. Due to the presence of water, salts tend to ionize and hence conductivity of the soil is increased. The conductivity is directly proportional to the dissociation constant of salt-water equilibrium reaction. Conductivity increases with valency of cation and anion and with the mobility of the ion (which is a function of its size). Another important property of these ionized salts is that both the cation and anion tend to hydrate themselves. This means that the total ionic radius includes the size of the ion along with the size of water molecules which are attached to this free ion. Ions with larger hydrated radius have lesser mobility and result in lesser contribution towards overall conductivity.

A method to determine the equivalent permittivity of a dielectric mixture like soil is by using the dielectric mixture models. Dielectric mixing models are used to determine the permittivity of a mixture in terms of the permittivity and concentration of its constituents. Many dielectric mixing models such as Maxwell-Garnett, Bruggeman Sihvola (1999) and deLoor’s mixing rule Dobson and Hallikainen (1985) have been shown to be efficient in describing soil moisture content given that the permittivities of other constituents like soil bulk, trapped air and bound and bulk water are known, where bound water can be assumed to have the electrical properties of ice. This assumption has been validated by making accurate measurements of soil moisture using the dielectric measurements Dobson and Hallikainen (1985). An empirical model has also been developed Wang and Schmugge (1980) to relate the dielectric constant with varying moisture content in the soil. It has been demonstrated in Wang and Schmugge (1980) that a combination of dielectric properties of ice, water, air and soil bulk can describe the dielectric behavior of soil-water mixture at 1.4 GHz and 5 GHz. Models for moist soils based on variations in soil complex refractive index with moisture have also been developed Mironov et al. (2004).
In a more recent work, the dielectric properties of saline deposits are used for mapping of moisture content Lasne (2007).

Most work in soil research has focused on soil moisture determination and little has been done so far to determine the concentration of ions such as nitrates, chlorides etc. In this paper, we use the dielectric mixture models to determine the concentration of an unknown constituent in the mixture. This is accomplished by first measuring the overall permittivity of the soil mixture at multiple frequencies and then using a dielectric mixture model to form equations in the unknown constituent of the mixture. Details of the method are discussed in later sections.

One key point here is that the permittivity itself is not a directly measurable quantity. In order to determine the permittivity of a mixture, measurement on a property that is directly measurable has to be made (such as impedance). One of the main approaches to measure input impedance of a sensing element embedded in soil is time domain reflectometry (TDR) which provides benefits like good accuracy and ease of measurement. In this method a square signal pulse of known properties is reflected from an unknown surface and time difference between the transmitted and received waves is used to determine the surface impedance. The time difference can be used to estimate the impedance as discussed in Gaskin and Miller (1996). The TDR approach has been extended to detect nitrate and chloride ions in soil as well. In one such study Krishnapillaia and Ranjanb (2009), authors present an in-laboratory method to detect nitrate concentration. A potassium nitrate solution of known concentration of 500ppm was flown through the TDR sensor contained in the soil inside a plexiglass cell. Nitrate concentration was changed with time and the TDR probes were placed at regular intervals along the flow cell. The bulk soil electrical conductivity and the water content values extracted from the TDR waveforms were used to predict the nitrate concentrations at different locations. The nitrate concentration values predicted from the TDR-measured bulk electrical conductivity and water content data were observed to correlate with the nitrate concentrations obtained by soil solution sampling method. This method however was implemented for laboratory operations only and was not extended to in-situ determination of nitrate concentration in soil. In another study Chighladze et al. (2011), the variation of soil permittivity and soil conductivity within the
frequency range 200 Hz to 13 MHz is presented. It is shown that the TDR approach can be used to detect nitrate concentration by measuring the soil permittivity and conductivity. The potential of using TDR to simultaneously estimate volumetric soil water content, soil solution electrical conductivity, and soil nitrate/nitrogen concentration in an irrigated peppermint field has also been demonstrated in Das et al. (1999). The work in Das et al. (1999) compares TDR-estimated nitrate/nitrogen concentration with the estimates obtained from direct soil measurements (soil cores and soil solution samples). It was observed that the estimates from all methods were comparable and had similar pattern, magnitude and variance. The work done on feasibility of using TDR to monitor changes in nitrate and nitrogen concentration in an irrigated agricultural soil Payero et al. (2006) concluded that the TDR probes could be used to measure nitrate/nitrogen concentration in non-saline soils and water after the proper calibration over a long enough period of time. Calibration is needed to include the expected variations in volumetric water content (VWC), temperature and nitrate/nitrogen concentration.

In other studies Neve et al. (2000, 2003), the effect of temperature on the extent of nitrogen mineralization in the soil due to various processes like microbial activity has been studied. This work also determines the feasibility of employing TDR based conductivity measurement to make these observations. It was observed that the mineralization of nitrogen results in a measurable increase in the electrical conductivity and hence nitrogen concentration could be monitored using the TDR probe.

TDR has the limit that it can help determine the concentration of only one constituent (like moisture). This is because the frequency of the incident signal is not known and the information as received is only in terms of one variable: time difference between incident and reflected signal. In this paper, we use the input impedance of a microstrip patch embedded in soil sample as a measurable property, which is known to be linearly related to the permittivity, and propose a method to determine the concentration of ions in a mixture like soil using impedance spectroscopy. The need for multiple frequency measurements arises because from a single frequency measurement, concentration of more than one unknown constituent cannot be determined, regardless of the accuracy of the measurements. Since all parameters, including permittivities, of each constituent are known except their concentrations, for a host with
$n$-constituent, measurements at $n$ frequencies can give adequate information to infer the $n$ unknown concentrations. If there are more than $n$ measurements, then least square methods are used to find the “best” fitting set of unknown concentrations that minimize the squared-error of the fit.

The contributions of our work are as summarized below:

- Proposing a method to determine the concentration of moisture as well as nitrate using multi-frequency impedance measurements and solving mixing plus relaxation models.
- Validation of the proposed method on a soil sample collected from the field.
- Presentation of an architecture for an underground wireless sensor prototype that has been developed for in-situ measurement of soil impedance and transmission of the measured impedance information.

Rest Following sections in the paper are organized as follows: Section 3.2 discusses the theory behind the proposed approach that has been employed to determine the ionic concentrations. Section 3.3 explains the experimental setup that was used to validate the theory; section 3.4 analyzes the result; section 3.5 discusses the circuit and package level architecture of a prototype that has been developed to make real time, multi-frequency impedance measurements. Section 3.6 concludes the paper with discussions for future directions.

### 3.2 Approach

A dielectric mixture comprises of a host material of certain dielectric constant value $\epsilon_{\text{host}}$, along with different scatterers, each with permittivity $\epsilon_i$ and fraction $f_i$ that are embedded in the host to form a mixture. Although such mixtures are microscopically heterogeneous, we can treat them as a macroscopically homogeneous mixture under certain assumptions about shape, concentration, conductivity and orientation of these inclusions. Many models have been developed in the past to explain the dielectric behavior of a mixture using dielectric constant of constituents and their concentrations. Maxwell Garnett mixing rule is one of the models which assumes inclusions to be spherical and uniformly distributed in a host medium. The
permittivity of the mixture $\epsilon_{eff}$ satisfies:

$$\frac{\epsilon_{eff} - \epsilon_{host}}{\epsilon_{eff} + 2\epsilon_{host}} = f_i \frac{\epsilon_i - \epsilon_{host}}{\epsilon_i + 2\epsilon_{host}}.$$  

(3.1)

For multiple inclusions, this formula extends to:

$$\frac{\epsilon_{eff} - \epsilon_{host}}{\epsilon_{eff} + 2\epsilon_{host}} = \sum_i f_i \frac{\epsilon_i - \epsilon_{host}}{\epsilon_i + 2\epsilon_{host}}.$$  

(3.2)

The Maxwell Garnett formula has been shown to work well for low concentrations of inclusions. However, when inclusion concentration becomes almost equal to the host, then a more symmetrical mixing rule is needed. Such a mixing rule has been proposed by Bruggeman:

$$\sum_i f_i \frac{\epsilon_i - \epsilon_{eff}}{\epsilon_i + 2\epsilon_{eff}} = 0.$$  

(3.3)

A more generalized model Sihvola and Kong (1988) considers a mixture of $n$ different types of ellipsoidal particles with different concentration, orientation and distribution, mixed in a host with permittivity $\epsilon_{host}$. The proposed equation for effective mixture permittivity, $\epsilon_{eff}$, in this model is:

$$\epsilon_{eff} = \epsilon_{host} + \frac{\frac{1}{3} \sum_{j=1}^{n} f_j (\epsilon_j - \epsilon_{host}) \sum_{i=1}^{3} \frac{\epsilon_{host}}{\epsilon_{host} + N_{ji}(\epsilon_j - \epsilon_{host})}}{1 - \frac{1}{3} \sum_{j=1}^{n} f_j (\epsilon_j - \epsilon_{host}) \sum_{i=1}^{3} \frac{N_{ji}}{\epsilon_{host} + N_{ji}(\epsilon_j - \epsilon_{host})}},$$  

(3.4)

where $f_j$ is the volume fraction of $j^{th}$ inclusion, $N_{ji}$'s are the depolarization factors of $j^{th}$ constituent along $i^{th}$ coordinate (value of depolarization factor depends on the shape of the inclusion) and $\epsilon_j$ is the permittivity of $j^{th}$ constituent. All the above dielectric mixing models are quasi-static in nature, meaning that they hold well only for an electric field that varies slow enough to let the particles attain their equilibrium distribution for ionization and polarization. For sufficiently low frequencies at which the particle is much smaller than the wavelength, the quasi-static approximation can be used Sihvola (1999). This implies that for a certain range of frequencies, the quasi-static models for effective permittivity holds well.

Permittivity can be measured using the impedance measurements since the two are linearly related, such as in case of a microstrip patch sensing element. Hence, accurate multi-frequency measurements on soil impedance can provide information about fractions of constituents of soil with known permittivities. To use impedance measurements to infer ionic concentrations,
we treat the soil as a homogeneous medium and various ions as inclusions embedded into this medium. Such homogenizing methods have been used in the past to obtain dielectric mixture models for soil Sihvola and Kong (1988). From the above models, if the properties of individual constituents are known and an accurate measurement of the permittivity of the mixture is made, it is possible to calculate individual ionic concentrations. For a host mixed with $n$ additional constituents, we need at least $n$ equations in $n$ unknown ionic concentrations. These $n$ equations can be obtained by making $n$ number of measurements of $\epsilon_{eff}$ at multiple frequencies. For different frequencies, permittivity of individual ions also varies due to dielectric relaxation. Many such relaxation models like debye relaxation, Havriliak-Negami relaxation etc. have been presented in literature Santamarina et al. (2001). The debye relaxation model is given by the equation:

$$
\epsilon = \epsilon_{\infty} + \frac{\epsilon_{S} - \epsilon_{\infty}}{1 + j\omega \tau},
$$

where $\epsilon_{S}$ the permittivity of the molecule at very low frequencies, $\epsilon_{\infty}$ is the permittivity at very high frequencies, $\tau$ is the relaxation time, which is defined as the time required by the molecular dipole to reach a new equilibrium when a time varying external electric field is applied.

### 3.3 Experimental Setup and Measurements

A soil sample was collected from the top 0.50 m layer at the Iowa State University Agronomy Research Farm situated in Boone County, Iowa. The soil was dried and collected in a rectangular container with 500 ml capacity (see Fig. 3.1). The microstrip patch was buried in the soil with a co-axial connector attached to it in order to measure the input impedance. Impedance of the soil was measured at different concentrations of constituent over a frequency range of 1MHz - 40MHz using the Network Analyzer (HP8714ES) (to first establish a proof-of-concept of our approach). The results also help validate the measurements obtained from our own sensor (see Section V).
3.3.0.8 Determination of permittivity from impedance

A lossless rectangular micro strip patch can be equated to a transmission line comprising of distributed differential components as shown in Fig. 3.2. At low frequencies however, the inductive part is very small compared to the capacitive part and can be ignored. This effectively implies that the patch is assumed to be a parallel plate capacitor. The capacitive energy is contained in 2 regions—In the substrate between two parallel layers and in the surroundings. The capacitance due to electric (E) field in the surroundings is known as the fringe capacitance and has been studied in the literature Nishiyama and Nakamura (1994). The capacitance of the substrate is given by the well known equation:

$$C_{ideal} = \epsilon_{sub} \frac{A}{D}$$

(3.6)

where $\epsilon_{sub}$ is the substrate permittivity, $A$ is the area of the parallel plates and $D$ is the distance between the plates. We are only interested in the fringe capacitance which can be viewed as a capacitor in parallel with the ideal parallel plate capacitor. The exact equation for the fringe capacitance is not needed and it suffices to know that the fringe capacitance is directly proportional to the surrounding permittivity. This is because by definition total energy
stored in the fringe electric field is given by:

\[ U = \frac{1}{2} \epsilon_{\text{eff}} \int_V |E|^2 dV = \frac{1}{2} CV^2 \]  

(3.7)

where \( V \) is the total potential difference between the plates and \( \epsilon_{\text{eff}} \) is the combined permittivity of the soil mixture and the substrate between the top and bottom plates of the patch.

Since the input impedance comprises of 2 components—capacitance due to substrate and capacitance due to fringe field in the surroundings, we need to separate the two in order to obtain a single term that is proportional to surrounding permittivity and hence can be used directly in the models discussed in previous section. This can be achieved simply by making a measurement of the microstrip patch capacitance embedded in the mixture and subtracting the value of the common substrate component from all the measurements. The resulting term does not contain the ideal parallel plate capacitance term and hence is independent of substrate permittivity and is also directly proportional to the surrounding permittivity. This is captured in equation (3.8) below:

\[ C_{\text{soil}} = C_{\text{measured}} - C_{\text{ideal}} \]  

(3.8)

where \( C_{\text{ideal}} \) is the ideal capacitor of just the parallel substrate without the fringe capacitance.
Variations in capacitance and conductance with varying nitrate concentration

To the 500 ml dry soil sample a 50 mM Sodium Nitrate solution was added in steps of 25 ml (i.e., 5% of the total volume of soil mixture used in experiment). The aim was to capture the variations in capacitance and conductance with changing amount of solution and to explain the results in terms of dielectric mixing models discussed in previous sections. After adding the solution to the soil, the mixture was allowed to rest for 15 minutes so that the solution is absorbed well and the mixture becomes uniform within the limits of the experiment. Due to high dissociation constant of sodium nitrate (NaNO$_3$), we assume that all of the salt exists in ionic form, Na$^+$ and NO$_3^−$.

Since each 25 ml addition of 50 mM NaNO$_3$ increases the concentration of both the ions and water present in soil, the capacitance and conductance are both expected to increase proportionately. But due to factors such as non-uniform distribution of solution in soil, interfacial polarization, loss due to scattering and presence of magnetic impurities and ionic salts, the actual behavior is different from ideal.

Also, lack in continuity of solution affects the conductivity. Hence, a 25 ml addition of sodium nitrate may increase the conductance only if a direct connection between top and bottom metal layers of the microstrip patch is formed. On the other hand, there may not be much effect on conductance if there is not much increase in the connectivity between the top
and bottom metal layers of the microstrip patch.

At each frequency, the measurements were repeated three times and the average value was calculated in order to minimize the effect of random noise affecting the measurements. The measured capacitance is shown in Fig. 2.13. It can be observed that for low concentrations of sodium nitrate solution, the capacitance is almost the same as that of dry soil. Apart from experimental errors, this might be because initial water added to dry soil is used to form the bound water layer which does not polarize as fast as free water in presence of an external electric field. Also concentration of sodium and nitrate ions is too small to add significantly to the capacitance. The conductance values shown in Fig. 2.14 show an increasing conductance with increase in concentration of sodium nitrate solution. Both capacitance and conductance curves show small peaks at regular intervals that can be attributed to the resonance of the coaxial cable that connects the patch to the network analyzer.

### 3.4 Analysis

Soil particles come in various shapes. The fraction of air in soil may vary depending on the type of soil and its particle size distribution. Once sodium nitrate solution is added to the soil, the permittivity of the soil starts to increase. This results in increasing capacitance and conductance of the soil dielectric mixture, as confirmed by Figs. 2.13 and 2.14, with some
initial “start-up” phase for capacitance as explained above. We assume that the amount of soil bulk as well as air does not change during the course of the experiment (i.e. any swelling has negligible effect on soil-bulk fraction and air is merely displaced due to addition of solution). The fraction of soil-air mixture represented by $f_{\text{soil}}$ is merely reduced due to addition of nitrate solution. Then using the Bruggeman’s symmetrical mixing rule, we obtain:

$$f_{\text{soil}} \frac{\epsilon_{\text{soil}} - \epsilon_{\text{eff}}}{\epsilon_{\text{soil}} + 2\epsilon_{\text{eff}}} + f_{\text{sol}} \frac{\epsilon_{\text{sol}} - \epsilon_{\text{eff}}}{\epsilon_{\text{sol}} + 2\epsilon_{\text{eff}}} = 0. \quad (3.9)$$

$$f_{\text{soil}} + f_{\text{sol}} = 1,$$

where $f_{\text{sol}}$ is the fractional volume of solution added. Note again that each $\epsilon$ term is a function of frequency and is assumed to follow the Debye relaxation model mentioned in Equation (5.2).

According to the discussion in Sihvola (1999) on lossy inclusions in lossless background dielectric mixtures, if the imaginary part of the permittivity of inclusion is much smaller than the real part, then the effective permittivity is indistinguishable from the corresponding material with equivalent real permittivity but no imaginary permittivity. This essentially implies that for low loss inclusions, the dielectric mixing formulas give close results even if only the real part of permittivity is considered. We make use of this fact to estimate the concentration of the sodium nitrate solution for which real part of permittivity is close to that of water ($\epsilon_{\text{water}} \approx 80$) while imaginary part, that is essentially the conductivity of 50 mM sodium nitrate divided by the angular frequency, is considerably small Janz et al. (1970).

The electric field due to the parallel plates of the patch is present in the substrate as well as surroundings. The capacitance due to substrate is a fixed value and needs to be subtracted from overall capacitance in order to get a value that is proportional to the surrounding permittivity. It has been discussed in Nishiyama and Nakamura (1994) that the capacitance due to fringe field in air is approximately 3 times that of substrate capacitance for the aspect ratio that has been used in our experiment ($\frac{11}{8}$). The fixed value of capacitance can thus be approximately estimated by taking a quarter of the capacitance measured with air as surrounding.

Accordingly, the estimated fraction of solution is shown in Fig. 3.5 and compared with the actual amount of solution added. High inaccuracy is observed for high concentrations (more than 20%). In the low-range (less than 8%) the maximum error was found to be less than
Figure 3.5 Estimation of saline water fractional volume

20%. One factor that causes larger error for lower concentrations is that the initial amount of solution acts as bound Santamarina et al. (2001) and hence needs to be treated separately. On assuming a 4% fraction of bound solution, the results are within 10% of the actual value of the saline water fraction.

3.4.0.10 Estimation of nitrate content

Figure 3.6 Estimation of sodium nitrate molar content

The conductivity of a dielectric mixture has been shown to increase with increasing concentration of saline solution. The conductance of soil dielectric mixture is proportional to the imaginary part of dielectric permittivity. For a dielectric mixture with saline solution as a
constituent, all the conductivity is provided by the moving ions in the saline solution. The other constituents, air and soil bulk are non conducting. Hence, once we have an estimate of fractional volume of saline solution, we can predict the amount of ions present by relating the conductivity at a single frequency with the molar concentration of ions present in the overall volume. The increase in conductivity at 1 MHz with increasing molar fraction on sodium nitrate is shown in Fig. 3.6. At concentrations above 20 $\mu$M, the conductivity is linear. Since a 50 mM solution of sodium nitrate was used for the experiment, the lower concentration of nitrate implies a lower concentration of fractional saline water volume which in turn means a larger fraction of saline water being in the bound form. Since conductance is directly proportional to the concentration of ions in water, a linear relation between conductance and concentration is expected. The measured value of average conductance for complete frequency range is quite close (within 5% of margin) to the expected linear model. Slight deviation is observed as soil is not a homogeneous solution and saline water is not uniformly distributed.

The approach can be extended to measure ionic concentrations when more than one ion is present in the mixture. The principal discussed above can still be applied with slight extension to account for the frequency and size dependent variations in the mobilities of the ions of various types.

### 3.5 Sensor for in-situ impedance measurements

We have recently designed and tested a dielectric measurement based soil impedance sensor that can sense at multi-frequencies (hence accurate & reliable), is self-calibrating (hence robust), possesses wireless interface (hence can be located in-situ), and is also energy-efficient Pandey et al. (2013b). The sensor architecture, consisting of probe and antenna, directional couplers, phase locked loop (PLL), amplitude and phase detector, switches/diplexer, microprocessor & transceiver, is shown in Fig. 4.2.

Upon startup, the microprocessor programs the I$^2$C interface of the programmable PLL to generate a signal of known frequency. The frequency of the probing signal is chosen in the range of 1-40 MHz, and is chosen so that a significant variation in real and imaginary part of the soil impedance can be observed. While the lower limit of 1 MHz on frequency is put by the
architecture of the sensor, the upper limit of 40 MHz was obtained experimentally as above this value the soil reactance becomes close to zero. The range of 1-40 MHz is sufficient for our application although same principle can be used to design an sensor with higher frequency range as well (using the PLL, demodulator and couplers that work for a higher frequency range). A slight increase in this value can provide more data points to analyze but beyond that no useful information on soil ionic concentration can be extracted from it. The probing signal is sent through the transmission line to the SP6T switch, which is programmed by the microprocessor to select among a set of known loads plus the unknown soil-sample load. The incident and
reflected signals to and from the load are captured using the directional couplers and are passed on to a detector which calculates the amplitude and phase of each signal and passes this information to the microprocessor for further processing and transmission via antenna. These values are received by the microprocessor through an in-built 12-bit ADC. In the calibration mode, when the loads are of known values, these values are used to calculate the calibration parameters that correlate the reflection coefficients (ratio of reflected to incident) measured at the couplers to those at the load through a 3-parameter bilinear transform. In the measurement mode, when the load is the soil-sample, these calibration parameters are used to find out the reflection coefficient for an unknown load from its value measured at the directional coupler, through the same bilinear transform whose parameters were determined in the calibration mode. The reflection coefficient value is then used to determine the unknown load impedance that contains the information about the soil contents (moisture and nutrients).

The resistive versus reactive soil impedance measurements over 1-40 MHz by our sensor are shown in Fig. 4.3. The accuracy of our in-situ sensor is confirmed against the measurements from a lab equipment, a network analyzer, HP8714ES (plots also shown in the same figures). A more than 90% accuracy over the range of 1-40 MHz in soil reactance was observed. This, together with the proof-of-concept of our ionic concentration sensing approach presented above, confirms that our own sensor can be used for estimating moisture and ionic concentrations in
soil mixtures (of bulk soil, air, free and bound water, and ions) using dielectric spectroscopy to populate the mixing and relaxation models.

### 3.5.0.11 Packaging

![Conceptual design of sensor package.](image)

A prototype has been designed on a PCB with dimensions $W = 2 \text{ in}$ and $L = 2 \text{ in}$. The printed circuit board (PCB) was packaged inside an acrylic box of dimension $W = 2.5 \text{ in}, L = 2.5 \text{ in}, H = 1 \text{ in}$. The package comprises of top and bottom hollow acrylic blocks. The central hollow space is used to contain the PCB and a battery. A coaxial cable connects the PCB to the microstrip patch sensing element which is located on the outer surface of the top acrylic block. The microstrip patch is designed to dual as an antenna at a frequency different from sensing frequency, and so the entire package can be buried underground where it can transmit data to a central station located above ground. Fig. 3.10 shows the dimensions of the prototype while Fig. 3.11 shows the picture of sensor inside the package. Package has a slot on its outer surface slot to fit a microstrip patch sensing-element cum antenna, antenna so it remains exposed to the surroundings while battery and PCB are kept inside the package to keep them waterproof. Acrylic box makes the sensor strong and durable. Due to non-degradable nature of acrylic, the sensor can last for a long period under the ground. Our initial calculations of sensing and communication energy indicate an off-the-shelf battery life of at least 2 years.
3.6 Conclusion

A method for estimating the in-situ nitrate concentration in soil by applying dielectric mixture models over mixture of Debye-type constituents is presented. It was shown that an estimate of saline water content is possible using existing dielectric mixture and relaxation models. It was also shown that the conductance of soil dielectric mixture varies linearly with concentration of ions present. Microstrip patch was shown to act as a good sensing element for the frequency range of 1-40 MHz. The same patch was also used as transmitting antenna at a higher frequency. The results of ionic concentrations derived from impedance measurements applying our approach were compared against the actual values (recall that solutions of known concentrations were poured into the dry soil) and an accuracy of 90% was found over 1-40 MHz frequency range, which is more than adequate for precision agriculture applications. The approach can extended to measure ionic concentrations in a mixture of multiple ions by accounting for the contributions of different ions into the overall conductivity that is a function of fraction, valency and mobility (which also depends on frequency). This is a direction for future research.

The proposed method can be implemented in real time using our in-situ, multifrequency sensor and hence can provide accurate, real time concentration of moisture and nitrates in the field. Knowledge of amount of moisture and nitrate present in the field at a given time can help us determine whether more nitrate or moisture need to be added to the soil or not, and which locations. Thus, increased resource utilization and minimal environmental affects can be achieved. This can provide huge benefits in soil nitrate management and can provide significant
agricultural and environmental benefits.

Although this work is presented in context of soil-ionic mixture, the same principals are applicable to any other Debye mixture. Also, the multi-frequency analysis can be extended to a mixture of any finite number of constituents.
CHAPTER 4. A low profile, low-RF band, small antenna for underground, in-situ sensing and wireless energy-efficient transmission

A key challenge to underground, in-situ soil sensing with wireless interface is the antenna size. Smaller operating frequency supports lower path losses but enhances the wavelength and hence the size of standard monpole (8.2 cm in height at 915MHz) or rectangular microstrip patch antenna (11.5 cm × 9.3 cm at 915 MHz), which is prohibitive for underground sensors. Lowering the frequency below 915MHz is not an option as it only further enhances the antenna size. To circumvent the size problem, a composite right-left handed (CRLH) microstrip patch antenna for wireless transmission at 915 MHz that doubles up as an underground, sensing element (external capacitor) has been designed and fabricated. The combined antenna/sensing CRLH patch integrates with the previously implemented on-board, multi-frequency dielectric based impedance sensor for soil moisture and nitrate determination and provides almost 93% reduction over the standard microstrip antenna size. As a proof of concept, the input impedance of the CRLH sensor, surrounded by the soil containing moisture and nitrate ions, is measured at multiple frequencies in the lab setting. It is shown that the change in moisture and nitrate can be successfully detected using the sensor. The small profile of the proposed antenna (3 cm × 2 cm), that is almost 93% smaller, makes it ideal for compact packaging.

An E-field as a function of spatial variable \(z\) and temporal variable \(t\) is given by, \(E(z,t) = e^{-\gamma z + j\omega t}\), where \(\omega\) is the frequency, \(\gamma = \sqrt{j\omega\mu\sigma - \omega^2\varepsilon\mu}\) is the propagation constant (\(\mu\) denotes the permeability, \(\sigma\) the conductivity and \(\varepsilon\) the permittivity). Letting \(\alpha\) and \(\beta\) denote the real and imaginary parts of \(\gamma\) and solving for \(\alpha\) and \(\beta\) we get:

\[
\alpha = \omega \sqrt{\frac{\mu}{\varepsilon}} (\sqrt{1 + \left(\frac{\sigma}{\omega\varepsilon}\right)^2} - 1) \quad \text{and} \quad \beta = \omega \sqrt{\frac{\mu}{\varepsilon}} (\sqrt{1 + \left(\frac{\sigma}{\omega\varepsilon}\right)^2} + 1).
\]

Then \(E(z,t) = E_0 e^{-\alpha z} e^{j(-\beta z + \omega t)}\), and it can be noted that the attenuation factor \(\alpha\) is approx-
approximately proportional to the square root of the frequency $\omega$, conductivity $\sigma$ and permeability $\mu$.

This means that in order to increase the transmission distance of a signal in a lossy medium ($\sigma \neq 0$) like soil with nutrients, a lower frequency of operation will be helpful in increasing the transmission distance. However, lowering the frequency means raising the wavelength, and this in turn means raising the antenna dimension, as for an efficient radiation from an antenna, antenna dimension should be of the order of quarter of a wavelength whether that be a wire antenna (like quarter wavelength monopole), a loop antenna or a patch antenna. Patch antennas are a natural choice for on-board sensors that are Systems on Package (SoP) or Systems on Chip (SoC) and are flat by design. Important properties which are desired from a good antenna are high $Q$ resonance at the frequency of operation (for high efficiency) and high gain/directivity. For the specific case of a rectangular patch antenna the dimensions needed for good radiation efficiency are given by equations below, derived using their transmission line model (Balanis (2005)):

Width of patch,

$$W = \frac{1}{2f_r \sqrt{\mu_0 \epsilon_0}} \sqrt{\frac{2}{\epsilon_r + 1}},$$

and

Length of patch,

$$L = \frac{1}{2f_r \sqrt{\epsilon_{reff} \mu_0 \epsilon_0}} \left( 2(0.412)h \right) \frac{(\epsilon_{reff} + 0.3)(W + 0.264)}{(\epsilon_{reff} - 0.258)(\frac{h}{W} + 0.8)},$$

where $h$ is the substrate height and $\epsilon_{reff}$ is the effective permittivity which is a function of substrate permittivity, $\epsilon_{sub}$. For a rectangular patch, it is given by the expression:

$$\epsilon_{reff} = \frac{\epsilon_{sub} + 1}{2} + \frac{\epsilon_{sub} - 1}{2} \left[ 1 + \frac{12h}{W} \right]^{-\frac{1}{2}}.$$

It can be observed that lowering the frequency increases the width as well as the length of a patch antenna. Also since $\sqrt{\epsilon_{reff}}$ appears in denominator, it seems that the size of the antenna can be reduced by using a very high permittivity substrate but this also has its own limit. To summarize, we need low frequency operation to counter the medium loss, but this amounts to a large size antenna (as wavelength is large for low frequencies).

To address this technological challenge, our proposal is to look beyond the natural materials, towards the so called metamaterials. One problem with small antenna design is that most of the energy fed to the antenna is stored in the near field of the antenna in form of electrical and magnetic energy and only a small fraction of supplied energy is radiated. Metamaterials
are specially constructed materials with properties that are the opposite of materials found in nature. Metamaterials can have negative permittivity or permeability or both. This creates a possibility of engineering a matching network using the metamaterials between the antenna and the surrounding medium in such a way that the energy stored in the near field is radiated away. Size improvements of factor of greater than 10 compared to standard antennas have been observed in Christophe Caloz and Rennings (2008), Richard W. Ziolkowski and Lin (2011), Ziolkowski and Erentok (2006). Metamaterials have also been employed more recently to improve radiation characteristics of small patch antenna. Authors in Christophe Caloz and Rennings (2008) discuss a composite right-left handed transmission line based antenna which has the potential to improve the efficiency of small patch antennas. One such implementation has been reported in Zhou Cheng and lian gang (2011). An elaborate design procedure of such antennas for lower frequencies taking into account details of parasitic and dielectric losses is still needed and we propose to investigate metamaterials to implement electrically small antennas for in-situ soil sensors.

4.1 Introduction

Nitrate based fertilizers are one of the most common type of fertilizers used for increasing the agricultural productivity. However, excessive use of nitrate based fertilizers can lead to severe environmental hazards. A deeper understanding of agricultural N cycling process is needed so that precise controls over N fertilizer inputs that are key to sustainable agriculture can be implemented. In a generic precision agriculture approach (see Fig. 4.1), intra- and inter-field variabilities are characterized using a network of sensor nodes spread over a large area. Each sensor node sends local information about the properties of the soil surrounding it. All the information collected is sent to a central node which processes the information and takes necessary control measures towards irrigation and fertilization. The in-situ, buried sensors require an efficient transceiver system that can provide enough power to overcome the losses incurred during signal transmission in soil and also have an antenna that is small enough to maintain a compact size of the sensor.

Our previous work on soil-sensing, that uses modified quarter wavelength monopole type
electrodes as probes Pandey et al. (2013b,c), has proven that multi-frequency impedance measurements of a soil mixture have the capability to provide information about the soil moisture together with the concentration of different ions like nitrates in soil. In Pandey et al. (2013b) we presented a self-calibrating, multi-frequency dielectric sensor for combined moisture and soil ions sensing, while in Pandey et al. (2013c) we showed how dielectric-mixing models can be reasoned to analyze the multi-frequency dielectric measurements to estimate the soil moisture and ion concentrations. We have also shown that the quarter wavelength monopole antenna can be used dually as a sensor probe as well as a transmitting/receiving antenna. This was achieved by separating the low-frequency sensing path from high frequency transmission path using a diplexer.

A limitation of the monopole electrodes is their size, which at carrier frequency of 915 MHz must be 8.2 cm standing vertically on a horizontal ground plane (Note we chose a carrier frequency of 915 MHz, as although a lower frequency will offer a superior range, the size of the antenna would become even larger). An antenna height of 8.2 cm is clearly not very convenient for underground applications as the in-situ nature of agricultural application calls for a small embedded antenna. Integration of our soil sensors with microstrip antenna, a flat structure, can make the sensors more compact and more usable for in-situ operation. For wireless interfacing, planar microstrip antennas exist and are widely used owing to their small size, low cost and ease of integration. Such antennas have also been used to dual as sensing probes Soontornpipit et al. (2006) in soil-moisture sensing applications.
Microstrip patch antenna/probe combination has also been investigated as part of our own earlier research Pandey et al. (2013a). The input impedance of the microstrip patch was shown to vary with surrounding nitrate and moisture concentrations and was used to detect the changes in moisture and nitrate concentrations in soil. While flattened in vertical dimension, regular microstrip patch antennas still suffer the size issue since they are not small enough in size in the other two dimensions (e.g. 11.5 cm x 9.3 cm at 915MHz carrier frequency). The 11.5 cm x 9.3 cm size of a flattened regular patch antenna size is an improvement over a monopole, but the size is still much larger compared to the rest of the circuit.

This work presents a metamaterial inspired small flat antenna that provides a practical solution for an underground application. The main contributions of this work are:

1. A new metamaterial inspired CRLH antenna that reduces the antenna size by about 93% of the original patch antenna.

2. Application of the CRLH antenna as the sensing element by using a diplexer that allows the use of the CRLH patch as a probe at low frequencies and as transmitting/receiving antenna at higher frequencies.

3. Mapping the measured input impedance of the CRLH patch embedded in its surroundings to its complex permittivity.

4. Improvement in the accuracy of the sensor by accounting for the influence of the parasitic capacitances in our measurements.

The new antenna design along with an inbuilt self-calibrating mechanism makes our sensor suitable for underground application such as soil nitrate management or for a hand held device such as in food safety or microbial detection applications. A multi-power mode transceiver system has been designed to support the implementation of an energy efficient medium-access-control (MAC) protocol. Rest of the paper is organized as follows: Section II provides an overview of our multi-frequency impedance measurement system. Section III presents a discussion on design and fabrication of CRLH patch. Section IV presents the experimental validation...
of real and imaginary parts of impedance measurements over the frequencies of 1-40MHz. Section V concludes the paper.

4.2 Overview: Multi-frequency Impedance Measurement

We have recently designed and tested a dielectric measurement based soil impedance sensor that can sense at multi-frequencies (hence accurate & reliable), is self-calibrating (hence robust), possesses wireless interface (hence can be located in-situ), and is also energy-efficient Pandey et al. (2013b). The sensor architecture, consisting of probe and antenna, directional couplers, phase locked loop (PLL), amplitude and phase detector, switches/diplexer, microprocessor & transceiver, is shown in Fig. 4.2.

Upon startup, the microprocessor programs the I2C interface of the programmable PLL to generate a signal of known frequency. The frequency of the probing signal is chosen in the range of 1-30 MHz, and is chosen so that a significant variation in real and imaginary part of the soil impedance can be observed. While the lower limit of 1 MHz on frequency is put by the architecture of the sensor, the upper limit of 30 MHz was obtained experimentally as above this value the soil reactance becomes close to zero. A slight increase in this value can provide more data points to analyze but beyond that no useful information on soil ionic concentration can be extracted from it. The probing signal is sent through the transmission line to the SP6T switch, which is programmed by the microprocessor to select among a set of known loads plus the unknown soil-sample load. The incident and reflected signals to and
from the load are captured using the directional couplers and are passed on to a detector which calculates the amplitude and phase of each signal and passes this information to the microprocessor for further processing and transmission via antenna. These values are received by the microprocessor through an in-built 12-bit ADC. In the calibration mode, when the loads are of known values, these values are used to calculate the calibration parameters that correlate the reflection coefficients (ratio of reflected to incident) measured at the couplers to those at the load through a 3-parameter bilinear transform. In the measurement mode, when the load is the soil-sample, these calibration parameters are used to find out the reflection coefficient for an unknown load from its value measured at the directional coupler, through the same bilinear transform whose parameters were determined in the calibration mode. The reflection coefficient value is then used to determine the unknown load impedance that contains the information about the soil contents (moisture and nutrients).

The resistive versus reactive soil impedance measurements over 1-30 MHz by our sensor are shown in Figs. 4.3 and 4.4. The accuracy of our in-situ sensor is confirmed against the measurements from a lab equipment, a network analyzer, HP8714ES (plots also shown in the same figures). A more than 90% accuracy over the range of 3-30 MHz in soil reactance was observed.

Figure 4.3 Measured soil resistance.
4.3 CRLH patch Antenna/Sensor-Probe

The propagation constant for a signal traveling in soil is given by:

$$\gamma = \sqrt{j2\pi f \mu (2\pi f (\epsilon'' + j\epsilon))} = \alpha + j\beta$$ (4.1)

where $f$ is the transmission frequency of 915 MHz, $\alpha$ is the attenuation factor while $\beta$ is the phase constant. On solving this equation for $\alpha$ we get:

$$\alpha = 2\pi f \sqrt{\frac{\mu \epsilon}{2} \left( 1 + \frac{\epsilon''}{\epsilon'}^2 - 1 \right)},$$ (4.2)

where $\epsilon'$ and $\epsilon''$ are the real and imaginary parts of soil permittivity. It can be observed that the attenuation factor $\alpha$ increases linearly with frequency (and so loss exponentially with frequency). Thus, increasing the frequency, $f$, increases the losses in the transmission signal and decreases the antenna range. On the other hand, lowering the frequency (to counter path losses) increases the width, $W$ as well as the length, $L$ of a patch antenna as can be seen from the equations (4.3) and (4.4) in which $f$ appears in the denominator Balanis (2005).

$$W = \frac{c}{2f \sqrt{\epsilon' + 1}}.$$ (4.3)

$$L = \frac{c}{2f} + 2\Delta L.$$ (4.4)
Here, $\epsilon_r$ is the relative permittivity of the substrate and $\Delta L$ is the length correction factor given by:

$$\Delta L = 0.412h \frac{\epsilon_{eff} + 0.3\frac{W}{h} + 0.264}{\epsilon_{eff} - 0.258\frac{W}{h} + 0.8}. \tag{4.5}$$

In (4.5), $h$ is the height of the dielectric substrate and $\epsilon_{eff}$ is the effective permittivity due to multiple media (substrate dielectric and air) involved, and is given by:

$$\epsilon_{eff} = \frac{\epsilon_r + 1}{2} + \frac{\epsilon_r - 1}{2} \left[ 1 + 12\frac{h}{W} \right]^{-\frac{1}{2}}. \tag{4.6}$$

Since $\sqrt{\epsilon_r + 1}$ appears in denominator of (4.3), it seems that the size of the antenna can be reduced by using a very high permittivity substrate. But a problem is that the antenna efficiency also goes down with increasing substrate permittivity.

For the transmission frequency of 915 MHz, the length and width were calculated to be 11 cm and 8.8 cm respectively for a relative substrate permittivity of 3.55 and substrate height of 0.813 mm. The ground plane size was decided based on the analysis presented in Kumar and Ray (2003) as: $(L + 6h) \times (W + 6h) = 11.5 \text{ cm} \times 9.3 \text{ cm}$. Since the sensor circuitry can be designed to fit into a relatively smaller size (7 cm $\times$ 5 cm for our on-board design), the antenna size is the limiting factor in overall sensor size. To address this technological challenge, we look beyond the standard materials, towards the so called metamaterials.

Metamaterials are specially constructed designs which offer electric and magnetic properties opposite of materials found in nature such as negative permittivity and permeability. This creates a possibility of engineering a small-sized metamaterials matching network between the antenna and the surrounding medium so that the energy stored in the near field is radiated away. Size improvements of factor greater than 10 compared to standard antennas have been observed in Christophe Caloz and Rennings (2008), Richard W. Ziolkowski and Lin (2011), Ziolkowski and Erentok (2006). Authors in Christophe Caloz and Rennings (2008) discuss a composite right-left handed (CRLH) transmission line based antenna, with potentially improved efficiency for small patch antennas. Another such implementation has been reported in Zhou Cheng and lian gang (2011). In this paper, we present another design based on a standard CRLH structure Christophe Caloz and Rennings (2008) that can also be dueled as an underground sensing element (see Fig. 4.5 and its fabrication in Fig. 4.16).
Figure 4.5  Dimensions of the CRLH patch.

Figure 4.6  Unit cell structure for a right handed transmission line.

Resonance in CRLH type antenna can be understood by considering the unit-cell structure in a small patch resonating structure. For a regular transmission line, the well-known distributed parameters structure is depicted by a series inductance followed by a shunt capacitance as shown in Fig. 4.6. Such structure supports only the right-handed wave propagation which means that the phase shift observed in an incident signal along the length of structure is positive Liao et al. (2009). In a CRLH structure, this phase shift can be either positive or negative due to the effect of apparent negative permittivity and permeability in the structure. One type of structure that can achieve this apparent negative permittivity/permeability is shown in Fig. 4.5, with its distributed parameter model depicted in Fig. 4.7, which contains additional series capacitances and shunt inductances. For such a structure, the series impedance is given
by:

\[ Z_{\text{series}} = j(2\pi f L_s - \frac{1}{2\pi f C_s}) \]  \hspace{1cm} (4.7)

Similarly, shunt reactance is given by:

\[ Y_{\text{shunt}} = j(2\pi f C_{sh} - \frac{1}{2\pi f L_{sh}}) \]  \hspace{1cm} (4.8)

It has been shown in Christophe Caloz and Rennings (2008) that better efficiency for a CRLH antenna is achieved when series and shunt parts of the structure resonant at same frequency which is given by:

\[ \omega_{\text{resonant}} = 2\pi f_{\text{resonant}} = \frac{1}{\sqrt{L_s C_s}} = \frac{1}{\sqrt{L_{sh} C_{sh}}} \]  \hspace{1cm} (4.9)

In our antenna, series capacitance is introduced by the inter digitized finger capacitor while shunt inductance was realized using a meander shaped microstrip stub (see Fig. 4.5 and its fabrication in Fig. 4.16). The CRLH antenna structure was simulated using ADS (Advanced Design System, Agilent Technologies) software and resonance was observed for both series and shunt structures at the desired frequency of interest (915 MHz).

The antenna is fabricated on a pc board (see Fig. 4.16) with relative substrate permittivity 3.55, thickness 0.813 mm and dielectric loss tangent of 0.002 (Rogers R4003C laminate). From simulations it was observed that although 5% improvement in efficiency can be achieved by reducing dielectric constant to 2, the size requirement goes up by 60%. The thickness was
chosen to fit the antenna structure in a compact volume in order to keep a small size for the overall sensor. Hence, the chosen values give a good balance between efficiency, availability and size for a patch structure. The metal on the top has a thickness of 0.0355 mm and a conductivity of 5.8e7 S/m (copper). The dimensions of the antenna are shown in Fig. 4.5 while Fig. 4.16 shows the actual fabrication. Resonant frequency of 915 MHz is demonstrated in the reflectivity plot of Fig. 4.9. The antenna dimensions are 3 cm × 2 cm which is 93.3% smaller in area than the standard patch antenna of size 11.5 cm × 9.3 cm).

Table 4.1 Signal attenuation for a sensor buried 1m below ground

<table>
<thead>
<tr>
<th>Real relative permittivity</th>
<th>Imaginary relative permittivity</th>
<th>Attenuation in soil (dB)</th>
<th>Range in air above soil (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>1</td>
<td>-31.25</td>
<td>4017</td>
</tr>
<tr>
<td>5</td>
<td>2</td>
<td>-35.75</td>
<td>2392</td>
</tr>
<tr>
<td>5</td>
<td>3</td>
<td>-40.03</td>
<td>1460</td>
</tr>
<tr>
<td>5</td>
<td>4</td>
<td>-44.05</td>
<td>919</td>
</tr>
<tr>
<td>5</td>
<td>5</td>
<td>-47.80</td>
<td>597</td>
</tr>
</tbody>
</table>

Figure 4.9 Return loss for the fabricated antenna.

The built in transceiver system in our sensor can transmit at a maximum power of 25 dBm while the receiver has a sensitivity of -110 dBm, meaning a 135 dB path-loss can be tolerated. The range of our antenna can be calculated using Frii’s equation for path loss:

\[
P_r(dB) = P_t(dB) + G_t(dB) + G_r(dB) + 20log_{10}\left(\frac{c}{4\pi fr}\right) + 20log_{10}(e^{-\alpha r})
\]  

(4.10)

where \(P_r\) and \(P_t\) are received and transmitted powers, \(G_t\) and \(G_r\) are transmitting antenna gain and receiving antenna gain, and \(r\) is the distance between transmitting and receiving antenna.
In our setting, $P_r = -110$ dBm, $P_t = 25$ dBm, $G_r = G_t = 0$ dBm, $f = 915$ MHz, and $\alpha$ is determined using Equation 4.2 by plugging $\epsilon'$ and $\epsilon''$ values from Table 4.1, which also lists the calculated ranges $r$, assuming that antenna is buried 0.25 m below the soil surface.

The real part of soil permittivity is dominated by saline water concentration, whereas the imaginary part of the soil permittivity is governed by the concentration of free ions. According to Chighladze et al. (2011), maximum value of nitrate concentration in a typical clay-loam field is 200 mg$^{-1}$L$^{-1}$ which results in a soil permittivity of approximately $5 + j5$ at 20% by volume moisture content for frequencies above 14 MHz. Hence, we have used this value as the worst case scenario for making range calculations; the other range values are calculated at the same moisture level but lower nitrate concentrations so the soil permittivity ranges from $5+j$ to $5+5j$, yielding range values between 4017 m and 597 m. It can be seen that even for large values of nitrate concentration in soil (which cause the imaginary part of relative permittivity to rise) while keeping the same moisture level, the antenna range remains 597 m. Thus, the proposed antenna can be effectively used to communicate with above the air satellite/base station located in the field while the sensors are buried 0.25 m below the ground.

4.4 Test Results

4.4.0.12 Comparison with Network Analyzer

The soil impedance was measured by our on-board sensor Pandey et al. (2013b) and for comparison of its accuracy also by a Network Analyzer (HP8714ES). The CRLH patch sensing element, along with its surrounding medium, namely soil, presents itself as a load impedance to the sensor. The measurement data recorded by the on-board sensor is transmitted to a receiver which first calculates the calibration parameters and using those, calculates the unknown load of the surrounding soil. The imaginary part of impedance measured using the on-board sensor showed a better than 85% match with those measured using the network analyzer in the range 1-40 MHz while real part showed accuracy better than 70% for frequencies above 15 MHz. (see Figs. 4.10 and 4.11). The lack of accuracy in real part could be due to non-uniform distribution of ions in the vicinity of the sensor during two sets of measurement. Also the higher range of
1-40 MHz as compared to the previous 1-30 MHz provides a larger dataset so that a more informative analysis on soil ionic concentration can be carried out.

**4.4.0.13 Admittance variation with varying moisture and nitrate conditions**

Figs. 4.12 and 4.13 show the variation in patch admittance with changing values of sodium nitrate solution. A 100 mili molar sodium nitrate solution was added in steps of 4% by volume increments to the soil that had sensor with the patch, acting as a probe (as well as antenna, at another frequency), buried into it. It was observed that the measured conductance (reciprocal of the real-part of impedance) of the patch increased as the concentration of sodium nitrate was increased in soil, whereas there was a much smaller variation in the susceptance (reciprocal of the imaginary-part of impedance) value. This demonstrates that the accurate measurement of soil impedance (equivalently, admittance) using microstrip patch sensing probe at multiple frequencies has the potential to successfully detect changes in ionic concentration in soil. The dielectric mixing models Sihvola (1999) that determine the permittivity of a mixture as a function of the composition and content of the mixture, together with the dielectric relaxation models Santamarina et al. (2001) that determine the permittivity as a function of the frequency can be employed to estimate the concentrations of moisture versus nitrates versus air in the soil from the measurements, as is the case in Pandey et al. (2013d).

**4.4.1 Estimation of water and nitrate using CRLH sensing element**

As done in previous chapter, the estimated fraction of solution is shown in Fig. 4.14 and compared with the actual amount of solution added. High inaccuracy is observed for high concentrations (more than 20%). In the low-range (less than 8%) the maximum error was found to be less than 20%. One factor that causes larger error for lower concentrations is that the initial amount of solution acts as bound Santamarina et al. (2001) and hence needs to be treated separately. On assuming a 4% fraction of bound solution, the results are within 10% of the actual value of the saline water fraction.

The conductivity of a dielectric mixture has been shown to increase with increasing concentration of saline solution. The conductance of soil dielectric mixture is proportional to the
imaginary part of dielectric permittivity. For a dielectric mixture with saline solution as a constituent, all the conductivity is provided by the moving ions in the saline solution. The other constituents, air and soil bulk are non conducting. Hence, once we have an estimate of fractional volume of saline solution, we can predict the amount of ions present by relating the conductivity at a single frequency with the molar concentration of ions present in the overall volume. The increase in conductivity at 1 MHz with increasing molar fraction on sodium nitrate is shown in Fig. 4.15. At concentrations above 20 µM, the conductivity is linear. Since a 50 mM solution of sodium nitrate was used for the experiment, the lower concentration of nitrate implies a lower concentration of fractional saline water volume which in turn means a larger fraction of saline water being in the bound form. Since conductance is directly proportional to the concentration of ions in water, a linear relation between conductance and concentration is expected. The measured value of average conductance for complete frequency range is quite close (within 5% of margin) to the expected linear model. Slight deviation is observed as soil is not a homogeneous solution and saline water is not uniformly distributed.

4.5 Antenna Characteristics

4.5.0.1 Simulations on metamaterial inspired patch antenna

A Metamaterial based Composite-Right-Left Handed (CRLH) transmission line antenna has been shown in Figure 4.16. This planar antenna has been designed to be built on a pcb with substrate permittivity 3.55, thickness 32 mils and dielectric loss tangent of 0.002. The metal on the top has a thickness of 1.4 mils and a conductivity of 5.8e7 S/m. It has been designed to resonate at the desired transmission frequency of 434 MHz as shown in the reflectivity plot (Figure 4.17). The small size of the antenna makes it ideally suited for in-situ sensor-network application.

To calculate the 3-D Electromagnetic radiation properties on the antenna, Finite element Method has been employed using ADS Momentum software. The antenna shows a maximum directivity of 5 dB (Figure 4.18). This means than with proper orientation the underground communication can be further increased by a distance which corresponds to 10 dB path loss
(approx. 1.1 meters).

4.6 Conclusion

An on-board self-calibrating multi-frequency dielectric sensor with small sized planar patch for sensing as well as wireless interfacing was designed, fabricated and validated against a network analyzer. The sensor was shown to accurately measure the soil impedance at multiple frequencies over 1-40 MHz, with less than 15% error in reactance when compared to a benchtop network analyzer (HP8714ES). The impedances measured by the sensor is useful in estimating the contents of individual ions and moisture in soil. This work improves upon our previous work on underground soil moisture and nitrate sensing Pandey et al. (2013c,d) by reducing by almost 93% antenna dimensions thus allowing the design of a compact overall sensor size, making it suitable for field-deployment and hand-held applications.

Currently, we are working towards developing a model to relate the input impedance of this CRLH patch sensor to the surrounding permittivity value. Such models can be applied to data obtained in this work to determine the permittivity of the surrounding soil, and the permittivity values at multiple frequencies can then be used to estimate ionic concentrations Pandey et al. (2013c,d).
Figure 4.10  Comparison of measured input reactance of patch buried in soil measured with on-board sensor and network analyzer.

Figure 4.11  Comparison of measured input resistance of patch buried in soil measured with on-board sensor and network analyzer.
Figure 4.12  Conductance variation with varying sodium nitrate solution concentration.

Figure 4.13  Susceptance variation with varying sodium nitrate solution concentration.

Figure 4.14  Estimation of solution volume fraction
Figure 4.15  Estimation of sodium nitrate molar content

Figure 4.16  A CRLH patch antenna.

Figure 4.17  Return loss for the CRLH patch antenna.
Figure 4.18  Maximum Directivity for the CRLH patch antenna.
CHAPTER 5. Conclusion and Future Work

5.0.1 Detection of multiple ions in soil (Nitrate and Chloride)

From the experiments done on detecting concentration of saline water solution in soil, we observed that the dielectric mixture models provided close estimation to actual saline water concentrations for low concentrations of salts. This result can be extrapolated in order to determine presence of multiple ions in water. In Anderko and Lencka (1997), it has been proposed that for low concentrations the specific conductance of a saline solution with 2 types of salts is given by:

\[ \kappa(K) = a_1 \kappa_1(K) + a_2 \kappa_2(K) \]  

(5.1)

where \( a_1 \) and \( a_2 \) are the fractions of salts with specific conductances \( \kappa_1 \) and \( \kappa_2 \) respectively and \( K \) is the concentration at which all 3 specific conductances are measured. For high concentrations however, an analytical expression does not exist and the dynamics of the reaction depends on dissociation constant of the reactions. Since typical nitrate and chlorides concentration in soil is of the order 100 mgL\(^{-1}\) (Chighladze et al. (2011)), we can assume approximate infinite dilution and hence above mentioned formula should hold true. Moreover, the ionic relaxation times increases with increase in size of ion (Santamarina et al. (2001)). Hence, according to debye model for permittivity at multiple frequencies

\[ \epsilon = \epsilon' - j\epsilon'' = \epsilon_{\infty} + \frac{\epsilon_{\infty} - \epsilon_{\infty}}{1 + j\omega\tau} \]  

(5.2)

nitrate and chloride ions will have different permittivity as well as different slopes for permittivity variation with frequency. 50 mM of nitrates and chlorides solution were added and concentration of each ion was increased in steps of 25 ml (5% by volume. The resulting change in soil conductivity is shown in Fig. 5.1. It was observed that at low concentrations, conductivity change with frequency is not observable but is observable at higher concentrations.
Figure 5.1  Conductivity change with increasing concentration of multiple ions in soil.

Un fortunately, the concentration of ions in soil is typically not high enough to detect the separation in ion conductivity at different frequencies. This leads to an interesting extension to the existing problem. With multiple ions present in soil, we need to first separate out the different ions using a method such as electrophoresis and then use the existing sensors to detect water and ions concentrations.

5.0.2 Field Testing

The previously discussed On-board Sensor and microstrip patch antenna (metamaterial based or normal) has been arranged into 2 layers. The upper layer is the patch antenna (with 3 layers: radiating top layer, dielectric filed between top and bottom layer and the bottom ground plane ) with a coaxial input. The other end of this coaxial connection is connected to the pcb board that contains the csensor electronic circuit. A conceptual diagram of such sensor is shown in figure 3.10. The patch antenna acts as 'external capacitor’ sensor at low frequency. At these frequencies, the sensor acts as a parallel plate capacitor with a known dielectric filled between the plates. Due to the fringing effects, a part of the electric field is spread in the region surrounding the antenna. Hence, any change in the nature of dielectric surrounding the antenna will lead to a change in the capacitance value of the sensor. Such fringing effects have been discussed in the past in Palmer (1937), Bai and Lonngren (2002),REINEIX and JECKO
(1989). A High frequency Structure Simulator (HFSS) software simulation shows the effect of changing surrounding dielectric permittivity on imaginary part of the impedance of a parallel plate capacitor.

For field testing, these sensors will be buried approximately a ft. under ground which is a nominal depth at which roots of the plants extract moisture and nutrients from the soil mainly to estimate the range of the antenna as the impedance measurements have already been verified in lab.

5.0.3 SoC Implementation

Implementing a System on Chip (SoC) of the sensor will help reduce the size as well as power consumption. The individual components of the architecture like PLL (Phase-Locked-Loop) for frequency synthesis, directional couplers, phase and amplitude detectors and switches can be combined together on one SoC design. The transmission and data processing can be done by the microprocessor which interacts with SoC based design by the programming the PLL divider values that changed the output frequency from PLL. This signal reflects back from sensor and is coupled to the inputs of phase and amplitude detector block. Design of each individual block and optimizations of area, noise and power are another set of research problems which can be looked into.

This work has been able to achieve the goals of developing an in-situ, underground, self-calibrating, wireless, impedance sensor. Such impedance sensor find application in many fields other than soil-content sensing. In any dielectric mixture, the change in concentration of a constituent can be detected by change in impedance of the sensing element buried in that mixture. We have also developed a small metamaterial-inspired antenna and shown that such antenna can be efficiently doubled up as a small sensing element used to detect soil contents. We have used dielectric mixing models to estimate the contents of soil. The impedance values that we measure using our sensor can be fitted into other mathematical models (such as in Chighladze et al. (2011)) as well.


