Determination of the Dielectric Constants of Hydrated Cement Paste and Cement Mortar Using a Contact Coaxial Probe

BY

Ibrahim Cagatay Solak
Bachelor of Science, (Middle East Technical University)

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
FOR THE DEGREE OF MASTER OF SCIENCE IN ENGINEERING
DEPARTMENT OF CIVIL AND ENVIRONMENTAL ENGINEERING
UNIVERSITY OF MASSACHUSETTS LOWELL

Signature of the Author..
Department of Civil and Environmental Engineering
(June), 2011

Signature of Thesis Supervisor ....
Tzu-Yang Yu
Assistant Professor

Committee Member Signature...
Professor Donald Leitch
Department of Civil and Environmental Engineering

Committee Member Signature...
Professor Susan Faraji
Department of Civil and Environmental Engineering

Determination of the Dielectric Constants of Hydrated Cement Paste and Cement Mortar Using a Contact Coaxial Probe

BY

Ibrahim Cagatay Solak
Bachelor of Science, (Middle East Technical University)

ABSTRACT OF A THESIS SUBMITTED TO THE FACULTY OF THE
DEPARTMENT OF CIVIL AND ENVIRONMENTAL ENGINEERING
IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
FOR THE DEGREE OF
MASTER OF SCIENCE
UNIVERSITY OF MASSACHUSETTS LOWELL
2011

Thesis Supervisor: Tzu-Yang Yu
Title: Assistant Professor

Dielectric properties of construction materials have become valuable information in the condition assessment of civil infrastructure using microwave and radar nondestructive evaluation (NDE) techniques and sensors. Multi-phase dielectrics are usually encountered when structures are made of cementitious composites (e.g., Portland cement concrete). In this thesis, the dielectric dispersion of cement paste and cement mortar samples in the frequency range of 0.5 GHz to 4.5 GHz was studied. Cement paste and cement mortar samples of various water-to-cement (w/c) ratios (0.35, 0.40, 0.42, 0.45, 0.50, 0.55) were manufactured and their dielectric constants were measured by a coaxial probe at room temperature 23 ^{o}C ± 2 ^{o}C with 25 % - 30 % relative humidity. Contact dielectric measurements were collected at different locations on each sample to study the dielectric heterogeneity of the cement paste samples. It was found that the measured relative complex permittivity varies even within one cement paste panel. The measured relative dielectric constant decreases with the increasing w/c ratio and measurement frequency. Change in dielectric constant due to the removal of evaporable water by oven drying cement paste samples was observed. The effect of the fraction of sand in cement mortar to its dielectric constant was investigated and modeled.

Acknowledgements

I would like to offer my deepest appreciations and utmost gratitude to my advisor, Dr. Tzu-Yang Yu, whose enormous and endless efforts have enhanced my ability beyond any expectations.

I also would like to thank my thesis committee members, Dr. Susan Faraji and Prof. Donald Leitch for their valuable comments and inputs.

Many thanks go to the National Institute of Standards and Technology (NIST) Technology Innovation Program (TIP) for their financial support during my graduate study.

I wish to express my sincere gratitude to Burak Boyaci, Samed Maltabas, O. Kubilay Ekekon and Mena Sliman for their support and help during the course of this research.

Finally, I wish to express my forever gratefulness to my mother, Sultan Solak, and my father, Ali Osman Solak, who always make me proud of being their son in every breath of my life. Their unmeasured and unquestionable love always gives me the courage to overcome every obstacle in life. I also wish to gratefully thank my brothers, Cagri Solak, Tugrul Solak and my sister, Aslihan B. Solak for their support.

Contents

List of Figures

List of Tables

Chapter 1

Introduction

Deterioration of structures is an inevitable process that determines the lifespan of structures. Usually structures are designed to have a lifespan from 30 years to 120 years depending on the type of the structure. In order to keep the structure operational until the end of design lifespan or to extend the service life of the structure, precautions should be taken. Both to ensure public safety and feasibility the structure may be repaired or strengthened. The need for repairing or strengthening may be determined by nondestructive evaluation (NDE, or nondestructive testing - NDT). NDE is an interdisciplinary field of study concerned with the analysis techniques and measurement technologies to determine the health of structures. The aim is to determine the physical and mechanical properties and to estimate the serviceability and sustainability without damaging the structure. Due to limitations, these parameters are measured using indirect methods. Depending on the NDE method the parameters that can be measured varies. The most common NDE methods and their corresponding measurement parameters are [37] ;

- **Optical Methods**; EM waves in optical spectrum
- **Acoustic Methods**; Mechanical waves
- **Thermal Methods**; EM waves in thermal radiation spectrum
- **Radiographic Methods**; X-ray, Gamma rays, neutrons
- **Magnetic and Electrical Methods**; Magnetic field and electrical field
- **Microwave / Radar Methods**; EM waves in microwave spectrum and radio frequency range

In most cases the evaluation of the condition of a structure may require using more than one NDE method. While observing a reinforced concrete structure, optical methods (visual inspection) gives us information about the outer surface of concrete. This method is not applicable to observe the subsurface conditions of concrete or the reinforcing steel conditions. The use of a method that is capable of evaluating the subsurface conditions is very important for NDE.

Magnetic and electrical methods are very applicable especially when concrete structures are being evaluated. Concrete, which is a dielectric material, allows EM waves to propagate inside. The EM waves change their properties depending on the medium in which they travel. By collecting the reflected or transmitted waves from a medium, in our case that medium was cementitious composites, the properties of the medium can be estimated by comparing the parameters (magnitude, phase, and group delay) of reflected and transmitted waves with the original wave sent. In this way subsurface analysis can be achieved by electrical measurements with the help of a network analyzer.

By using microwave and radar methods anomalies inside a medium can be detected. The size and the location of an anomaly can be calculated by using finite difference time domain methods. But to be able to obtain reliable and accurate results, one needs to know the dielectric properties of the medium being tested. The speed of electromagnetic wave propagation depends on the relative dielectric constant of the medium.

1.1 Research Objective

Dielectric properties of cementitious materials depend on their components. Major ingredients of cementitious materials are Portland Cement, fine aggregate (sand), coarse aggregate and water. During the hydration process, the structure of the cementitious materials are formed by hydrated cement, sand, fine aggregate, coarse aggregate and evaporable water and air within the voids. The proportion of these materials after hydration depends on many factors such as the quantity of a material used, the type of aggregates, the method of casting and curing, the age of the cementitious material and environmental conditions such as relative humidity and temperature. Due to the high number of parameters, the dielectric constant of cementitious materials are variable. This thesis aims to investigate the effects on the dielectric constant by influencing parameters such as w/c ratio, sand and the evaporable water and air content in voids. The characterization of cementitious materials this way, will provide a more accurate and reliable database for cementitious materials, which will be very helpful for radar measurements.

1.2 Thesis Approach

In this thesis we observed the variations on dielectric constant caused by different proportioning of water, air and sand. To achieve the objective, cement paste and cement mortar samples with varying w/c ratio and s/c were cast. The porosity difference caused by using different w/c ratios, and oven drying of the samples allowed us to detect the effect of evaporable water and air within the voids. The effect of sand was measured by casting specimens with varying s/c ratios. Contact coaxial measurements were used to measure the dielectric constant.

The reliability of the measurements has been investigated by using a statistical approach. Monte Carlo simulations have been used to analyze the data set. Also the dielectric heterogeneity of samples has been studied by statistically analyzing the collected data set statistically.

1.3 Organization of the Thesis

The organization of this thesis is as specified below.

Chapter 2 reviews the applied methods for determining the dielectric properties of cement paste and cement mortar.

Chapter 3 introduces the methodology and equipment for our measurements.

Chapter 4 includes the measurement results for cement paste samples and the effect of moisture on the dielectric constant.

Chapter 5 describes the effect of sand on the dielectric constant of cement paste.

Chapter 6 describes the reliability of the measurements using Monte Carlo simulations.

Chapter 7 summarizes the research findings.

Chapter 2

Literature Review

Dielectric properties of cement paste have been studied by many researchers with different methods. Among the most used ones are;

- **Parallel Plate Capacitor** - The capacitance of a material is related to its dielectric constant. So by measuring the capacitance, one can obtain the dielectric constant of a material with the parallel plate capacitor method. In this method the specimen is located between two parallel plate electrodes as a capacitor, and an electrical field is applied across the plates. The measured capacitance of the dielectric medium between the plates is a function of dielectric constant with the relation [7];

  $$C = \frac{\epsilon_r' A}{d} \tag{2.1}$$

  where,

  C = Capacitance

ϵ_r' = Dielectric Constant

d = Plate seperation

- **Coaxial (Transmission Line Theory)** - The dielectric constant can be derived by measuring the phase and amplitude of a reflected microwave signal from a sample placed against the end of a coaxial line. The electromagnetic waves are transmitted to the specimen through coaxial cable and the waves hit the specimen and are reflected back [31].
- **Waveguide (Transmission Line Theory)** - A waveguide is very similar to coaxial probe that conveys electromagnetic waves between its endpoints. The sample may be located inside the waveguide or may be at the end of the waveguide. By measuring the the phase and amplitude of the reflected signal, the complex permittivity can be obtained [31].
- **Resonator Cavity** - The working mechanism is very similar to waveguide but blocked at both ends. The inner sides of the cavity is built from reflecting material to create infinite reflection conditions for electromagnetic waves. When the wave has the same resonant frequency as the cavity, high intensity wave can be created. The specimen is located inside the cavity and the reflected and transmitted waves can be measured. When a dielectric material is introduced into the cavity, the resonance frequency shifts (Δ f) and the quality factor Q of the cavity changes. (Δ f) is considered to be correlated to the dielectric constant and Q is associated with dielectric loss [14].

- **Free Space** - A pair of horn antennas and a network analyzer connected to the horn antennas with coaxial cables are needed for free space measurements. The sample is located between the antennas and the reflected waves (S_{11}) and transmitted waves (S_{21}) are measured with the antennas. This method is a non-contact method that provides complex permittivity from the transmission coefficient measured [6].

A schematic for the discussed measurements is provided in Figure 2-1.

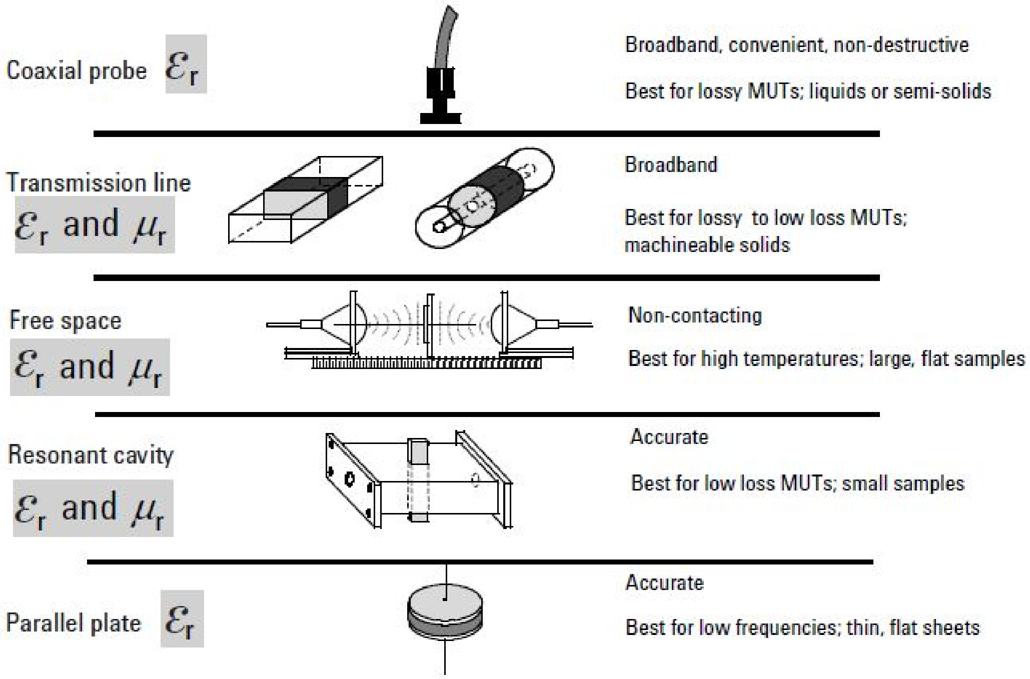


Figure 2-1: Schematic of measurements methods (Source: Agilent® Technologies Inc.)

Each method has advantages and disadvantages when the shape and the mechanical properties of the samples to be tested are considered. Some measurements methods are more suitable for liquids and powders while some are more suitable for solid samples. Also the surface of the sample is an important factor to be considered for measurements.

Scattering of the waves from rough surfaces may lead to experimental errors. Contact or non-contact conditions are also important for the method used.

2.1 Dielectric Measurements on Cement Paste

Determination of the dielectric properties of cementitious materials is not an easy task due to the high number of influential parameters. And some of these parameters are not independent from each other. For example, the effect of humidity on samples is expected to vary with porosity. So to create a controlled environment all the parameters should be kept constant other than the ones being observed. Here we summarized the research conducted on cement paste and cement mortar samples by many research groups and the parameter observed with their findings.

Some definitions of the terms used in this chapter are,

admittance: measure of how easily a circuit or device will allow a current to flow.

capacitor (condenser): a device for storing electric charge.

capacitance: the ability of a body to hold an electrical charge.

resistance: a measure of the degree to which an object opposes an electric current through it.

impedance: a measure of opposition to alternating current.

Tobio *et. al.* (1959) [29] used a closed circuit system with a capacitor and resistor to calculate the admittance of cement paste. The admittance is the resultant of variations of two magnitudes which define a condenser; capacitance and loss resistance. The measurements were collected from cement paste cylinders from Hour 0 to Hour 20 within the frequency range of 28 MHz - 60 MHz. The setting time (where peak of the admittance is observed) of 20 different cement paste brands of 5 different cement types was measured.

De Loor *et. al.* (1961) [15] prepared disc samples with different w/c (water-to-cement) ratios. Their dielectric constant was determined between 0.1 MHz and 10 MHz and at 3000, 3750, 7450 and 9375 MHz. During the measurements a condenser consisting of two electrodes (0.1 - 10 MHz), and a coaxial waveguide system (for other frequencies) was used. The samples were cured in water for one month and dried in oven for ten days. The increase in dielectric constant and loss factor was shown with the increase in moisture content during the measurements.

Hasted *et. al.* (1964) [11] measured the complex permittivity of concrete, cement mortar and cement paste panels with different moisture contents by using free space method with horn antennae. The change in dielectric constant was observed by replacing the air in the cavities with water. The increase in the amount of evaporable water leads to an increase in dielectric constant. The measurements were collected at 3 GHz and 9 GHz.

Wittmann *et. al.* (1975) [34] used free wave method to measure the complex permittivity of hardened cement paste. The microwave absorption was calculated by placing the samples between a receiving and transmitting antennae. Also a sweep generator was used to change the frequency of continuous wave from 8.5 GHz to 12.3 GHz. Using a computer program, the attenuation data was used to calculate the complex permittivity.

It was observed that as the duration of hydration increases the dielectric constant decreases. Also with the increase in temperature and moisture content, both the dielectric constant and complex permittivity increased.

Olp *et. al.* (1991) [22] used a close-ended coaxial probe with a large dielectric measurement cell to measure the complex permittivity and conductivity of cement paste and cement mortar. During the hardening stage (0-28 hours) the measurements were conducted for frequencies below 300 MHz. The variation of paste properties with frequency was observed over the range of 1 MHz to 3 GHz. Chemical admixtures were introduced to the mix during the measurements. An increase in the dielectric constant was observed over time during early setting.

Moukwa *et. al.* (1991) [20] have measured permittivity of Type I, II and III cement pastes during the first 24 hours of hydration process at 10 GHz. The sample holder consisted of a section of waveguide closed off by a thin mica sheet. The cement paste was poured into another waveguide section butting against the mica sheet. A sufficient depth of sample was used to ensure absorbtion of all signals in the waveguide, so the sample appeared as electromagnetically infinite. It was observed that Type II has the highest dielectric constant and Type III has the lowest. The difference in the hydration speed between different types of cement paste affected the dielectric constant since it is directly related to the amount of water used in hydration and evaporable water in the paste.

Shalaby *et. al.* (1995) [27] prepared cement paste samples that were cured for three days and then kept in room temperature with relative humidity of 20 %. Four samples with w/c ratios of 0.35, 0.40, 0.50 and 0.55 were prepared and measured within the frequency range of 5 - 12 GHz with a waveguide. Monopole probes were inserted into the cement paste sample (waveguide in contact with the sample) to correlate the reflection coefficient with the 28-days compressive strength of hardened cement paste (Type II Portland cement). The relation between the compressive strength and the w/c ratio was used to estimate the 28-days compressive strength from the complex permittivity (greatly affected by the w/c ratio) measurements, but it was noted that other parameters such as aggregate inclusions and chemical additives also need to be studied to make reliable predictions.

Zhang *et. al.* (1995) [38] used the transmission waveguide method within the frequency range of 8.2 GHz - 12.4 GHz to investigate fresh cement paste and blast furnace slag blend samples with and without suitable alkali activators. The measurements were conducted by filling a rectangular wave-guide section with the sample and then determining the complex permittivity from measurements of the reflection coefficient. It was observed that the higher the slag content in the blend, the higher the dielectric constant of the sample.

Al-Qadi *et. al.* (1995) [2] used a parallel plate capacitor setup for dielectric measurements of portland cement concrete using a network analyzer. The complex permittivity was measured over 28 days of moist curing within the frequency range of 0.1 - 40 MHz. The decrease of dielectric constant with measurement frequency and the curing time was reported.

Yoon *et. al.* (1996) [36] measured the dielectric response of hydrating fresh cement paste. A w/c ratio of 0.4 was used and the paste was poured into a rectangular dielectric cell right after mixing. Measurements were collected every 30 minutes up to 10 days using an impedance analyzer over the frequency range of 5 Hz to 13 MHz. A decrease in the dielectric constant has been observed with increasing hydration time.

Ford *et. al.* (1997) [4] built a physical model to represent young cement pastes. A system consisting of polycarbonate box with electrodes at each end, divided into two compartments by a polycarbonate barrier with a single hole, and filled with electrolyte solution, which was used to simulate the impedance response of young cement pastes. The barrier was used to represent hydration products, and the hole represented constriction between two adjacent capillary holes. The cement paste was described as a 3-3-0 electro composite having interconnected pore fluid and C-S-H gel phases (3D connectivity) and various other disconnected solid phases (0D connectivity). 3 stands for the inter connectivity of particles in 3 dimensions (in this case pore fluid and C-S-H gel) and 0 stands for no inter connectivity in any dimension (disconnected) between particles (in this case solid phases). By changing the dimensions of the system parameters like the barrier thickness and the hole diameter, the effect of each variable on the dielectric constant was observed and the relation between cement paste was studied. They found that factors which increase pore size (w/c) or decrease product layer thicknesses (short times of hydration) lead to higher dielectric constants.

Miura *et. al.* (1998) [19] conducted microwave dielectric relaxation measurements using the time-domain reflectrometry (TDR) method from 100 kHz to 20 GHz on Portland cement paste. The dielectric relaxation process due to hydrated water in the cement paste structure was observed at frequencies of 100 MHz and 1 MHz. The samples had a w/c ratio of 0.4 and were kept in plastic cases to avoid evaporation.

El Hafiane *et. al.* (1999) [8] used cement paste samples with 0.4 w/c ratio and 10 hours of moist curing time. They started the measurements after moist curing and collected measurements up to 8 hours after the curing process. The measurements were carried out in the frequency range of 1 MHz to 1.8 GHz using impedance spectroscopy. Relaxation time was calculated at early stages of hydration process. They observed two relaxation phenomena at 2.2 GHz around Hour 10 and at 0.1 GHz around Hour 14.

Hu *et. al.* (1999) [12] studied the humidity dependance of the dielectric constant of cement densified with small particles (DSP) in the relative humidity range of 0 % - 93 %. DSP cement is a high strength cement produced from Portland cement, silica fume and superplasticizers. An open ended coaxial probe was used in the measurements within the frequency range of 1 MHz to 1 GHz. The w/c ratio of the specimens was 0.4. The reported values show the increase in dielectric constant with an increase in relative humidity and measurement frequency. It was indicated that the influence of salt is more prominent in the loss factor, while the effect of loading is more evident in the dielectric constant.

El Hafiane *et. al.* (2000) [9] used aluminous cement paste with w/c ratio of 0.4 and conducted the experiments within 1 MHz - 1 GHz frequency range using an impedance analyzer. Dielectric constant was found by measuring the S_{11} parameter with an open ended coaxial probe. A decrease in dielectric constant is observed when the samples are dried.

Wen *et. al.* (2001) [32] observed the effect of admixtures on the dielectric properties of cement paste. Silica fume, latex and carbon fibers were used as admixtures. Parallel-plate capacitor method was adopted in the measurements from 10 kHz to 1 MHz. Cement paste samples with w/c ratios of 0.35 and 0.23 were prepared. It was observed that, the dielectric constant of cement paste samples decreases with the addition of silica fume and steel fibers, while it increases with the addition of latex and carbon fibers.

Mubarak *et. al.* (2001) [21] used a monopole probe to estimate the w/c ratio of fresh cement paste and concrete right after the mixing process, while the mixing is still physical rather than chemical (no hydration yet) at 3 GHz frequency. It was shown that the dielectric constant of fresh cement paste varies according to the w/c ratio used in the mix design and it is possible to estimate the w/c ratio of cement paste using monopole probe. According to their results, this relation may be correlated to the 28-days compressive strength of cement paste since w/c ratio has an important role on the compressive strength.

Wen *et. al.* (2002) [33] used carbon filaments and steel fibers as stress sensors embedded in cement paste to relate dielectric constant with the stress level. The samples (w/c = 0.35) were removed from the molds kept at room conditions after 24 hours and were moist cured at room temperature with a relative humidity of 100 % for 28 days. Dielectric constant of the samples was measured using a two-probe method and a RLC (resistance, capacitance or inductance) meter at frequencies ranging from 10 kHz to 1 MHz. It was observed that dielectric constant increases nonlinearly and quite reversibly when the stress level is up to 6.4 MPa, but does not return to its exact original value when the stress is removed.

Smith *et. al.* (2002) [28] measured aluminous cement samples at the early age of hydration up to 25 hours after casting. The cement paste samples were mixed in a Perrier blender for 6 minutes with a w/c ratio of 0.4. Then the paste was cast in a cell where the temperature was controlled. Measurements were conducted by using a coaxial line in a frequency range of 1 MHz to 1.8 GHz. They observed that the dielectric constant decreases with respect to time and there is a sudden drop between Hour 3 and Hour 4. Hager *et. al.* (2004) [10] monitored the broadband complex permittivity of hydrating cement paste from initial mixing to several weeks of curing in the frequency range of 10 kHz to 8 GHz. Measurements were conducted by Time Domain Reflectrometry (TDR) dielectric spectroscopy, using an adjustable capacitance sensor embedded in the paste. They have found that as the curing time increases, the dielectric constant decreases. Xing *et. al.* (2008) [35] measured the capacitance of cementitious materials using an impedance analyzer. Dielectric constant of cement paste samples was measured using a plate condenser at 1 kHz. It is found that the addition of lead zirconate titanate (PZT) ceramic particles to cement paste samples increased the calculated dielectric constant of the sample. Makul *et. al.* (2010) [16] used a network analyzer with open ended coaxial probe to investigate the early hydration (first twenty four hours) period of cementitious materials at a frequency of 2.45 GHz. They investigated the variations in the early strength of concrete when it is heated by microwave energy. It has been investigated that the dielectric propertied are relatively high during the dormant period and than decreases rapidly. Rianyoi *et. al.* (2011) [25] investigated the dielectric and ferroelectric properties of 1-3 barium titanate (BT) - Portland cement (PC) composites which are lead-free. Impedance analyzer was used for the measurements at 1 kHz frequency. It has been observed that as the volume fraction of BT increases the dielectric constant of the composite increases. BT volume fraction was increased up to 70 % and this fraction is where the highest dielectric constant was observed. On the other hand the loss factor showed an opposite trend and the dielectric constant decreased while the BT volume increased.

2.2 Dielectric Measurements on Cement Mortar

Ding *et. al.* (1996) [3] measured the dielectric constant and electrical properties of cement mortar samples with various s/c (sand-to-cement) ratios for the first 30 hours of hydration using transmission waveguide method in the frequency range of 8.2 GHz to 12.4 GHz. In their experiment the s/c ratio ranged from 0.35 to 1.70 and the water to solid ratio w/(s+c) ranged from 0.15 to 0.40. Also, the relation between dielectric constant and compressive strength (at twenty fourth hour of hydration) was investigated but no direct relation was found.

Janoo *et. al.* (1999) [13] proposed a methodology for determining the moisture content in cement mortar samples by relating the moisture content to the dielectric constant of the samples. TDR probes were embedded in fresh cement mortar samples. The cement mortar samples were kept in a mold for 24 hours and then placed in lime-saturated water for duration of 28 days. The dielectric properties of cement mortar were collectively measured with the contributions of water, air, cement paste and sand. The effect of water was significant on the effective dielectric constant of mortar since it has the highest dielectric contribution among the materials in cement mortar.

Peer *et. al.* (2004) [23] [24] used open-ended rectangular waveguide probes for measuring the complex permittivity of cement mortar at 3 GHz. Five cement mortar cube samples were prepared with a w/c ratio of 0.50 and a s/c ratio of 0.25. Cubes were removed from the molds after 24 hours and left at room temperature with low humidity for seven months. After this process, cyclically soaking in water, mechanical loading and salt-water (salinity of 2.8 %) solution curing methods were used to determine their effects on complex permittivity. It is found that the influence of salt is more prominent in the loss factor, while the effect of loading is more evident in dielectric constant of the samples.

McCarter *et. al.* (2004) [18] studied the effect of low-lime fly ash on the dielectric properties of cement mortar. Cement mortar specimens used in the measurements were designed by a s/c ratio of 3, a w/c ratio of 0.5, and replacing the ordinary portland cement with fly ash with replacement fractions from 0.33 to 1. The measurements were conducted using an impedance analyzer in a frequency range from 1 Hz to 1 MHz. It was observed that the addition of fly ash to cement mortar led to the increase of dielectric constant.

Sagnard *et. al.* (2005) [26] conducted measurements on cement mortar using the free-space method. Network analyzer-based measurements were performed in an anechoic chamber of length of about 4 meters (the distance between the the horn antennas) in the frequency range from 8 GHz to 12.5 GHz. The samples used in the measurements had a thickness of 4.78 cm. They found that in dry conditions (no moisture within the samples), the dielectric constant was lower for high w/c ratio samples due to greater porosity of these samples.

Tsonos *et. al.* (2009) [30] used dielectric spectroscopy to investigate the complex permittivity of cement mortar using a LCR (Inductance (L), Capacitance (C), and Resistance (R)) meter in the frequency range of 10 Hz to 1 MHz. Cement mortar samples with a s/c ratio of 3 and a w/c ratio of 0.5were prepared and stored in plastic cases at room temperature and 40 % relative humidity. The imaginary part of the complex permittivity was monitored up to 39 days after casting the mortar. Their results show that both the dielectric constant and the loss factor decreases with time.

Table 2.1: Measurements conducted on cement paste by different groups

Cement Paste		
Parallel Plate Capacitor	28 MHz - 60 MHz 0.1 MHz - 10 MHz 0.1 MHz - 40 MHz 10 kHz - 1 MHz	Tobio *et. al.* De Goor *et. al.* Al-Qadi *et. al.* Wen *et. al.*
Free Space	3 GHz, 9 GHz 8.5 GHz - 12.3 GHz	Hasted *et. al.* Wittman *et. al.*
Coaxial	1 GHz - 3 GHz 1 MHz - 1 GHz 3 GHz 1 MHz - 1.8 GHz	Olp *et. al.* Hu *et. al.* Mubarek *et. al.* Smith *et. al.*
Waveguide	10 GHz 5 GHz - 12 GHz 8.2 GHz - 12.4 GHz 5 Hz - 13 MHz	Moukwa *et. al.* Shalaby *et. al.* Zhang *et. al.* Yoon *et. al.*

Table 2.2: Measurements conducted on cement paste by different groups with other methods

Cement Paste		
Time Domain Reflectometry	100 kHz - 20 GHz 10 kHz - 8 GHz	Miura *et. al.* Hager *et. al.*
Impedance Spectroscopy	1 MHz - 1.8 GHz 10 Hz - 1 GHz 1 kHz	El-Haffiane *et. al.* El-Haffiane *et. al.* Xing *et. al.*
RLC Meter	10 kHz - 1 MHz	Wen *et. al.*

Table 2.3: Measurements conducted on cement mortar by different groups

Cement Mortar		
Free Space	8 GHz - 12.5 GHz	Sagnard *et. al.*
Waveguide	8.2 GHz - 12.4 GHz	Ding *et. al.*
	3 GHz	Peer *et. al.*
Time Domain Reflectometry	Not Indicated	Janoo *et. al.*
Impedance Spectroscopy	1Hz - 1MHz	McCarter *et. al.*
RLC Meter	10 Hz - 1 MHz	Tsonas *et. al.*

2.3 Summary

In this chapter different methods for dielectric measurements have been presented. These methods are;

- **Parallel Plate Capacitor**
- **Coaxial (Transmission Line Theory)**
- **Waveguide (Transmission Line Theory)**
- **Resonator Cavity**
- **Free Space**

Each method has advantages and disadvantages due to the shape and the mechanical properties of the sample to be tested. Also a selected method may not be applicable for a desired frequency range. The work conducted by many groups in the past have been reported with the aspect being investigated and the frequency range used.

Chapter 3

Experiment Methodology and Sample Preparation

As mentioned previously there are many methods for determining the complex permittivity of cementitious materials. In our study we used Agilent® E5071C ENA Series Network Analyzer (Figure 3-1) that has a frequency range from 0.0001 GHz to 4.5 GHz.

Contact measurements were conducted using an Agilent® 85070E Performance Coaxial Probe (Figure 3-2) capable of performing coherent measurements in the frequency range from 0.5 GHz to 50 GHz.

3.1 Contact Coaxial Probe Measurements

A network analyzer consists of a signal source, a receiver and a display (Figure 3-3). Since we are conducting the measurements with coaxial probe method, we are

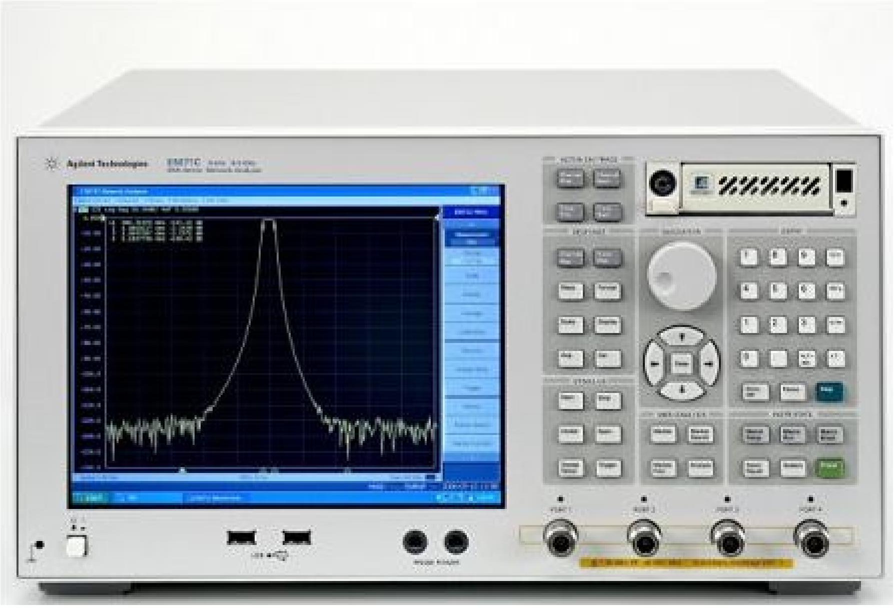


Figure 3-1: E5071C Agilent ® ENA Series Network Analyzer (Source: Agilent® Technologies Inc.)

Figure 3-2: Agilent Technologies 85070E Performance Coaxial Probe and Shorting Kit (Source: Agilent® Technologies Inc.)

using only one port of the network analyzer, meaning that both the incident signal and reflected signals are transmitted with the same gate. The transmitted waves are not measured in our case so will not be considered. The source launches a signal at a single frequency to the sample being measured. The receiver is tuned to that frequency to detect the reflected signals from the material. The measured response produces the magnitude and phase data at that frequency. The source is then stepped to the next frequency and the measurement is repeated to display the reflection and transmission measurement response as a function of frequency. The signal generator creates a signal and this signal is routed to the measured material by the test set which in our case is a coaxial cable and a coaxial probe (Figure 3-4). The reflected signal (S_{11}) is than collected again by the test set and sent to the signal receiver within the network analyzer for measurement of the magnitude and phase of the signal. In our measurements we collected data for 101 different frequencies from 0.5 GHz to 4.5 GHz with 0.04 GHz increments [1].

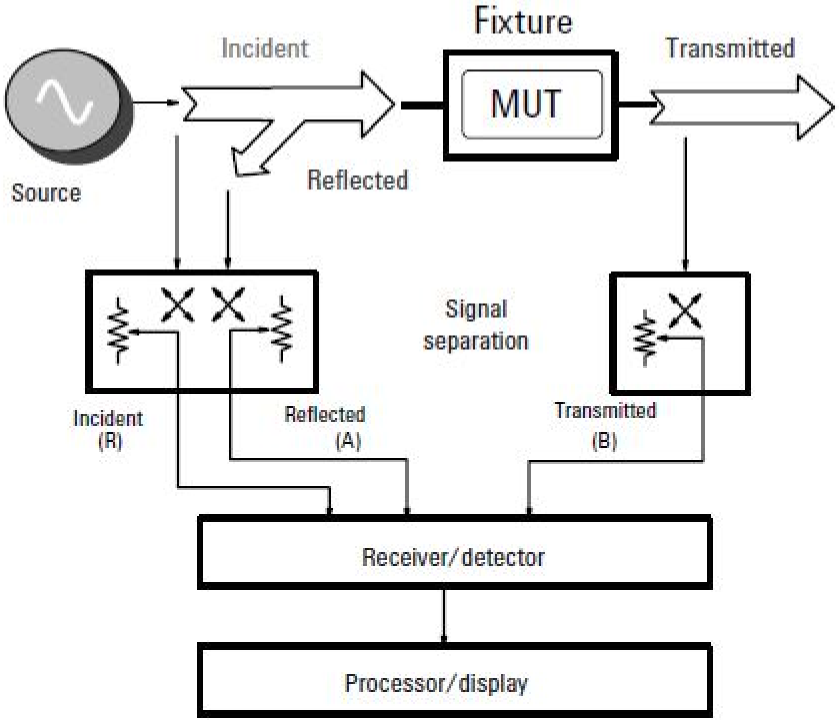


Figure 3-3: Network analyzer architecture (Source: Agilent® Technologies Inc.)

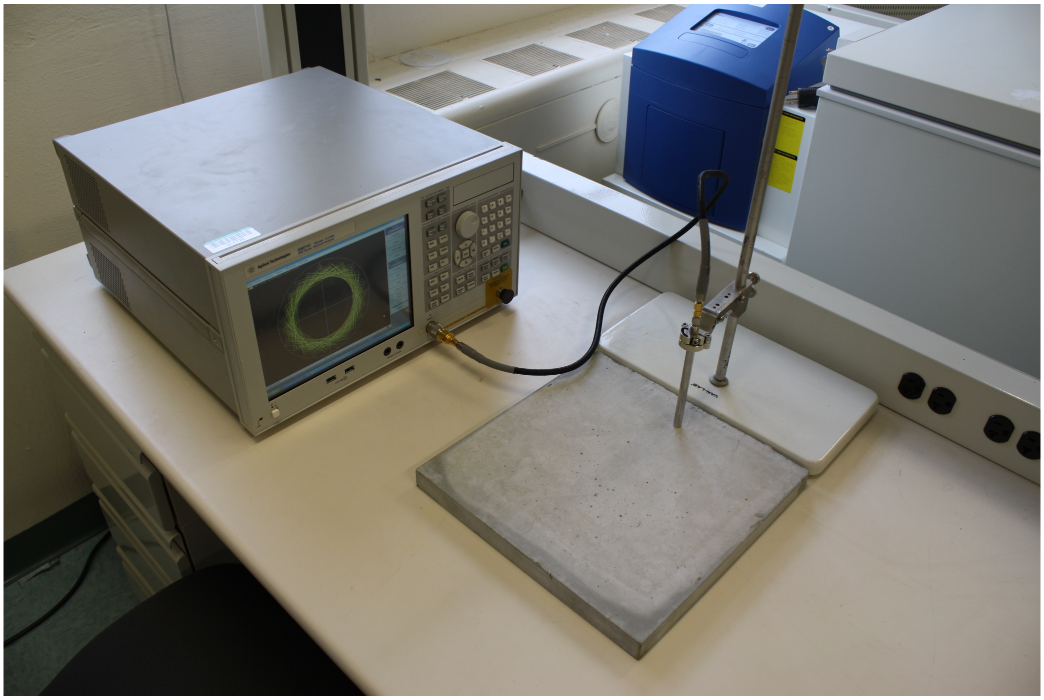


Figure 3-4: Test Set: Coaxial Cable and Coaxial Probe Connected to Network Analyzer

Coaxial cable is an electrical transmitter (transmission line) that has an inner conductor, surrounded by an insulating layer, which is again surrounded by a tubular conducting shield. Coaxial cable transmits the signal from the network analyzer to the coaxial probe. The open-ended coaxial probe (Figure 3-5) is a cut off section of transmission line. The material is measured by immersing the probe into a liquid or touching it to the flat face of a solid (or powder) material. The fields at the probe end fringe into the material and change as they come into contact with the sample (Figure 3-6). The reflected signal (S_{11}) can be measured and related to ϵ_r' [1].

Before measuring, calibration at the tip of the probe must be performed. A three-term calibration corrects for the directivity, tracking, and source match errors that can be present in a reflection measurement. In order to solve for these three error terms, three

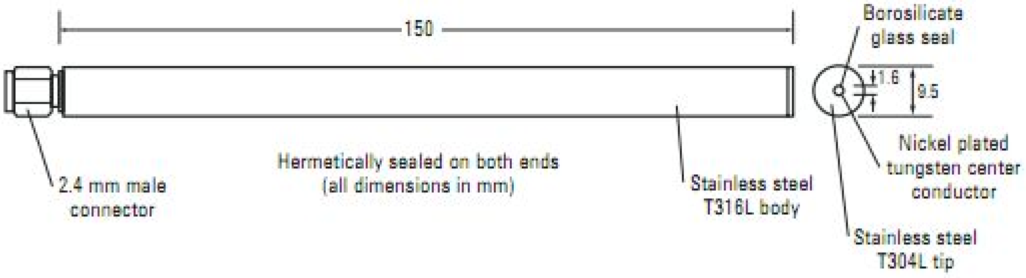


Figure 3-5: Structure of Coaxial Probe

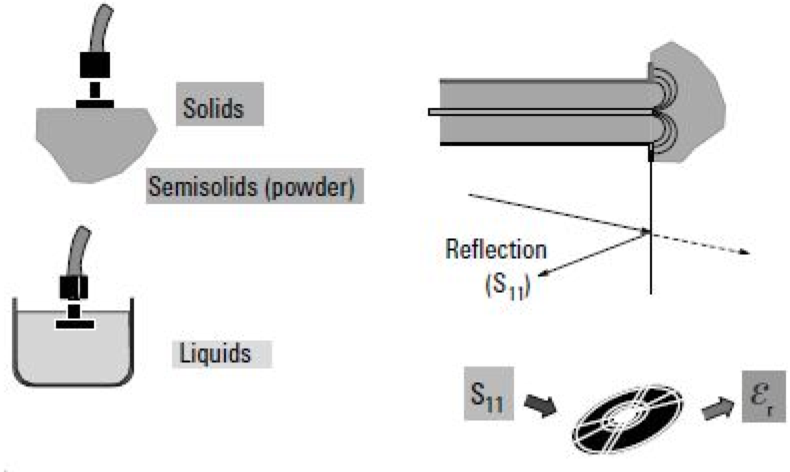


Figure 3-6: Coaxial Probe Method (Source: Agilent® Technologies Inc.)

well-known standards are measured. The difference between the predicted and actual values is used to remove the systematic (repeatable) errors from the measurement. The three known standards are air, a shorting kit, and water. Even after calibrating the probe, there are additional sources of error that can affect the accuracy of a measurement. There are three main sources of errors:

- **Cable Stability**
- **Air Gaps**
- **Sample Thickness**

It is important to allow enough time for the cable (that connects the probe to the network analyzer) to stabilize before making a measurement and to be sure that the cable is not flexed between calibration and measurement. For solid materials, an air gap between the probe and sample can be a significant source of error unless the sample face is machined to be at least as flat as the probe face. Figure 3-7 shows the desired contact condition and possible bad contact conditions. For liquid samples air bubbles on the tip of the probe can act in the same way as an air gap on a solid sample. The sample must also be thick enough to appear infinite to the probe. A simple practical approach is to put a short behind the sample and check to see if it affects the measurement results [1].

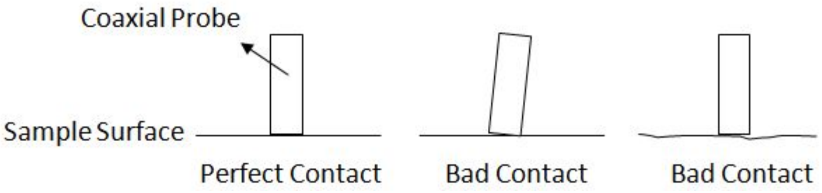


Figure 3-7: Possible contact conditions while conducting measurements using a coaxial probe

3.2 Sample Preparation

Dielectric properties of cement paste depend on the dielectric properties of hydrated cement, air and water in the pores, so while collecting the measurements any air gap between the probe and the sample as a result of non perfect contact will increase the ratio of air of the measured medium, which will decrease the dielectric constant significantly. To achieve such contact condition, the surface of the samples must be very smooth. The samples have been cast with the mold shown in Figure 3-8. The inner surfaces of the mold have been covered with plexiglass which provides a very smooth surface when the cement paste or cement mortar hardens. Using a vibrator while the cement paste or cement mortar is still fresh prevents the formation of large air voids. Since the thickness of the mold is not large enough to insert a vibrator inside several rods with smaller diameters have been inserted inside the mold and the vibrator has been attached to the rods. This has been an effective way to remove the air voids. The surface of a cement paste panel used in measurements is shown in Figure 3-9.

Figure 3-8: The inner surfaces of molds have been covered with plexiglass

Dimensions of the samples that have been used for the experiments are 1 in.-by-1 ft.-by-1 ft (Figure 3-10). We used Quikrete® Type I Portland Cement and Quikrete® All-Purpose Sand in making our samples. Cement paste and cement mortar samples with six different water-to-cement (w/c) ratios by weight (0.35, 0.40, 0.42, 0.45, 0.50, 0.55) were cast for the measurements. The cement paste samples have been measured two times, one before the oven-drying procedure and one after the oven-drying procedure.

Figure 3-9: Surface of CP50

The sand to cement (s/c) ratio used in the cement mortar samples was 2.53. Additionally we also cast a cement mortar sample with w/c 0.50 and s/c 1.90. All samples used in our study are listed in Table 3.1 and Table 3.2.

After casting the samples, they were kept in room conditions for one day within the molds and then removed from the molds. The samples were then moist cured for seven days. After the seven-day moist curing process, the panels were kept in room conditions for three months before the measurements were collected. Followed steps during sample preparation are shown on Figure 3-11.

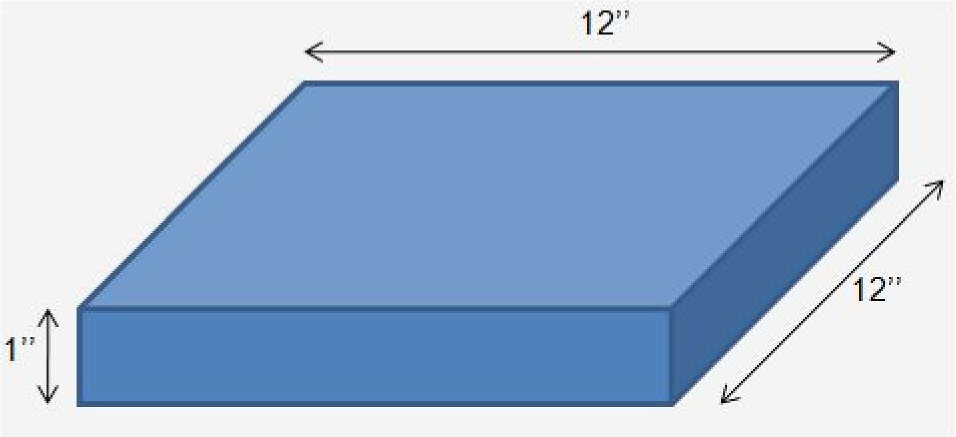


Figure 3-10: Sample Dimensions are 1 ft. by 1 ft. by 1 in.

Table 3.1: Cement Paste Sample Set

Cement Paste	
W/C	Sample
0.35	CP35
0.40	CP40
0.42	CP42
0.45	CP45
0.50	CP50
0.55	CP55

Table 3.2: Cement Mortar Sample Set

Cement Mortar		
S/C	W/C	Sample
2.53	0.35	CM35
	0.40	CM40
	0.42	CM42
	0.45	CM45
	0.50	CM50
	0.55	CM55
1.90	0.50	CM50S

3.3 Data Collection

Since the contact coaxial measurements were collected from single points of the samples, measurements need to be collected from different locations of a sample in order to obtain an average value representing the entire sample. When the heterogeneity of the cementitious materials is recognized, a reliable average value is necessary. The measurements were collected from six main regions of a panel with the shown locations in Figure 3-12. Ten measurements were collected randomly from each region, totalling at sixty measurements per sample.

Throughout this thesis, the average value is used when the complex permittivity of a

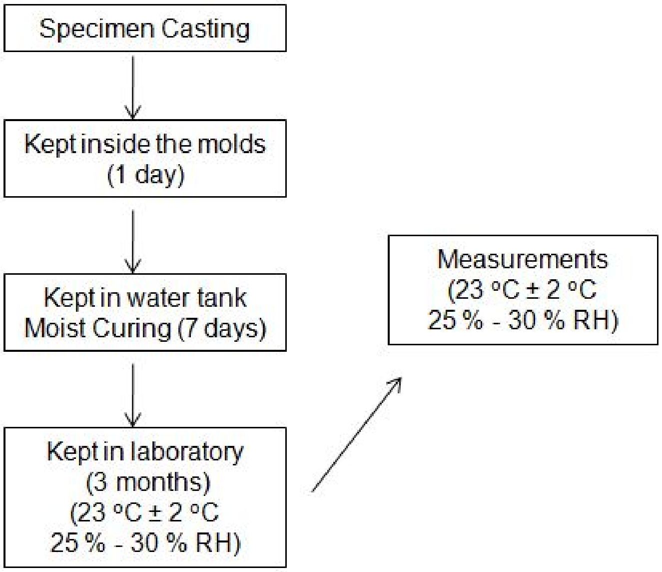


Figure 3-11: Followed steps for sample preparation

sample is indicated. Also, all the measurements have been collected in room temperature 23 ^{o}C ± 2 ^{o}C (73 ^{o}F ± 4 ^{o}F) with 25 % - 30 % relative humidity. When the samples are measured after the oven drying procedure, the samples were cooled down to room temperature and then measured. The oven drying procedure will be discussed in Chapter 4. Reliability and error estimation of the collected data will be further analyzed in Chapter 6.

3.4 Summary

In this section the equipment used for the measurements have been explained and the specifications were described. Also sample preparation method has been explained with the type of cement and sand used. The molds have been designed to give us required properties for the measurements. Sample dimensions are chosen to provide enough

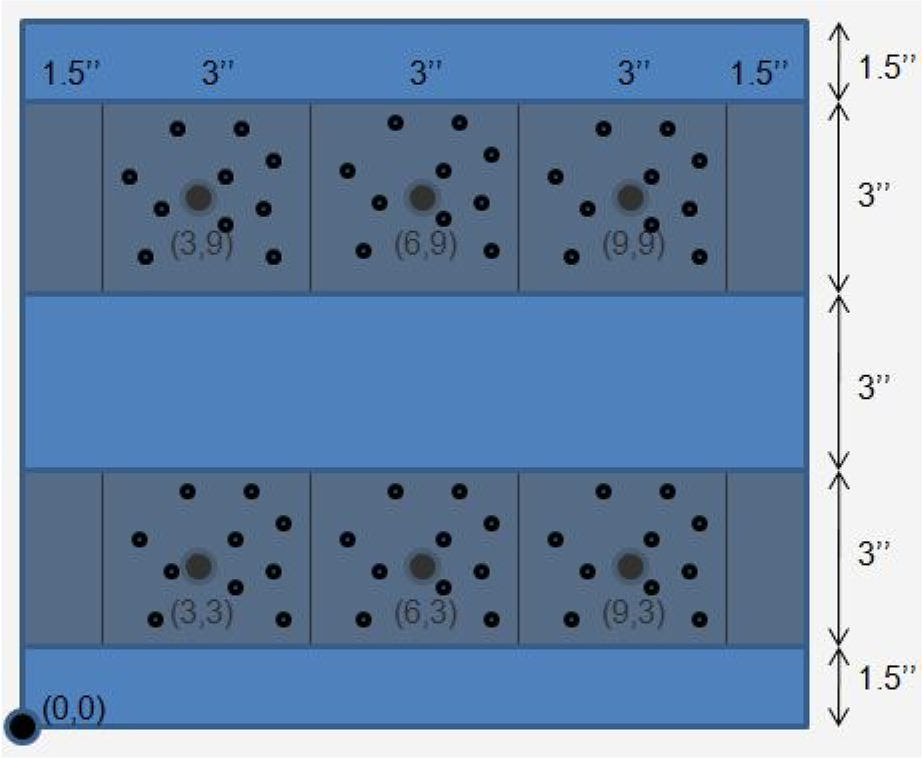


Figure 3-12: Data Collected From Different Regions

thickness and surface area for measurements.

Chapter 4

Experimentally Measured Dielectric Constant of Hydrated Cement Paste and Influence of Moisture

The dielectric constant of cement paste depends on the dielectric constants of three major contents in its structure; hydrated cement, air and water (Figure 4-1). Existence and volume of water and air are directly proportional to the porosity of cement paste, which is related to the water-to-cement (w/c) ratio. The influence of porosity on the dielectric constant is very important due to the water content. Dielectric constant of water at room temperature is shown in Figure 4-2. Also, the proportion of water to air within the pores is related to the ambient temperature and humidity at storage and during measurement. To create a controlled environment and to prevent the effect of these parameters we have stored the specimens in room temperature 23 ^{o}C ± 2 ^{o}C (73

^{o}F ± 4 ^{o}F) and 25 % - 30 % relative humidity. In our study we did not investigate the effect of temperature and humidity of the environment, these parameters were kept constant to control the variation of their influences.

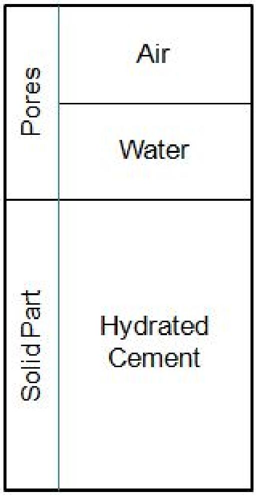


Figure 4-1: Contents of Cement Paste

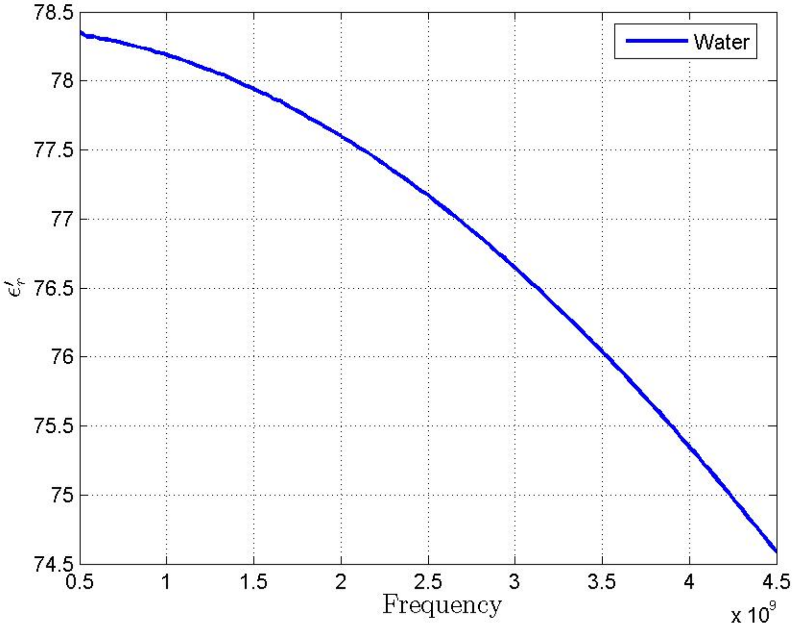


Figure 4-2: Dielectric constant of water at room temperature

4.1 Change in Dielectric Constant due to W/C Ratio of Cement Paste

The w/c ratio is a major parameter determining the void ratio of cementitious materials. When the w/c ratio increases the void ratio also increases. Our measurements showed that, as the void ratio of a cement paste increases, the dielectric constant decreases. This observation is true for samples kept and measured at 23 ^{o}C ± 2 ^{o}C (73 ^{o}F ± 4 ^{o}F) and 25 % - 30 % relative humidity. The results are shown in Figure 4-3.

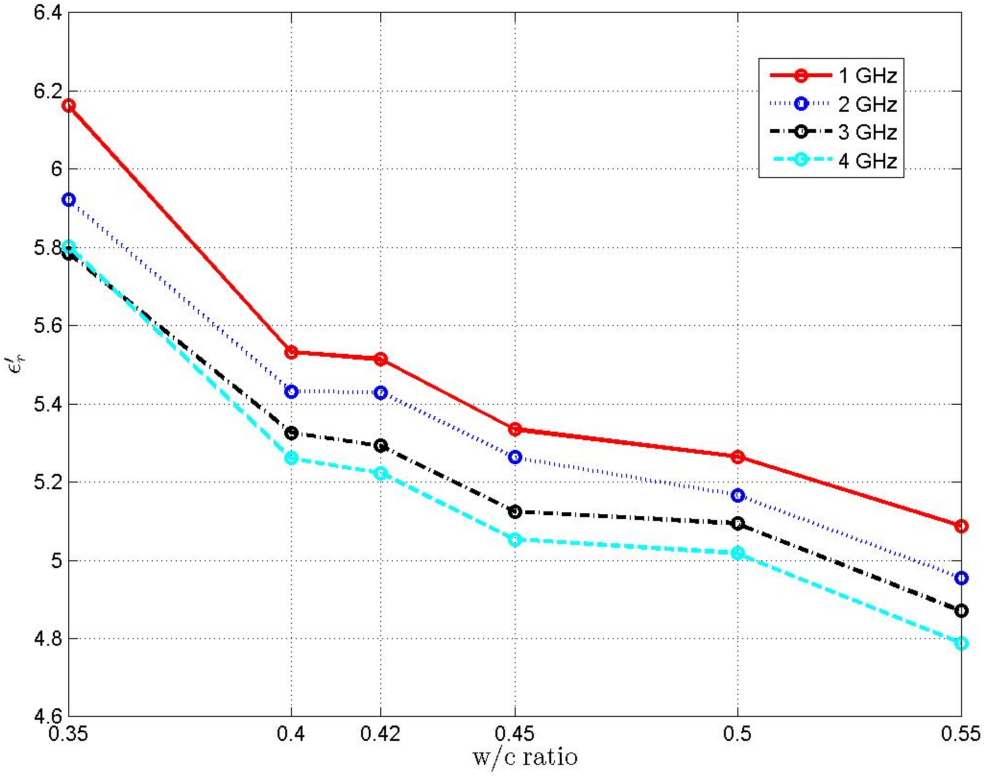


Figure 4-3: Dielectric constant of cement paste samples at room conditions

We see that as the w/c ratio increases the dielectric constant decreases. It means that the dielectric constant of voids is lower than the dielectric constant of hydrated cement paste. When the samples were kept at room conditions, the amount of water within the voids is low so the overall dielectric constant of the sample decreases with an increasing void ratio. Also, when we look at the dielectric constant at different measurement frequencies, we observed that the dielectric constant decreases as the measurement frequency increases.

4.2 Effect of Evaporable Water on the Dielectric Constant of Cement Paste

The evaporable water in cement paste has a very high influence on the dielectric constant of cement paste. To observe its effect we removed the water content within the voids with the oven drying procedure. Samples were kept in an oven twenty four hours prior to the measurements. The oven temperature was kept at 105 ^{o}C (221 ^{o}F) to achieve the boiling point of water. After the sample was taken out of the oven, it was covered with a layer of paper and a plastic stretch film to prevent the moisture in the sample from liberating. It was found that the temperature of the sample decreased to room temperature within an hour. Followed steps for oven drying procedure are shown in Figure 4-4.

When the evaporable water is removed from the voids, it will be replaced with air. Therefore, it is expected to observe a decreased dielectric constant value after the oven drying procedure. We expected to see a decrease in dielectric constant related to the

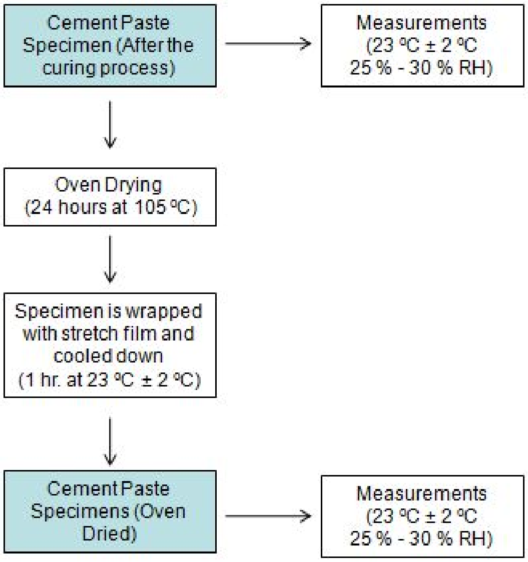


Figure 4-4: Steps followed for oven drying procedure

amount of water being removed from the sample. In Table 4.1 the weight of the samples before and after the oven drying procedure is shown. Also, the percentage of water loss (by weight) is shown in the same table.

Table 4.1: Weight of the Samples

Sample	Weight Before OD (lb)	Weight After OD (lb)	Water Loss by Weight (%)
CP35	10.035	9.650	3.84
CP40	9.225	8.870	3.85
CP42	9.160	8.775	4.21
CP45	8.910	8.520	4.38
CP50	8.790	8.385	4.61
CP55	8.100	7.725	4.63

As we can see from these values, the water loss is higher for samples with high w/c ratios. This means that as the w/c ratio increases, higher water loss will lead to a higher reduction of dielectric constant. However, when we look at the dielectric constant values after oven drying, we see that the dielectric constant increases as the w/c ratio increases. In Figure 4-5, the dielectric constant values of cement paste samples are shown with respect to their w/c ratios before and after oven drying. The reason for this kind of behavior is the formation of micro cracks on the surface of the samples due to the oven-drying procedure. Figure 4-6 to 4-11 show the cracks formed on the surface of each sample after the oven-drying procedure. Each photo shows an area of approximately 3 inches by 2.5 inches. The photos were taken using a macro lens camera (Canon® Rebel XS with Canon® Ultrasonic Macro Lens)) and processed using photo post-processing programmes to highlight the cracks. When oven-dried, the micro crack formation is higher for samples with lower w/c ratios as seen in these figures. Due to the formation of micro cracks, the void ratio of low w/c ratio samples was greater than high w/c ratio samples. This leads to a higher reduction of dielectric constant for low w/c ratio samples.

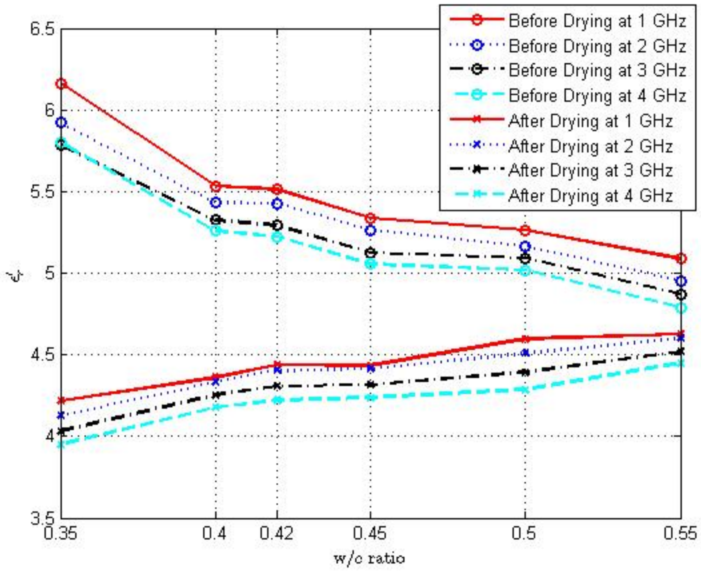


Figure 4-5: Dielectric constant of cement paste samples before and after oven drying

Yu (2009) [37] studied the dielectric properties of oven-dried cement paste by ob-

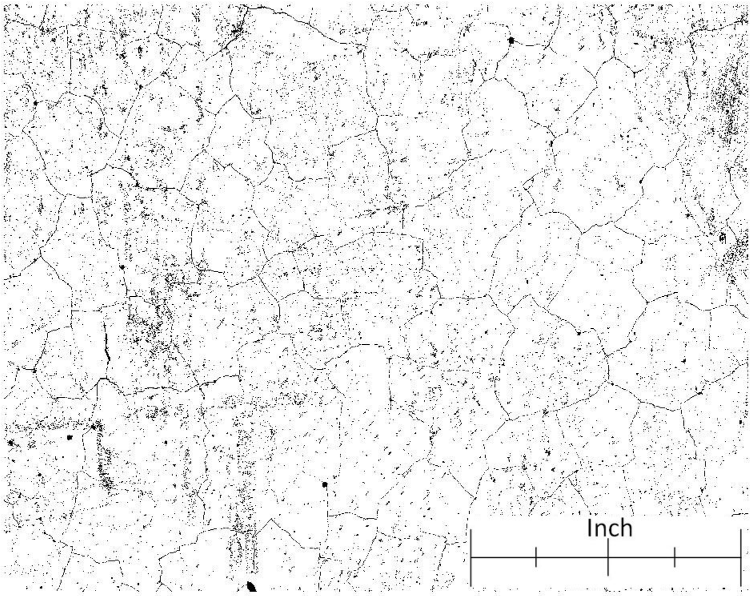


Figure 4-6: Micro cracks on the surface of CP35

serving the data reported by other groups. His conclusion is that the product of the dielectric constant and the w/c ratio is a constant, which is approximately 1.9 for the investigated data set. Yu mentioned that his proposed model is only valid in the frequency range of 3 GHz to 24 GHz and in the w/c ratio range of 0.25 to 0.4. To achieve this, we multiplied the dielectric constant of cement paste samples before oven drying with a reduction factor to obtain the same constant value 1.9 for the product of dielectric constant and the w/c ratio. Figure 4-12 shows that, after applying the reduction factors, the product of dielectric constant and the w/c ratio is approximately constant at 1.9. We used the dielectric constant values at 4 GHz frequency and used only the samples with w/c ratios of 0.35, 0.40 and 0.42 to stay in the valid range of Yu's model. We included the cement paste samples with w/c ratio of 0.42 to increase the data points even though

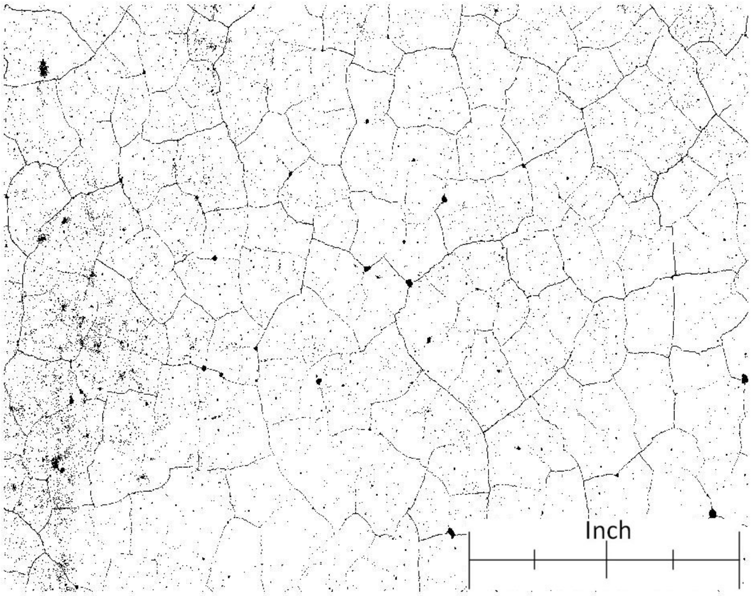


Figure 4-7: Micro cracks on the surface of CP40

it is not in the valid range. The reduction factors used for each sample are shown in Table 4.2. After obtaining the reduction factors for each cement paste sample, estimated dielectric constant of our cement paste samples are shown in Figures 4-13 to 4-15. It was found that the estimated dielectric constant values are lower than the measurements collected before oven drying but higher than the measurements after oven drying. This is because the surface cracks formed after oven drying were not considered.

The dielectric constant of cement paste samples measured after oven drying was lower than the estimated dielectric constant by Yu's model due to the formation of micro cracks on the surface of cement paste samples. When we applied Yu's model, the additional air caused by the cracks was not considered. In order to determine the

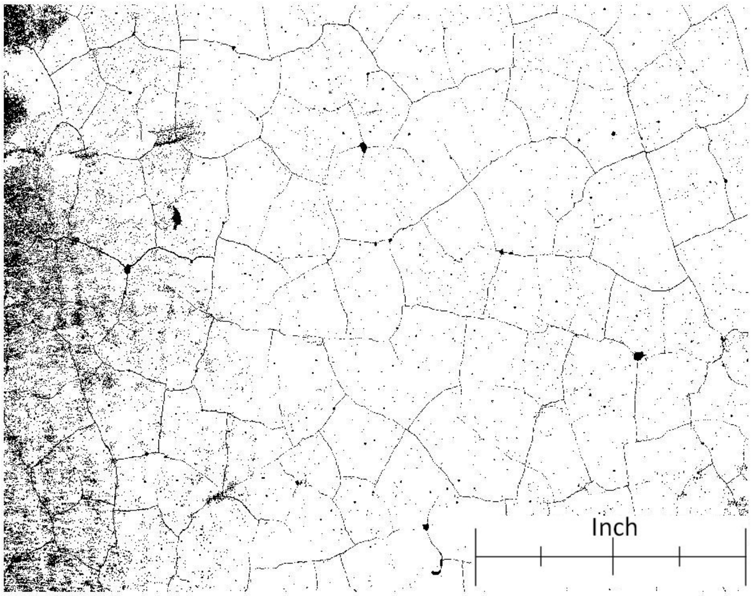


Figure 4-8: Micro cracks on the surface of CP42

Table 4.2: Reduction factors used to find estimated dielectric constant after the oven drying procedure using Yu's model

Cement Paste	
Sample	Reduction Factor
CP35	0.93
CP40	0.90
CP42	0.87

volume of surface cracks, we used Maxwell's dielectric mixture model (1891) [17].

$$\epsilon = \epsilon_h + \cfrac{3v_i\epsilon_h}{\cfrac{\epsilon_i + 2\epsilon_h}{\epsilon_i - \epsilon_h} - v_i} \tag{4.1}$$

where

ϵ = the effective complex permittivity of the mixture,

ϵ_h = the complex permittivity of the host dielectric,

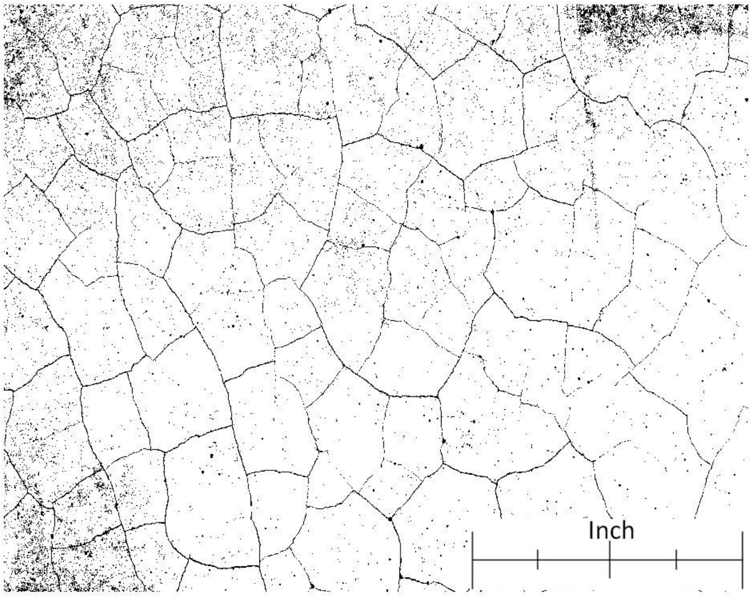


Figure 4-9: Micro cracks on the surface of CP45

ϵ_i = the complex permittivity of the inclusion dielectric, and

v_i = the volume fraction of the inclusion dielectric.

In our case the host dielectric was the estimated dielectric found by using Yu's model and the inclusion dielectric was the air since the cracks are filled with air. Using Maxwell's model we found the volume of air caused by the micro cracks for our cement paste samples. The estimated dielectric constants found by Maxwell's model was same as the dielectric constants found in our measurements after oven drying (Figures 4-16 to 4-18). The volume fractions of air introduced by the cracks are given in Table 4.3. The crack volume is higher for cement paste samples with a low w/c ratio. Steps followed for crack volume calculations are provided in Figure **??**.

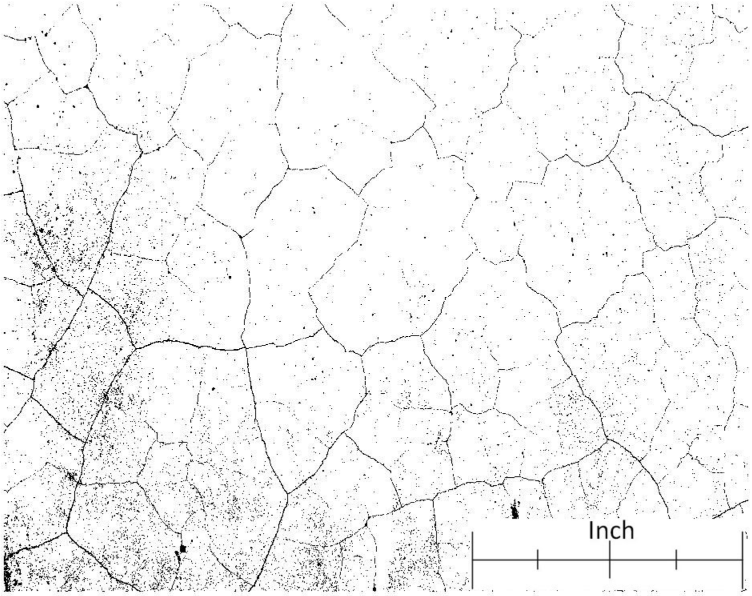


Figure 4-10: Micro cracks on the surface of CP50

Table 4.3: Volume fraction of the air caused by the micro cracks

Cement Paste	
Sample	Volume Fraction
CP35	0.14
CP40	0.07
CP42	0.04

4.3 Summary

In this chapter we reported the dielectric constant of cement paste samples before and after the oven drying procedure in the measurement frequency range of 0.5 GHz to 4.5 GHz. We observed that the dielectric constant of cement paste samples decreases as the measurement frequency and the w/c ratio both increase. After the oven drying procedure, the water within the voids were removed and the dielectric constant decreased.

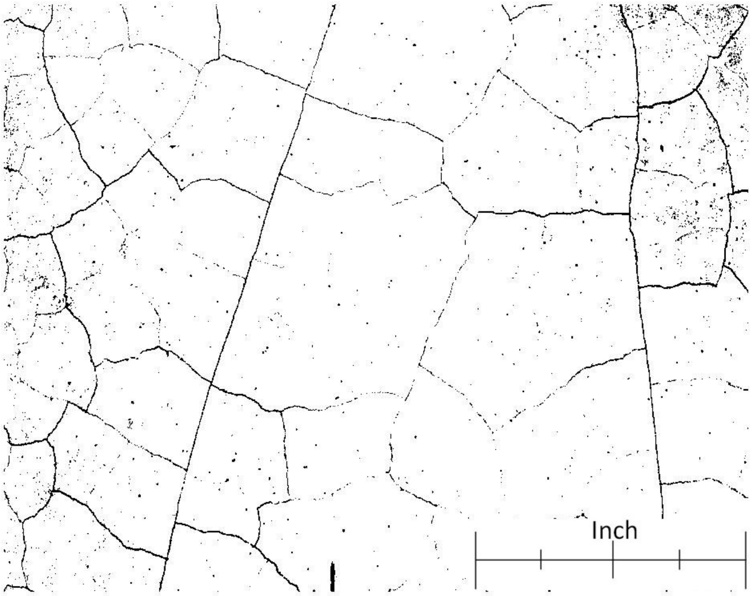


Figure 4-11: Micro cracks on the surface of CP55

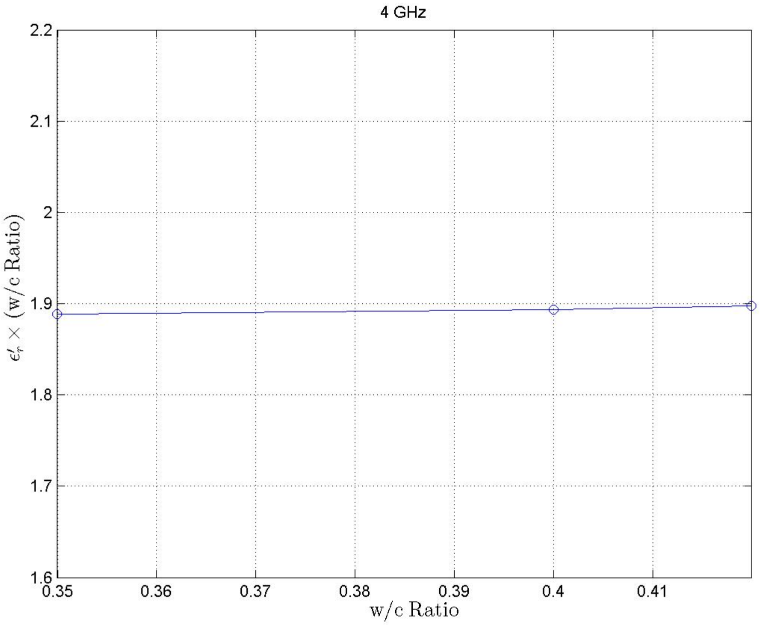


Figure 4-12: The product of the dielectric constant and the w/c ratio

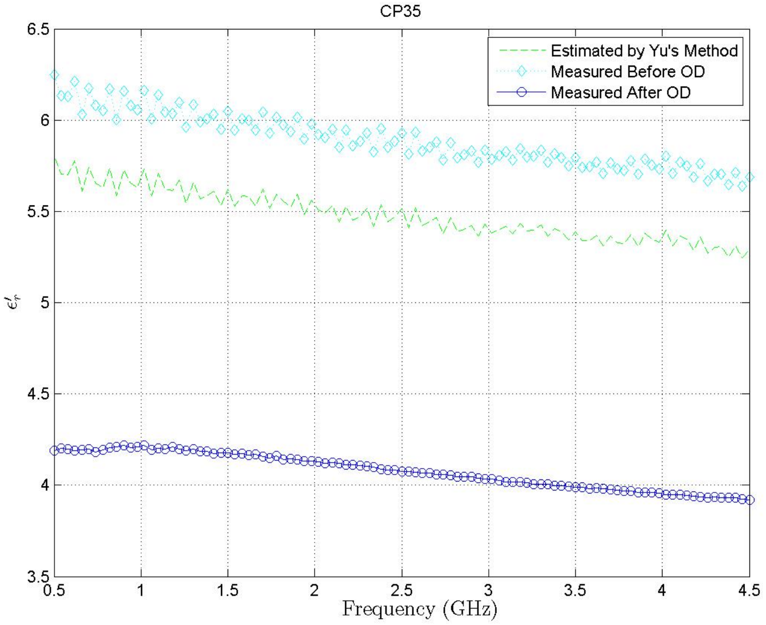


Figure 4-13: The estimated dielectric constant after oven drying procedure using Yu's model (w/c ratio = 0.35)

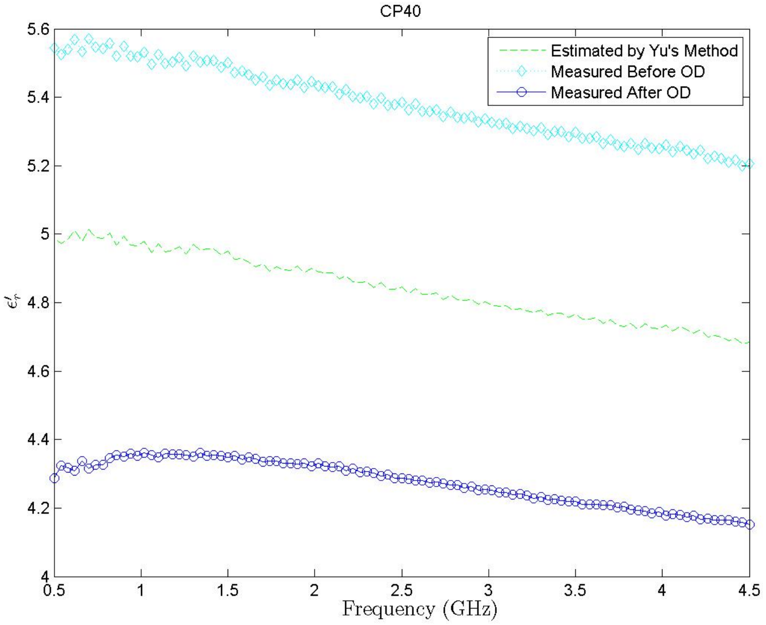


Figure 4-14: The estimated dielectric constant after oven drying procedure using Yu's model (w/c ratio = 0.40)

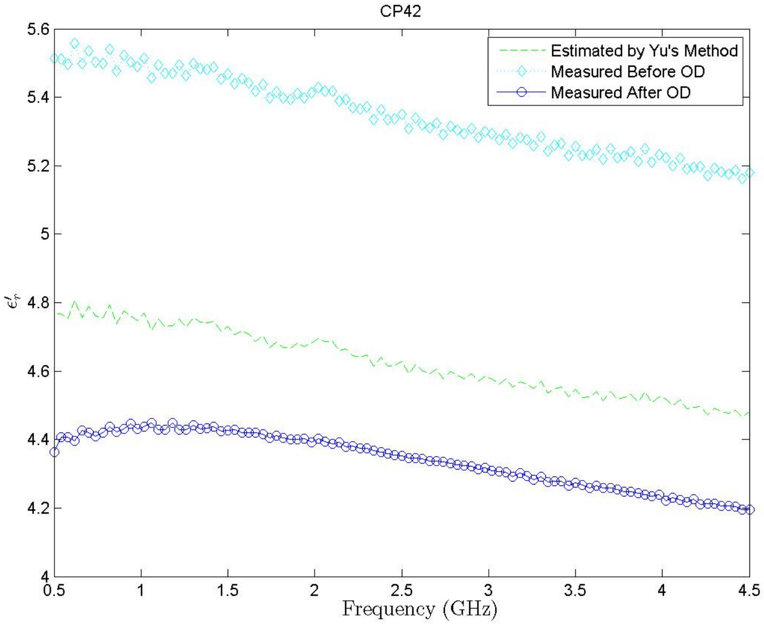


Figure 4-15: The estimated dielectric constant after oven drying procedure using Yu's model (w/c ratio = 0.42)

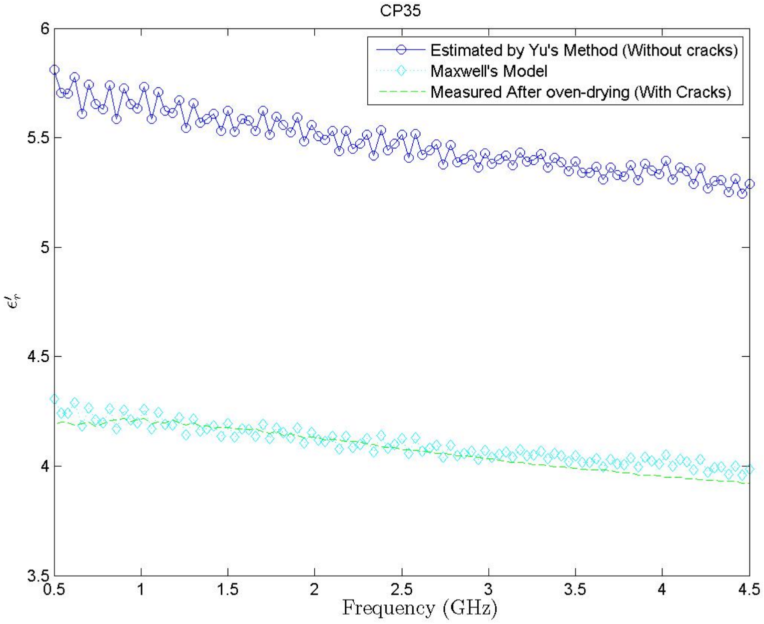


Figure 4-16: Dielectric constant using Maxwell's model with the calculated volume of cracks (w/c ratio = 0.35)

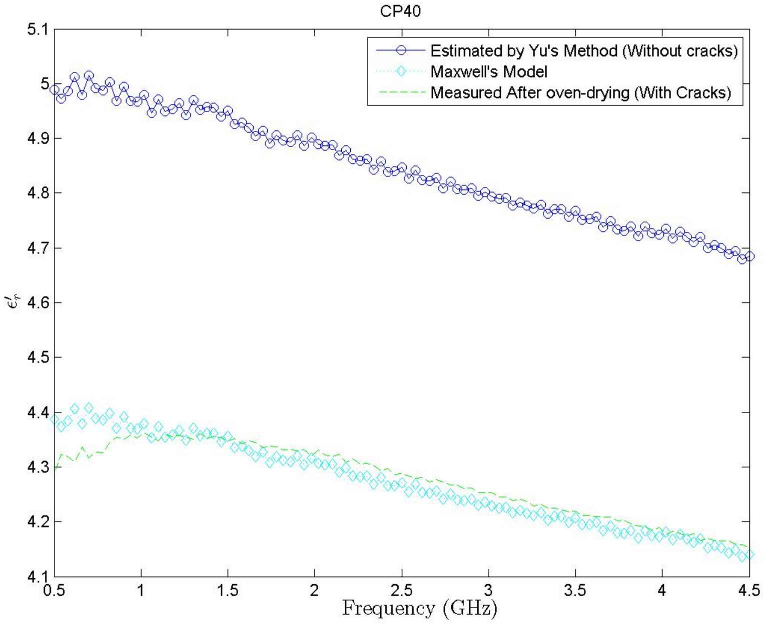


Figure 4-17: Dielectric constant using Maxwell's model with the calculated volume of cracks (w/c ratio = 0.40)

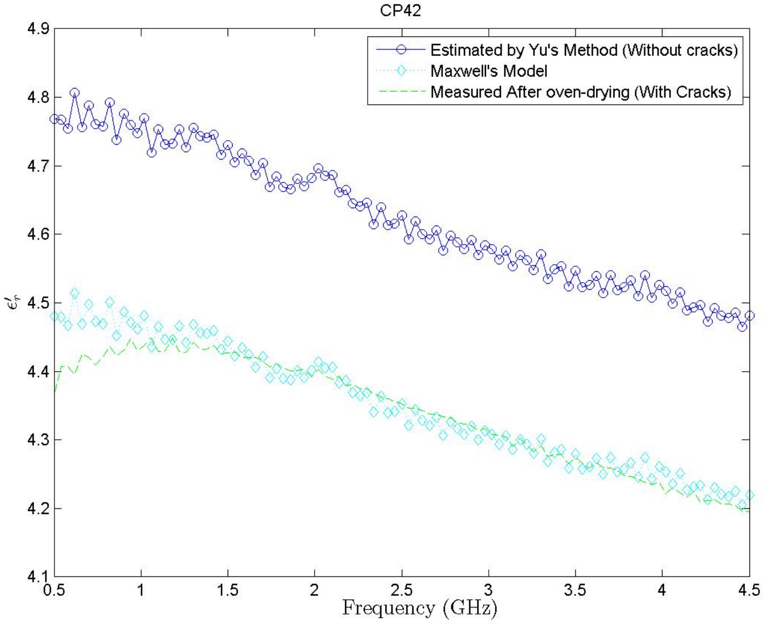


Figure 4-18: Dielectric constant using Maxwell's model with the calculated volume of cracks (w/c ratio = 0.42)

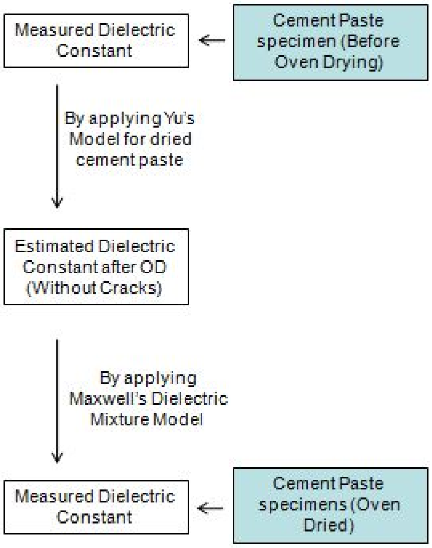


Figure 4-19: Steps followed for crack volume calculations

Due to the formation of micro cracks on the surface of samples, the change in dielectric constant varies for each sample with different w/c ratios. After applying Yu's model for oven dried cement paste samples and Maxwell's dielectric mixture model we found the volume of cracks formed on the surface of samples. We also found that the volume of micro cracks is larger for low w/c ratio samples.

Chapter 5

Experimentally Measured Dielectric Constant of Cement Mortar Panels and Effect of the S/C Ratio

We have showed our results about cement paste in the previous chapter. In this chapter we will present our findings on cement mortar. The cement mortar samples are made of hydrated cement and sand with voids partially filled with water and/or air (Figure 5-1). To better understand the effect of sand on the dielectric constant of cement mortar samples we also measured the dielectric constant of sand. The sand we have used for our samples was All-Purpose Sand from Quikrete®. The dielectric constant of the sand is shown on Figure 5-2.

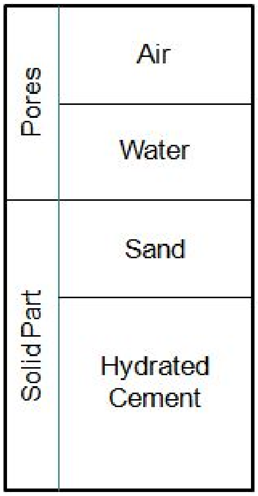


Figure 5-1: Contents of Cement Mortar

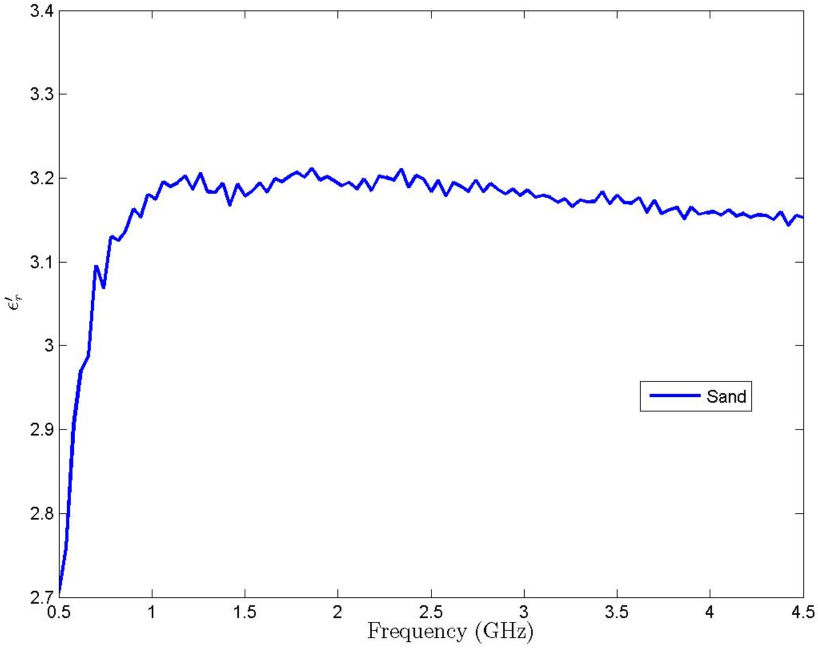


Figure 5-2: Dielectric constant of Quikrete® All-Purpose Sand

5.1 Change in Dielectric Constant due to W/C Ratio of the Cement Mortar

Dielectric constant of six cement mortar samples with w/c ratios ranging from 0.35 to 0.50 has been shown on Figure 5-3. As seen on Figure 5-3 the dielectric constant of cement mortar samples decreases as the w/c ratio increases and the measurement frequency increases. This supports our results for cement paste samples which have been discussed in the previous chapter. An increase in w/c ratio increases the void content of the cement mortar samples. A cement mortar sample with a higher void content has a lower dielectric constant. This is due to the contribution of low dielectric constant value of air voids.

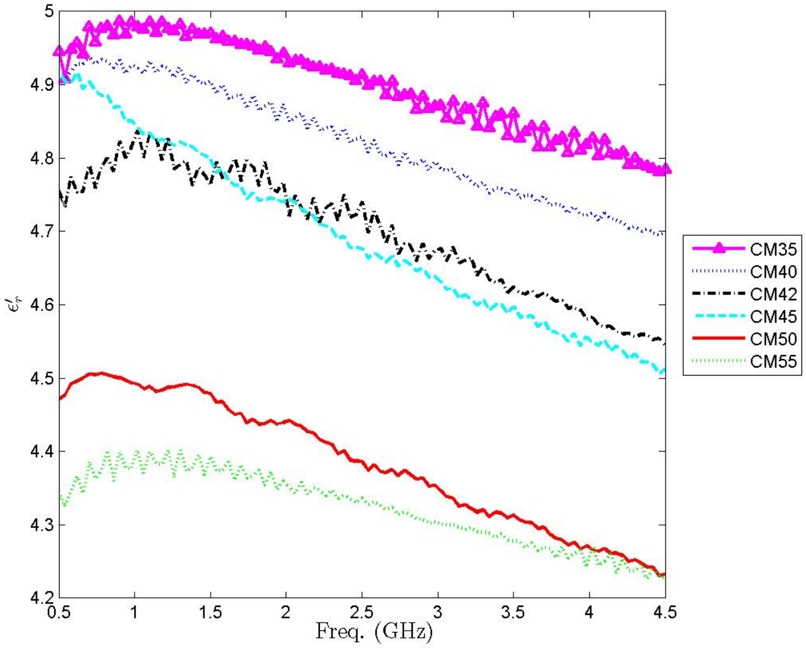


Figure 5-3: Dielectric constant of cement mortar samples with various w/c ratios

5.2 Effect of the S/C Ratio on the Dielectric Constant of Cement Mortar

The relatively low dielectric constant of sand, as compared to cement paste, is useful in terms of determination of the effect of sand on dielectric constant. Cement mortar is expected to have a lower dielectric constant when compared to cement paste. The dielectric constant values of cement paste and cement mortar with corresponding w/c ratios are compared and shown on from Figures 5-4 to 5-9. As seen on these figures, when the w/c ratio is kept constant, the addition of sand decreases the dielectric constant of a cement mortar sample.

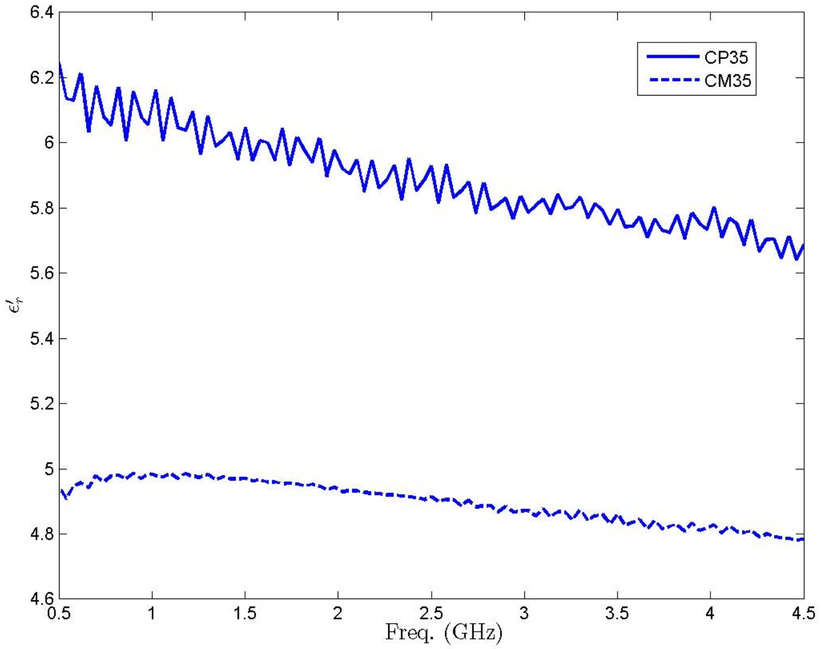


Figure 5-4: Dielectric constant of 0.35 w/c ratio cement paste and cement mortar panel specimens

The decrease in dielectric constant is better shown on Figure 5-10 at 2 GHz fre-

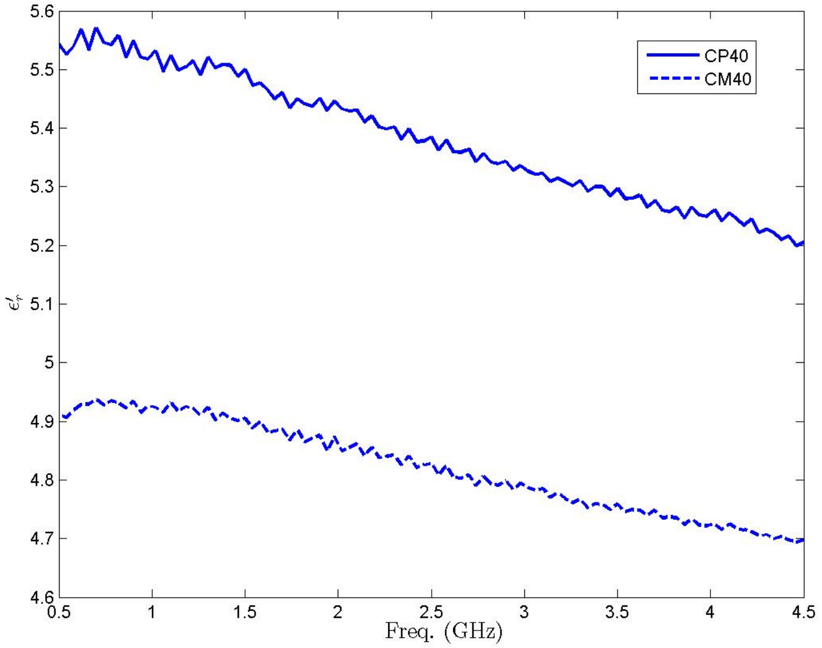


Figure 5-5: Dielectric constant of 0.40 w/c ratio cement paste and cement mortar panel specimens

quency. With the obtained experimental result, it is believed that there should be a relation between the amount of sand and dielectric constant of cement mortar. To investigate such relation we have cast a cement mortar sample with a w/c ratio of 0.50 and a s/c ratio of 1.9 (CM50s) which has a different s/c ratio used for the other cement mortar samples (s/c = 2.53). On Figure 5-11 the x-axis shows the weight (mass) proportion of sand to all solid ingredients used in cement paste including cement and sand. Zero for the x-axis means no sand is used which corresponds to a cement paste (CP50) specimen and unity for the x-axis represents a sand specimen. We used the s / (s+c) ratio instead of the s/c ratio in order to scale the ratio from zero to unity; zero for cement paste and unity for sand. Figure 5-12 shows the values of dielectric constant in terms of percentage, where the dielectric constant of cement paste is accepted as 100 %. The

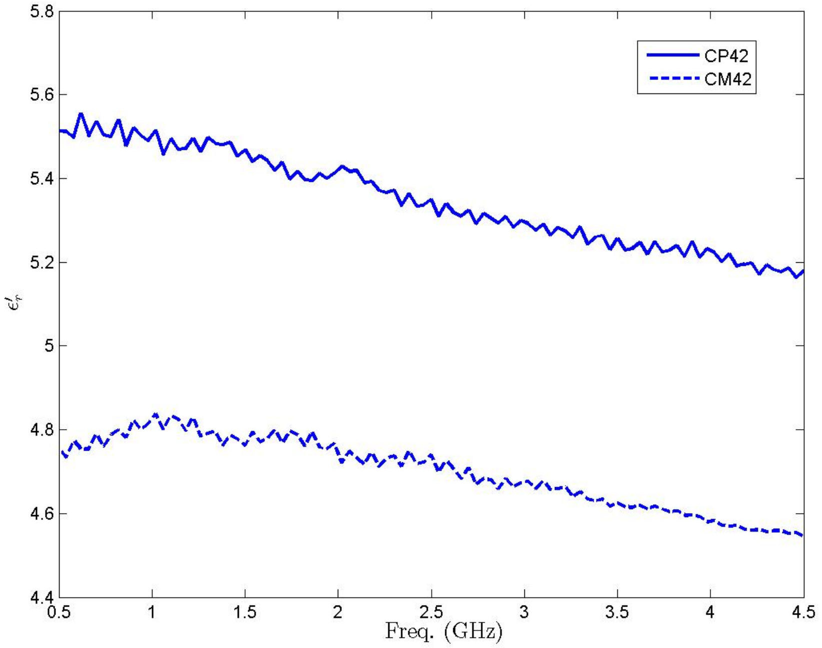


Figure 5-6: Dielectric constant of 0.42 w/c ratio cement paste and cement mortar panel specimens

dielectric constant of sand is equal to about 60 % of the dielectric constant of cement paste which can be obtained from Figure 5-12 when the s / (s+c) ratio is equal to unity. The amount of decrease is modeled using the amount of sand used in cement mortar. A cubic equation model is proposed in the following, where y is the calculated reduction factor to obtain the dielectric constant of cement mortar with a known s/c ratio by using the dielectric constant of cement paste with the same w/c ratio, and x is the s / (s+c) ratio (by weight) as shown in Figure 5-13.

$$y(x) = ax^3 + bx^2 + cx + d \tag{5.1}$$

a = -27.89, b = - 19.12, c = 8.76, d = 100

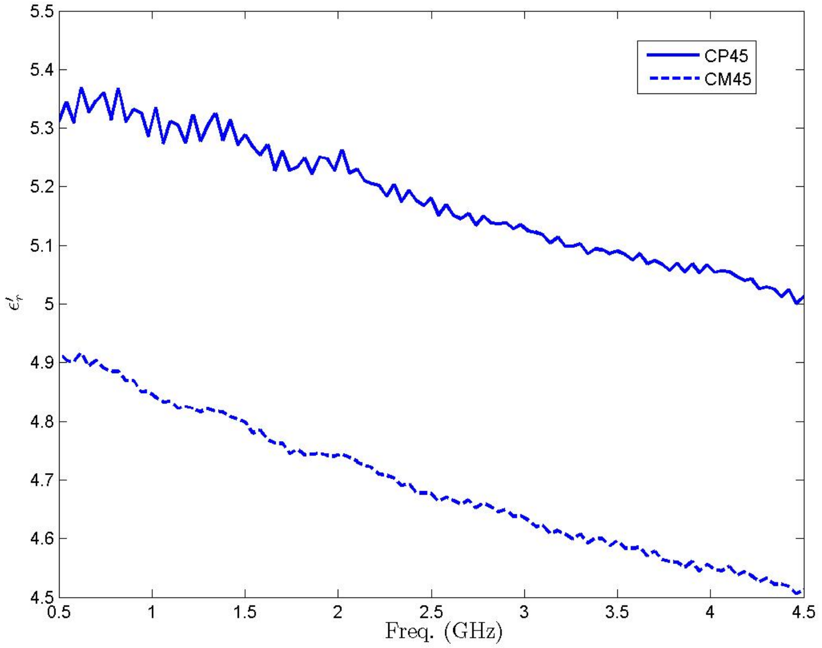


Figure 5-7: Dielectric constant of 0.45 w/c ratio cement paste and cement mortar panel specimens

Note that this approach which is illustrated on Fig. 5-14 is only applicable for measurements conducted at room temperature 23 $^oC \pm 2$ ^oC (73 $^oF \pm 4$ ^oF) and 25 % - 30 % relative humidity. The use of the reduction factor determined by Eq. 5-1 allows us to estimate the dielectric constant of cement mortar (with a known s/(s+c) ratio) which is a four-phase composite (hydrated cement, water, air, sand), using the dielectric constant of cement paste (with the same w/c ratio) which is a three-phase composite (hydrated cement, water, air) simply by multiplying the dielectric constant of cement paste with the reduction factor found with Eq. 5-1. The reduction factor may change at different relative humidities since the ratio of water to air in the voids changes. Using Eq. 5-1 the reduction factor for s/c ratio of 2.53 is calculated to be 0.86, in which 0.717 is used for the x value. Using this reduction factor we have can

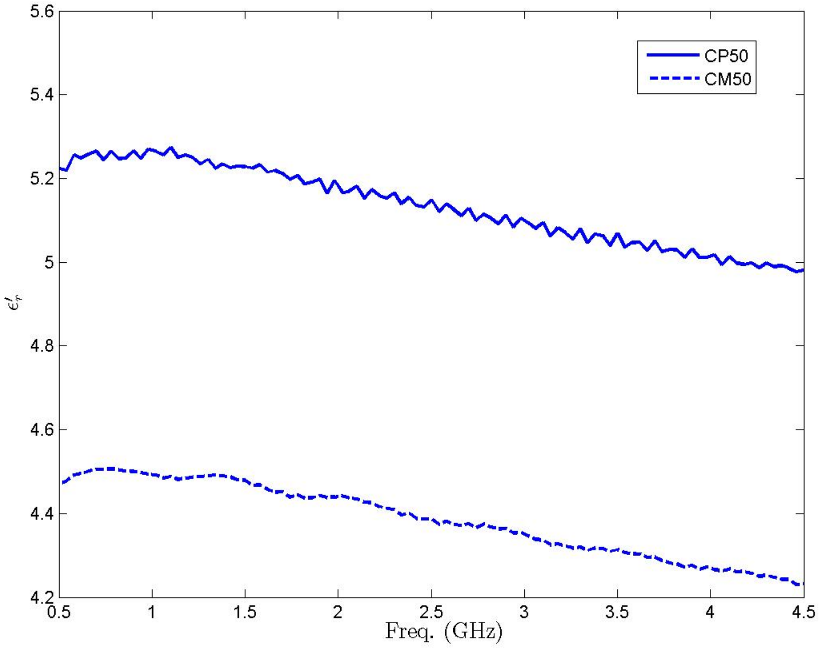


Figure 5-8: Dielectric constant of 0.50 w/c ratio cement paste and cement mortar panel specimens

estimate the values of dielectric constant of cement mortar samples with various w/c ratios. As seen in Figure 5-15 the calculated values are very close to the measured values of dielectric constant of cement mortar at 2 GHz. It is also believed that the reduction factor is applicable to cement mortar samples with different w/c ratios. It indicates that the effect of sand on dielectric constant is independent of the w/c ratio. Calculated reduction factor for 2 GHz is also applicable for 4 GHz as seen in Figure 5-16, as well as for all frequencies in the range of 0.5 GHz to 4.5 GHz. In Fig. 5-17, the estimation error is a function of frequency and the w/c ratio as calculated. The highest error is approximately 8 %, and the average error is approximately 4 %.

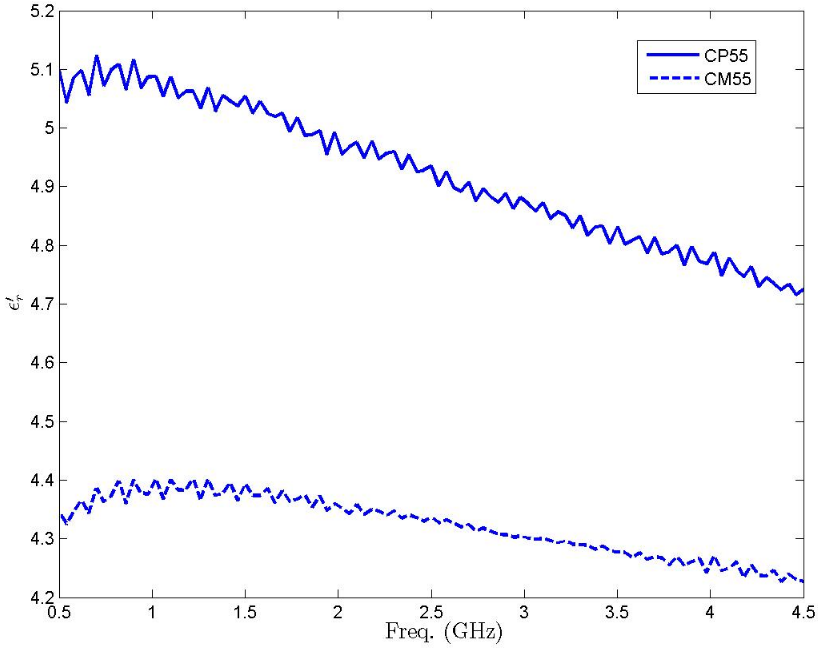


Figure 5-9: Dielectric constant of 0.55 w/c ratio cement paste and cement mortar panel specimens

5.3 Summary

In this chapter we reported the dielectric constant of cement mortar samples in the frequency range of 0.5 GHz to 4.5 GHz. We also studied the effect of sand on the dielectric constant of cement mortar. Due to the relatively low dielectric constant of sand, the dielectric constant of cement mortar (cement paste plus sand) is reduced as opposed to the one of cement paste. Also, as the amount of sand (s/c) increases the dielectric constant decreases. The expected decrease has been modeled by a cubic equation. The effect of sand on the dielectric constant of cement mortar is not related to the measurement frequency or the w/c ratio of cement mortar. Therefore, the proposed cubic model is applicable for cement mortar with w/c ratio in the range of 0.35 to 0.55

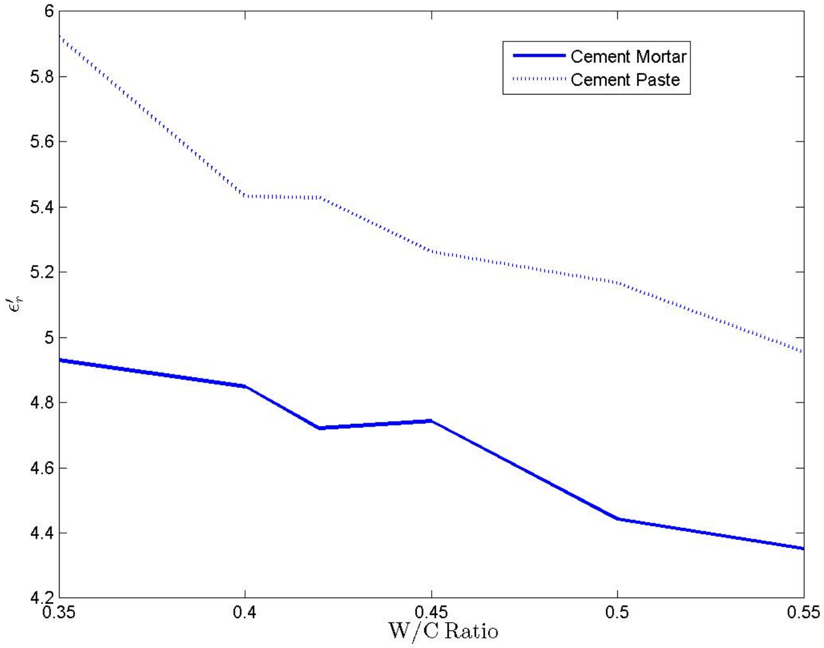


Figure 5-10: Dielectric constant of cement mortar with various w/c ratios

and in the measurement frequency range of 0.5 GHz to 4.5 GHz.

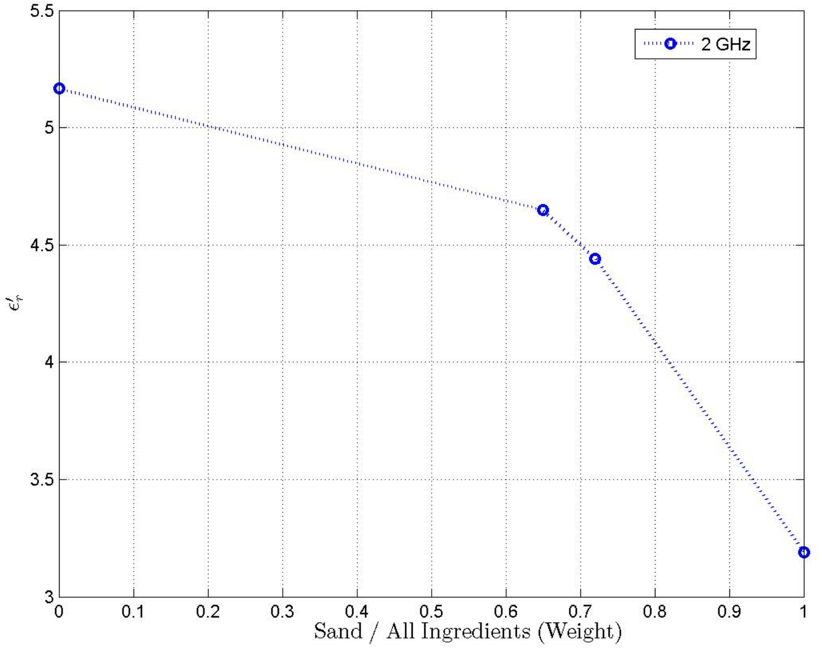


Figure 5-11: Dielectric constant versus the sand content in cement mortar (w/c ratio = 0.50)

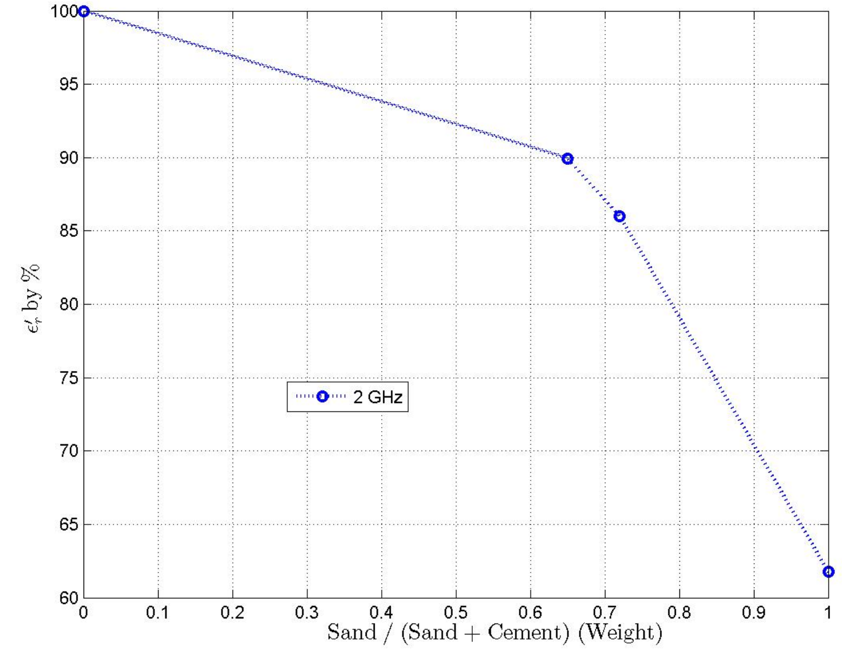


Figure 5-12: Dielectric constant of cement mortar versus the sand content in percentage (w/c ratio = 0.50)

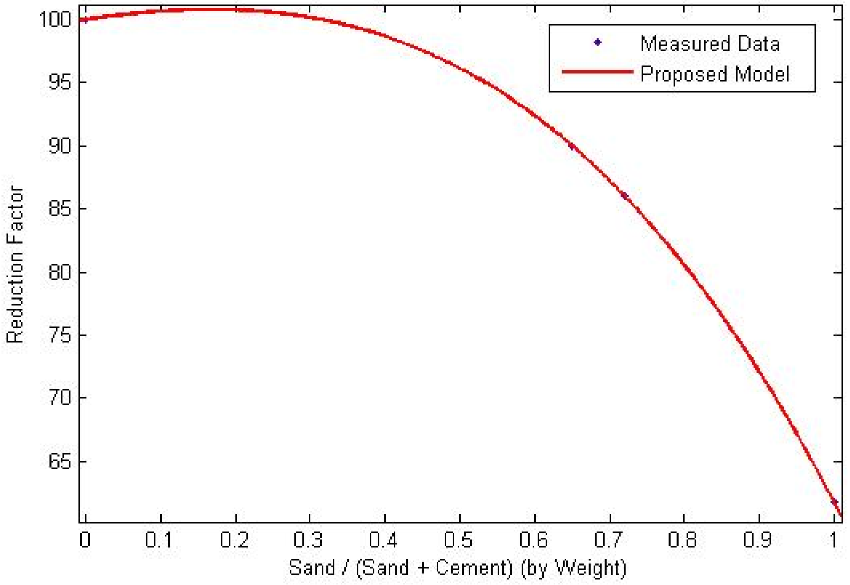
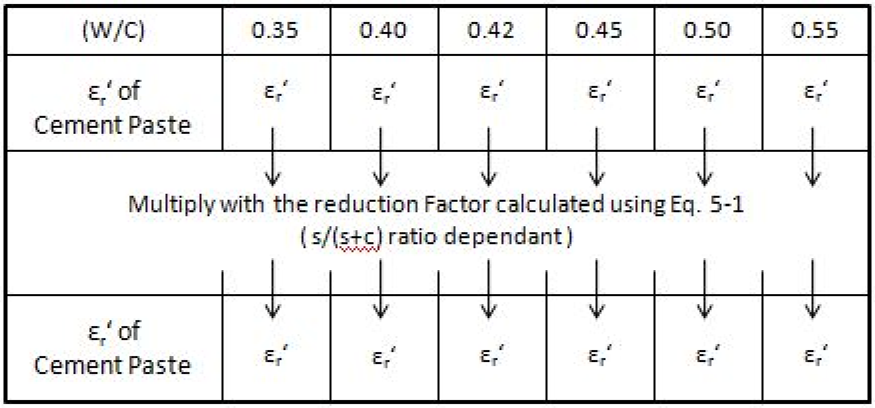


Figure 5-13: A cubic model for estimating the reduction factor with a given s/c ratio



(W/C)	0.35	0.40	0.42	0.45	0.50	0.55
ε_r' of Cement Paste	ε_r'	ε_r'	ε_r'	ε_r'	ε_r'	ε_r'
Multiply with the reduction Factor calculated using Eq. 5-1 (s/(s+c) ratio dependant)	↓	↓	↓	↓	↓	↓
ε_r' of Cement Paste	ε_r'	ε_r'	ε_r'	ε_r'	ε_r'	ε_r'

Figure 5-14: By using the reduction factor, dielectric constant of cement mortar can be calculated if the dielectric constant of cement paste with the same w/c ratio is known

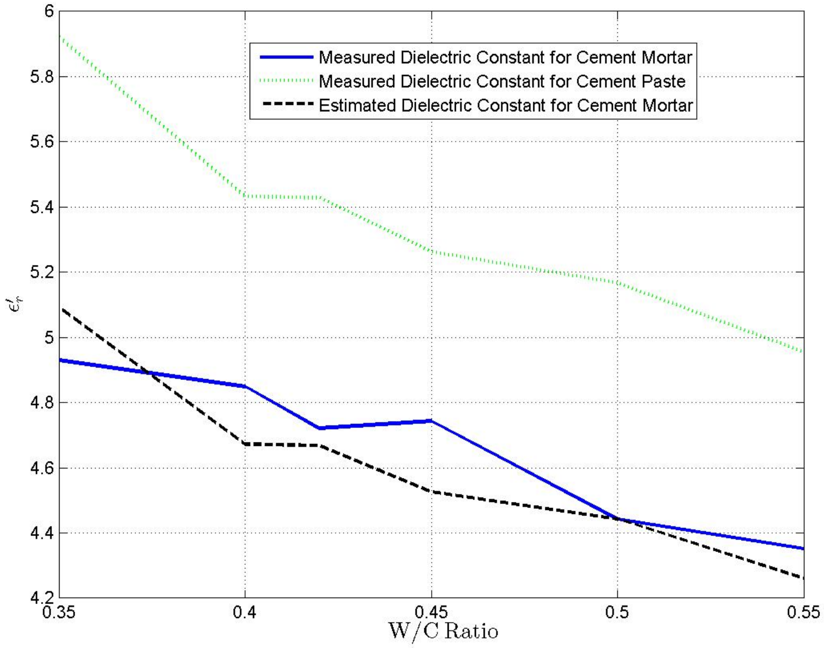


Figure 5-15: Measured and estimated dielectric constant values of cement mortar at 2 GHz

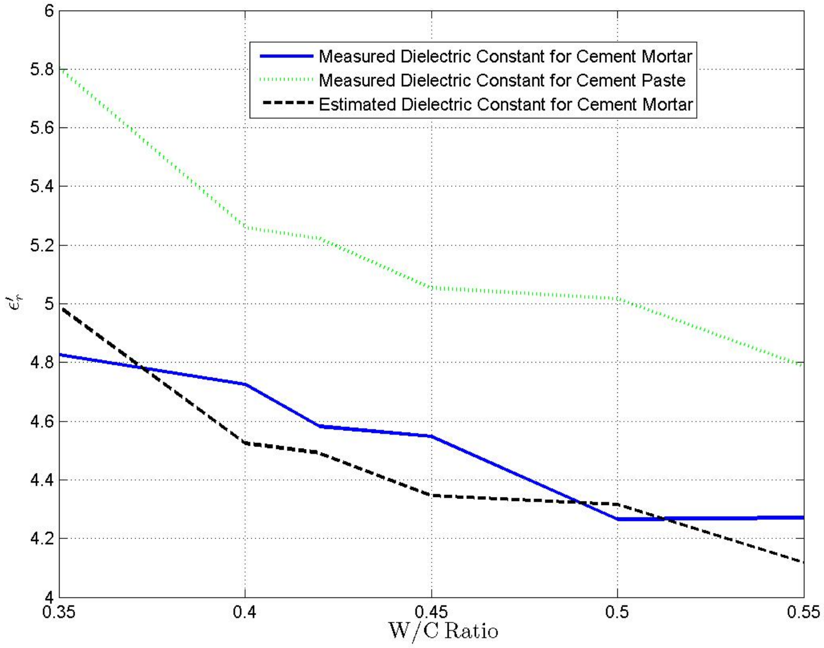


Figure 5-16: Measured and estimated dielectric constant values of cement mortar at 4 GHz

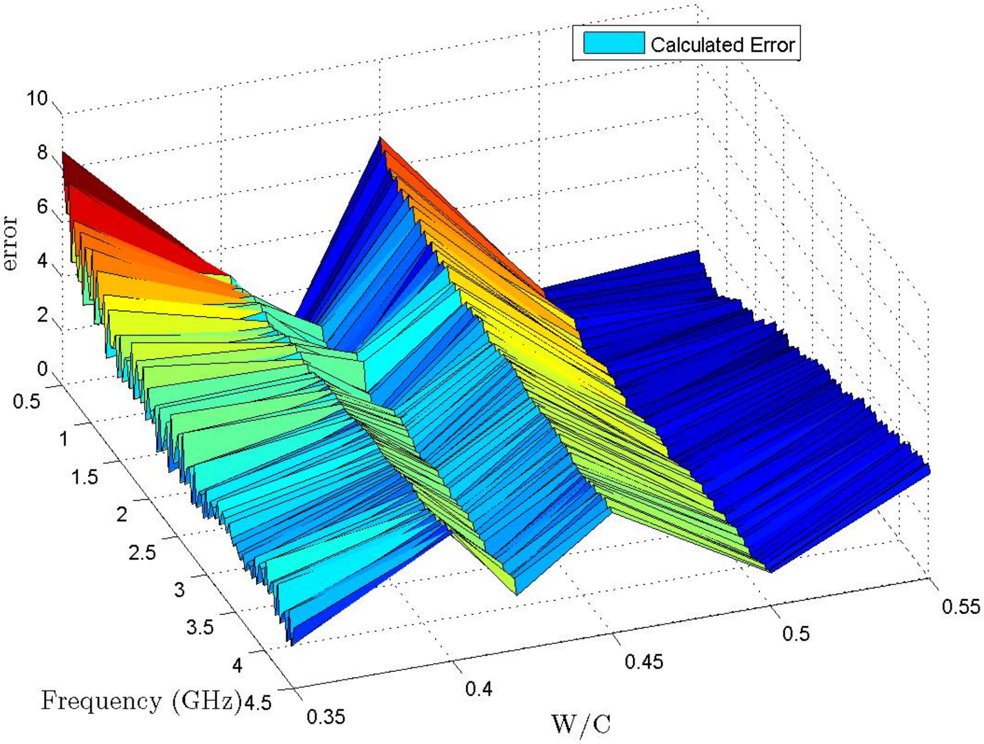


Figure 5-17: Calculated errors for the proposed model described by Eq. 5-1

Chapter 6

Reliability of Dielectric Measurements

The complex permittivity of cement paste depends on many factors such as curing time, w/c ratio, relative humidity. Cement paste has a heterogeneous structure that consists of hydrated cement, and voids which are partially filled with water and air. The heterogeneity of cement paste makes it very challenging to measure the dielectric properties especially when contact measurement methods are used. Due to heterogeneity of cement paste, a point on the surface of a sample may have different properties compared to overall properties of that sample such as w/c ratio and water fraction inside the pores. Point readings on the surface of the cement paste samples do not necessarily represent the whole sample, therefore multiple measurements are required. By increasing the number of point readings on a cement paste sample, statistically a more reliable average value can be obtained to represent the overall property of the cement paste sample.

In this chapter, we discuss the reliability of measured complex permittivity of cement paste panels using contact measurement, as well as the effect of choosing different frequencies and w/c ratios in the reliability of the measurements. We also used Monte

Carlo Simulations to determine the expected error for a given number of point measurements. The expected error for dielectric measurements of cement paste samples before and after oven drying, has been related to heterogeneity of cement paste samples.

6.1 Monte Carlo Simulations

In statistical interference, there are certain parameters needed [5]. If someone needs to test the mean of a distribution, type of the distribution is needed. For example if the distribution of the sample set is normal, T distribution can be used for that set. In cases where the properties of the distribution are very difficult to determine analytically or not known Monte Carlo methods may be used. The Monte Carlo Method was first coined by John von Neumann and Stanislaw Ulam in 1940s. Monte Carlo methods generate sample sets by using repeated random sampling. Monte Carlo methods vary in their application but the main idea is usually similar, which in our case is;

- **Definition of Inputs** - Sixty measured points are the possible inputs.

$$y = f(x_1, x_2, ..., x_{60}) \tag{6.1}$$

- **Generation of Inputs Randomly** - A set is created by randomly selecting the possible inputs.

$$y_i = (x_{i1}, x_{i2}, ..., x_{i60}) \tag{6.2}$$

- **Generation of Random Sets** - One thousand random sets are generated from the sixty measured points (possible inputs).

$$i = 1 \ to \ 1000 \tag{6.3}$$

- **Generation of a Single Set** - The average of all random sets have been calculated.

$$x_1 = \frac{\sum x_{i1}}{1000}, \ i = 1 \ to \ 1000 \tag{6.4}$$

- **Analysis of the Results**

Since the dielectric constant of cement paste is affected by the air and water filling the voids, void ratio is important for determination of dielectric constant. But the void ratio is not the same for every section in a single cement paste sample due to the heterogeneity, so it is expected that different compositions of air, water and hydrated cement result in varying dielectric properties. This heterogeneity of cement paste results in different dielectric constant values at different points of the same sample. All the dielectric constant values used were average values of sixty measurements for each sample. Statistically, we calculated that the average value obtained by using sixty measurements contains an error less than 3 % with 95 % confidence level.

With more measurements collected from a sample a more representative dielectric constant value can be obtained. In Figure 6-1 a Monte Carlo simulation was ran which randomly selects the measurements from sixty different points at 1 GHz frequency. As the number of data points increases the average value of the selected data converges to the average value of sixty measurements as seen in Figure 6-1. When the same simulation was used at different frequencies and w/c ratios similar trends were observed. To be able to observe the relation between the error and the number of measurements more clearly, Monte Carlo simulations have been used. A Monte Carlo simulation such as the one used in Figure 6-1 was ran one thousand times and their average was calculated. The results are shown in Figure 6-2. This way a smoother curve was obtained.

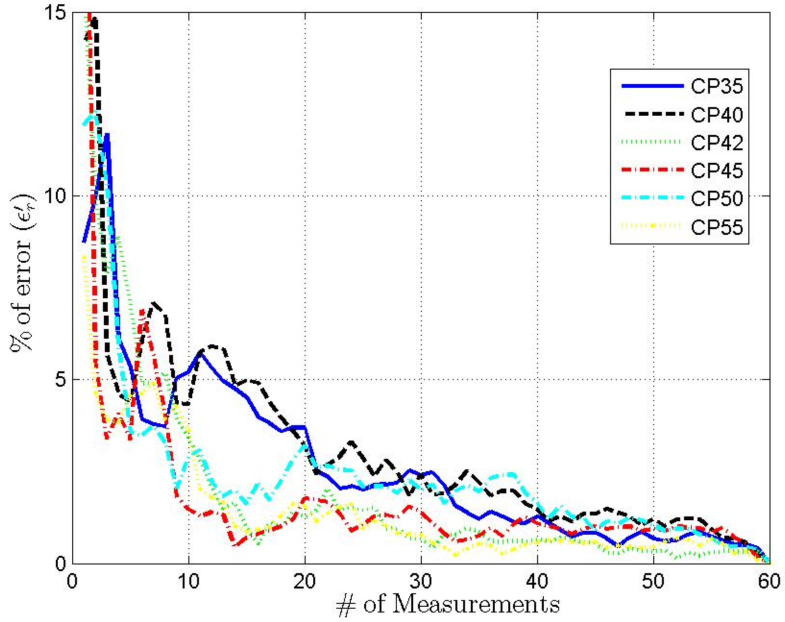


Figure 6-1: Percentage of error vs. the number of measurements

6.2 Error Estimation

The obtained data from a sample does not give us the true average value of the dielectric constant of that sample since we cannot collect measurements from infinite number of points from the sample surface. But with increasing number of measurements we can find an average value that is closer to the true value. Statistically, using the collected

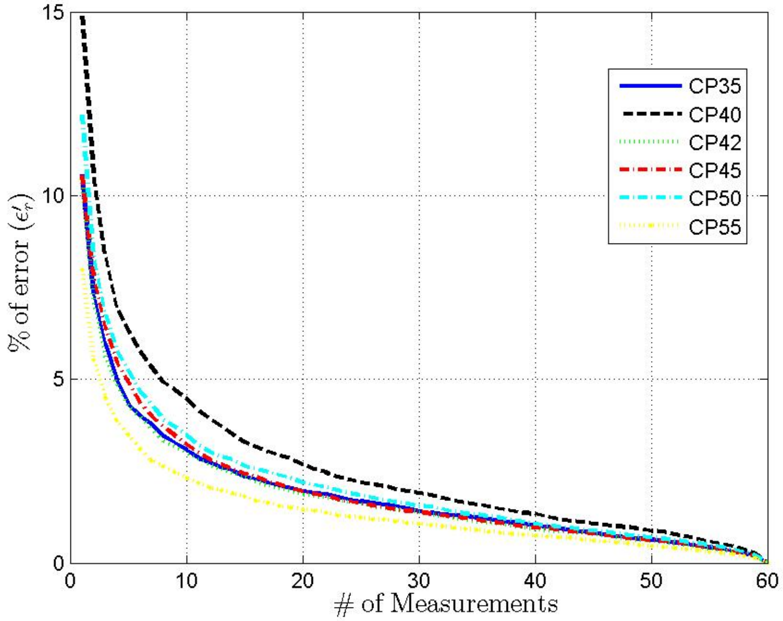


Figure 6-2: Percentage of error vs. the number of measurements obtained by using Monte Carlo Simulations

data, we can calculate the possible range of the true value or the percentage of error in the calculated mean value that represents the whole sample. Also, other than the number of measurements conducted, there may be other parameters that may have an influence on the percentage of error. Possible parameters considered in this thesis are the frequency of the measurements, w/c ratio and the presence of evaporable water within a cement paste sample.

$$Percentage\ of\ Error = \frac{\frac{\sum x_i^*}{i} - \sigma}{\sigma} \times 100 \tag{6.5}$$

$$y^* = (x_1^*, x_2^*, ..., x_{60}^*) \tag{6.6}$$

6.2.1 Effect of Frequency on Error

As we discussed before the dielectric properties of cement paste is a composition of the dielectric properties of hydrated cement, air and water. The dielectric constant of hydrated cement is around 4 to 5 within the frequency range we are working and it is 1 for air. But the dielectric constant of water is much more higher compared to other ingredients which is shown on Figure 4-2 and it ranges from 78.5 at 4.5 GHz to 74.5 at 4.5 GHz. So it is expected to observe the influence of water to decrease when the measurements are collected at higher frequencies, since water has a dielectric constant closer to dielectric constant of hydrated cement at higher frequencies within the range we are working with. The percentage of error for all six cement paste samples with different w/c ratios are shown for frequencies from 1 GHz to 4 GHz from Figure 6-3 to Figure 6-8. The influence of frequency on the percentage of error could not be detected as seen on the figures. The calculated percentage of error is very close to each other at different frequencies within our frequency range. The difference in dielectric constant of water within the frequency range of 0.5 GHz to 4.5 GHz is not very high so we were unable to observe an effect due to this change in dielectric constant. But the difference in the amount of evaporable water is expected to have a large effect on the error since water is the most important component due to its high dielectric constant and possible uneven distribution which is also analyzed further.

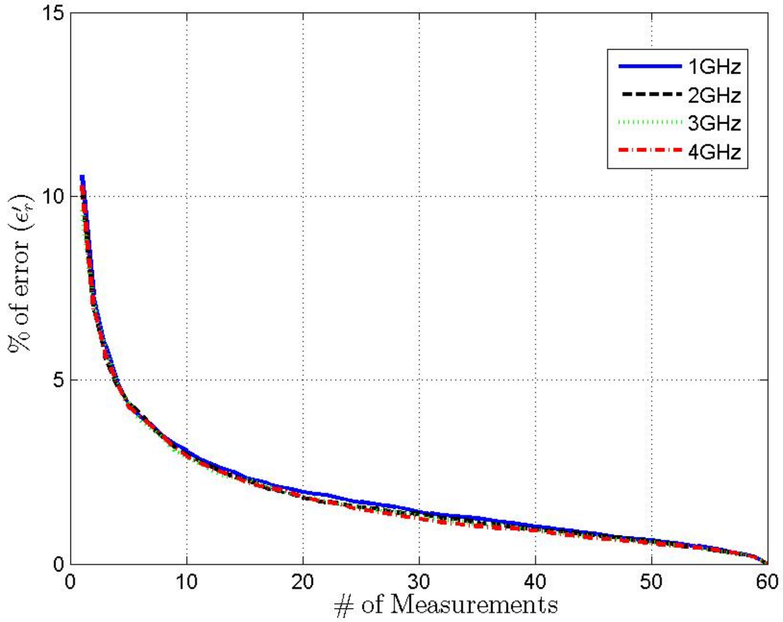


Figure 6-3: Frequency dependency of the percentage of error for sample CP35

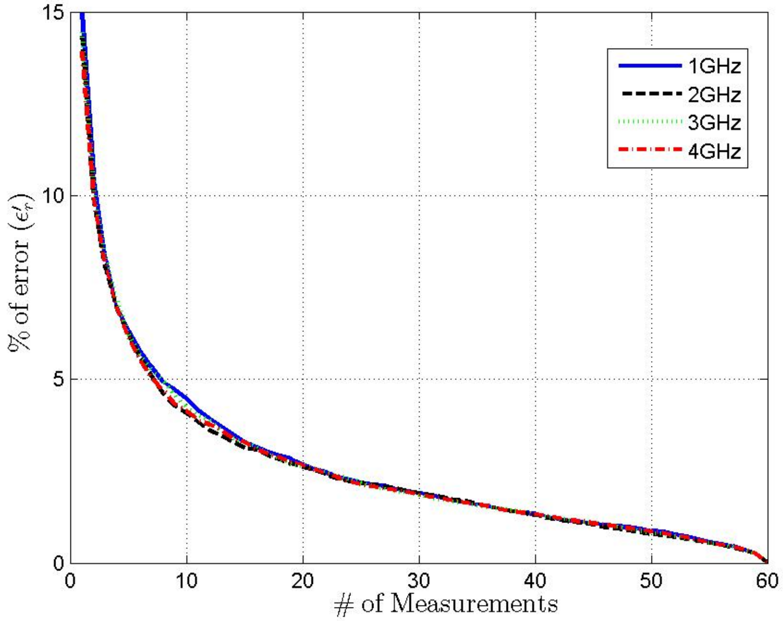


Figure 6-4: Frequency dependency of the percentage of error for sample CP40

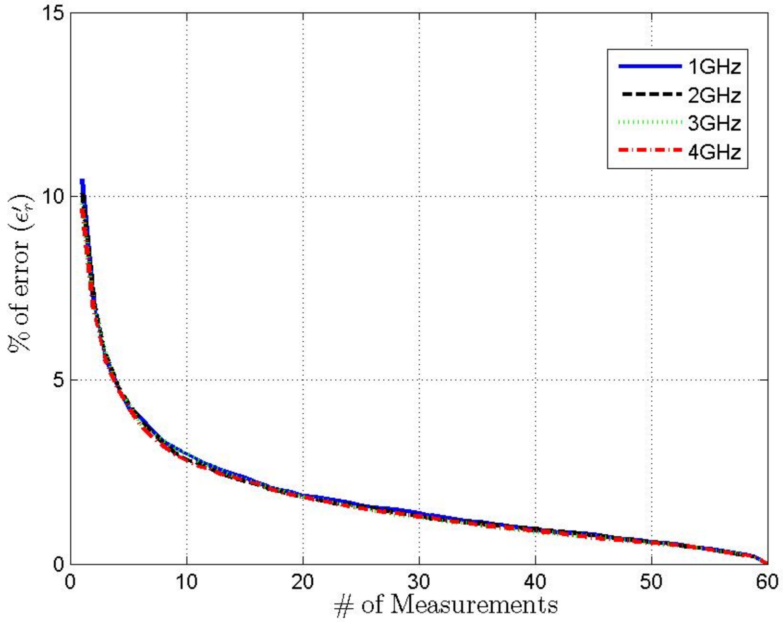


Figure 6-5: Frequency dependency of the percentage of error for sample CP42

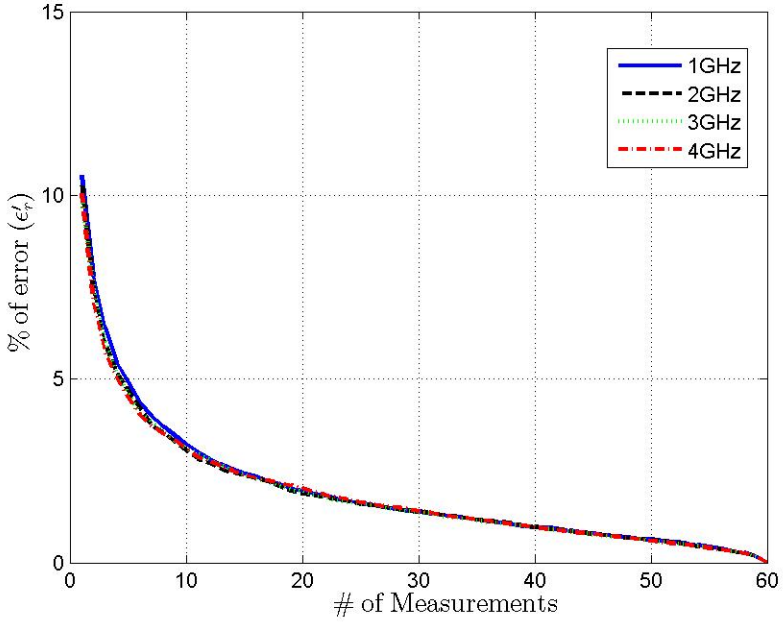


Figure 6-6: Frequency dependency of the percentage of error for sample CP45

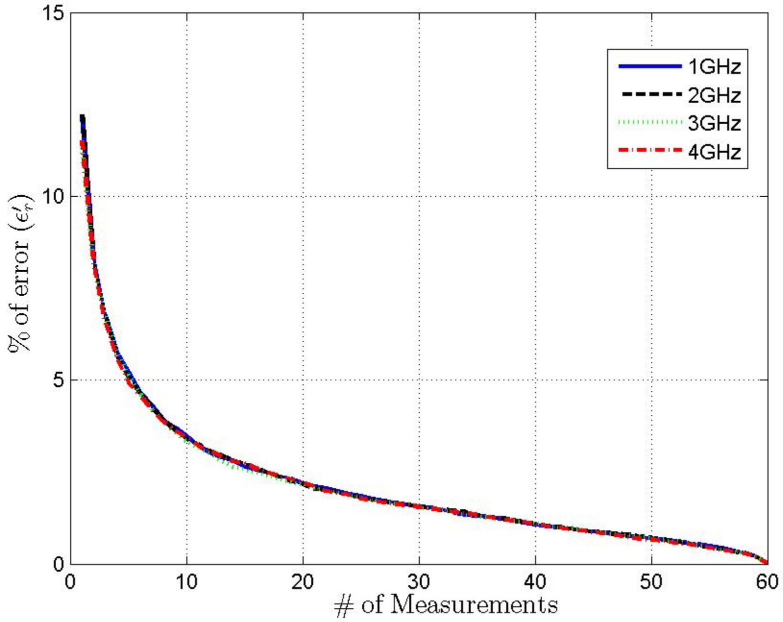


Figure 6-7: Frequency dependency of the percentage of error for sample CP50

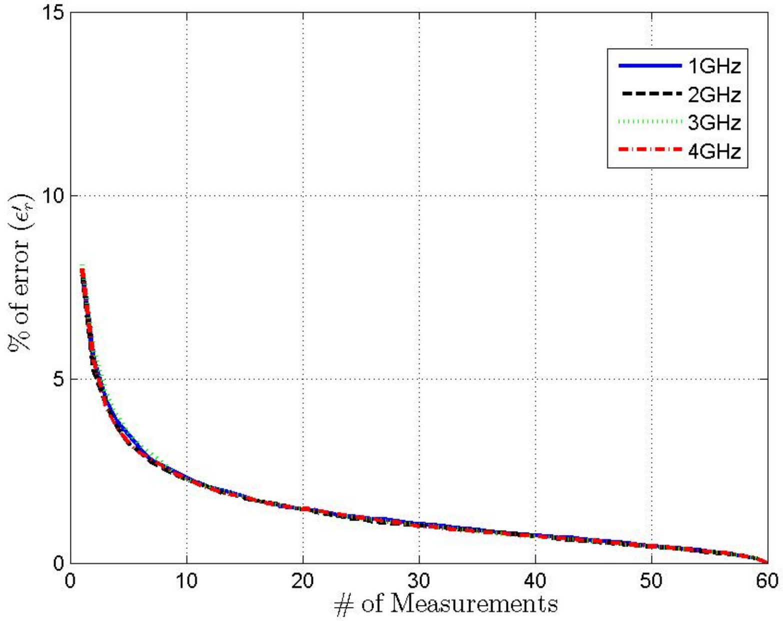


Figure 6-8: Frequency dependency of the percentage of error for sample CP55

6.2.2 Effect of the w/c Ratio on Error

The w/c ratio used in the design is very important on the heterogeneity of cement paste since it is directly related to porosity. The difference in the porosity leads to varying evaporable water content of cement paste. So uneven distribution of water throughout cement paste may result in considerable difference at dielectric constant of cement paste at different points measured. This parameter has been checked by comparing cement paste samples with different w/c ratios. Figure 6-9 and Figure 6-10 show the percentage of error vs. w/c ratio measured at 1 GHz and at 4 GHz. Observing different frequencies is unnecessary since we concluded earlier that the frequency has no considerable effect on error. As seen on Figure 6-9 and Figure 6-10 we were unable to detect a trend between error and w/c ratio, meaning that the evaporable water is evenly distributed within the pores, or the uneven distribution does not have a large influence that is detectable.

6.2.3 Effect of Evaporable Water on Error

Although the distribution of evaporable water throughout the sample does not effect the error, the removal of evaporable water from the sample is expected to have effect on error because of the high dielectric constant value of water. The dielectric constant of cement paste after oven drying procedure only depends on the air in pores and hydrated cement which have dielectric constant values close to each other as mentioned before. We analyzed the samples in the same manner after oven drying procedure. Figure 6-11 to Figure 6-16 shows the expected percentage of errors before and after the removal of

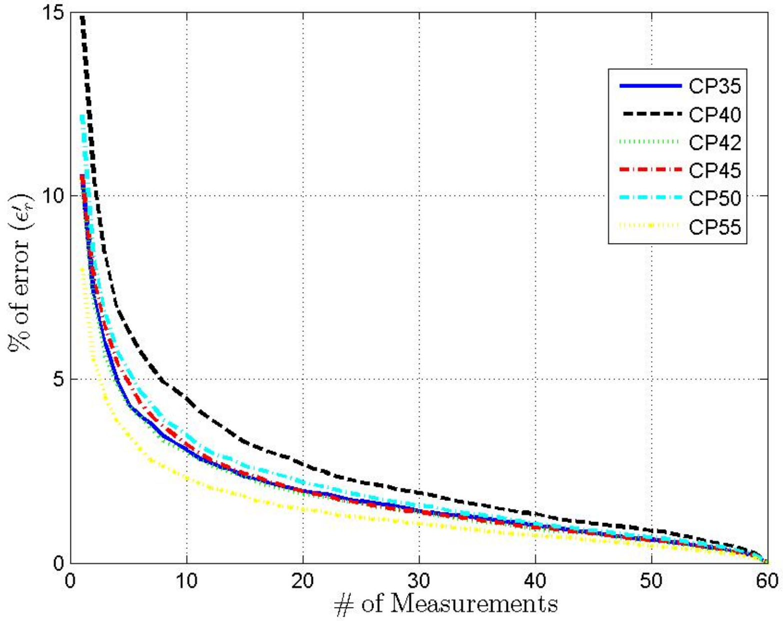


Figure 6-9: w/c ratio dependency of estimated error at 1 GHz

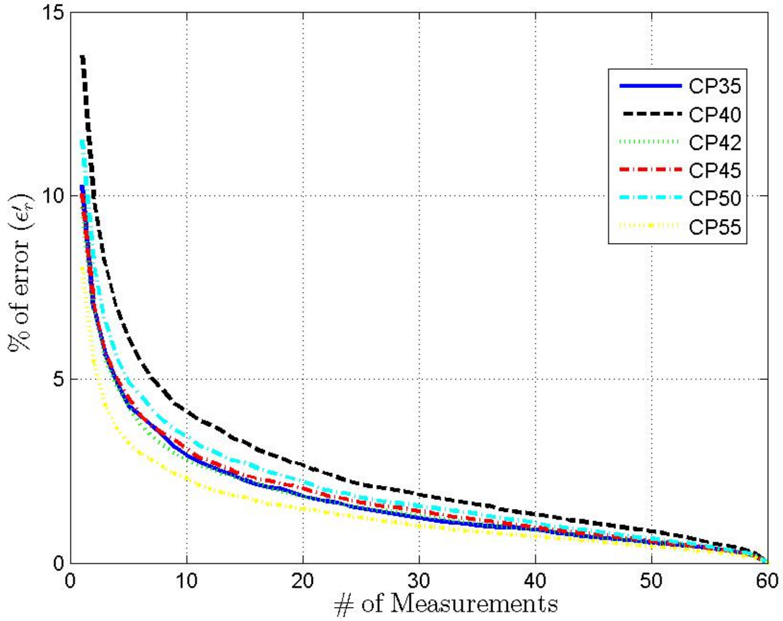


Figure 6-10: w/c ratio dependency of estimated error at 4 GHz

evaporable water. One important conclusion that can be made after observing the figures from Figure 6-17 to Figure 6-22, that compare the expected percentage of errors before and after oven drying, is that the percentage of error decreases when equal number of measurements are collected for all samples except for CP55. This is shown more clearly on Figure 6-23, where the average percentage of error of all samples with different w/c ratios are compared before and after oven drying procedure. The decreased expected error is a result of the decreasing influence of pores on the dielectric constant of cement paste. Cement paste is considered as a combination of hydrated cement and pores, and pores can be defined as a combination of air and water. When the pores are partially filled water the dielectric constant of pores is high compared to the pores with no water which causes values to fluctuate at different points of a sample. So when the water is removed we are left with only air and hydrated cement which have close dielectric constants, so a more even distribution through a sample is expected in terms of dielectric constant. Furthermore switching from three-phase content (hydrated cement, water, air) to two-phase content (hydrated cement) also allows us to observe a trend for the effect of w/c ratio (or porosity) after the removal of water. When we look at Figure 6-24 (1 GHz) and Figure 6-24 (4 GHz) we see that the percentage of error decreases as the w/c ratio decreases after oven drying procedure. The percentage of error after collecting ten measurements are compared for samples with different w/c ratios in Figure 6-26. It is observed that cement paste with low w/c ratio, has a more homogenous composition in terms of dielectric constant.

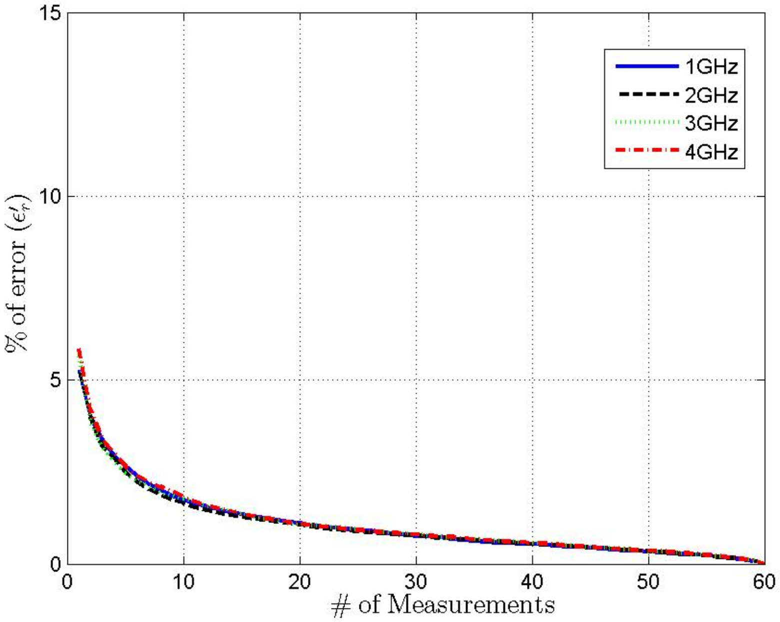


Figure 6-11: Frequency dependency of estimated error for sample CP35 after oven drying

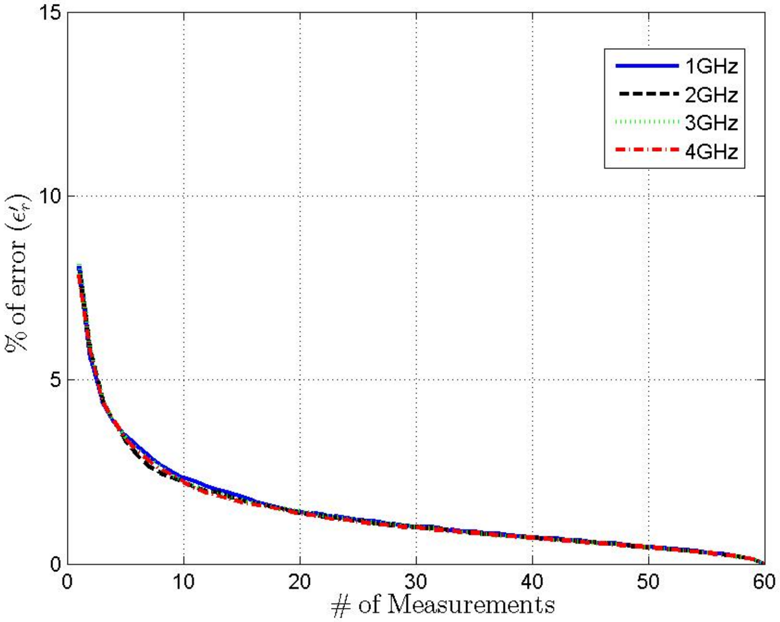


Figure 6-12: Frequency dependency of estimated error for sample CP40 after oven drying

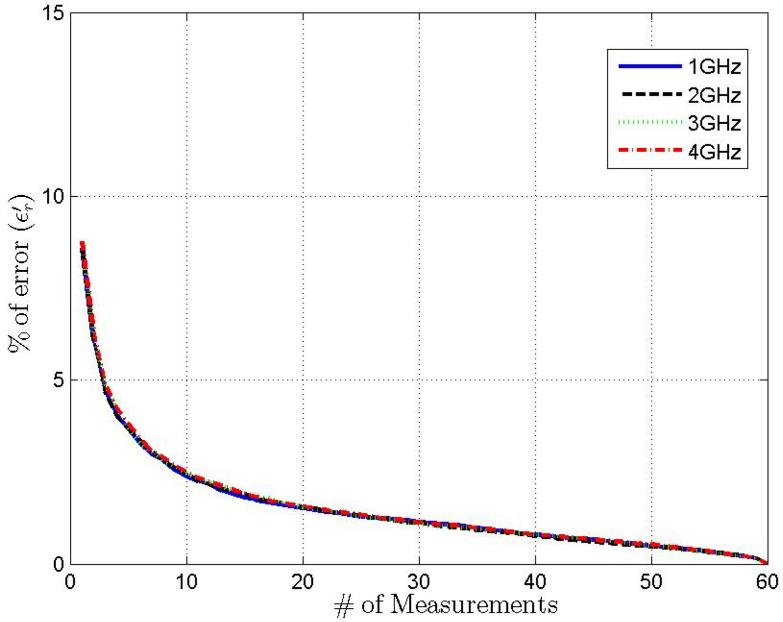


Figure 6-13: Frequency dependency of estimated error for sample CP42 after oven drying

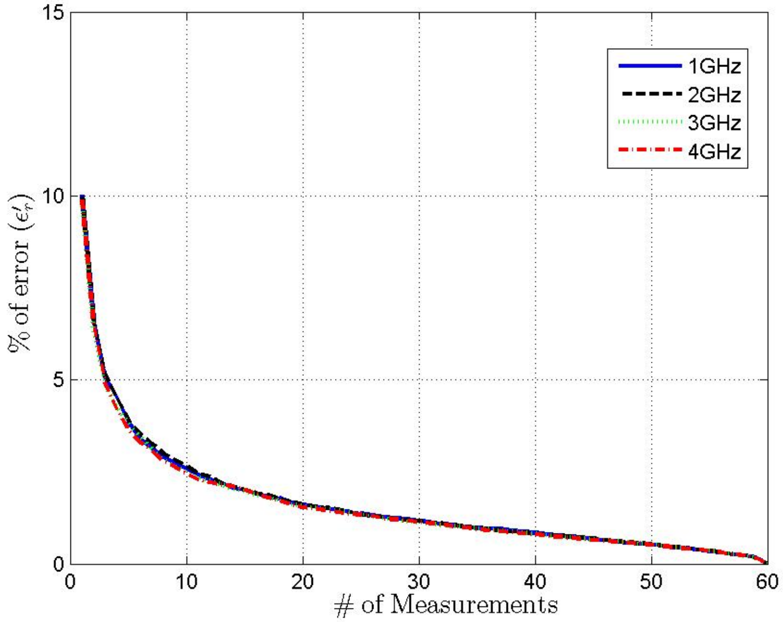


Figure 6-14: Frequency dependency of estimated error for sample CP45 after oven drying

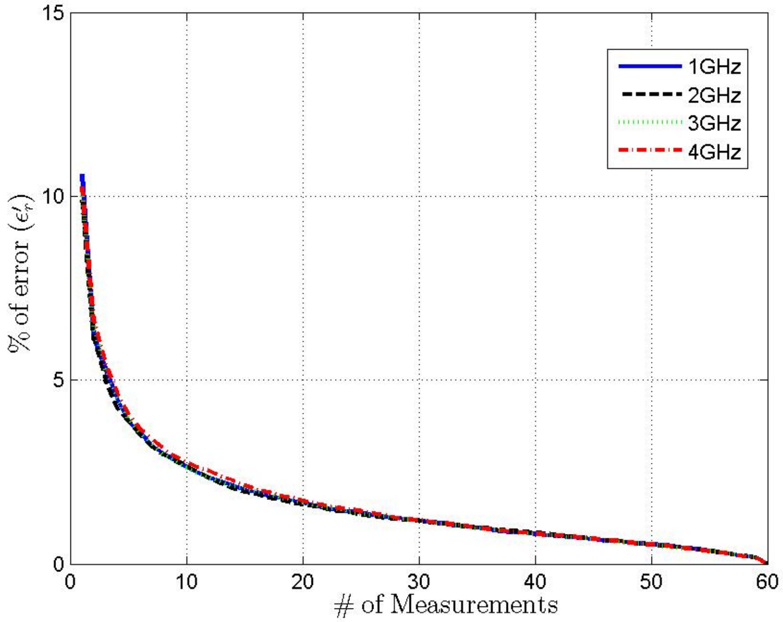


Figure 6-15: Frequency dependency of estimated error for sample CP50 after oven drying

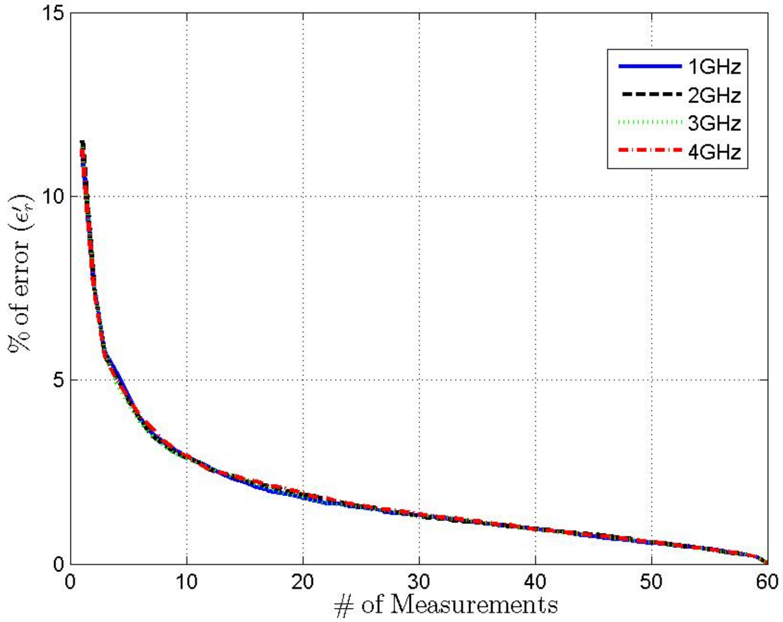


Figure 6-16: Frequency dependency of estimated error for sample CP55 after oven drying

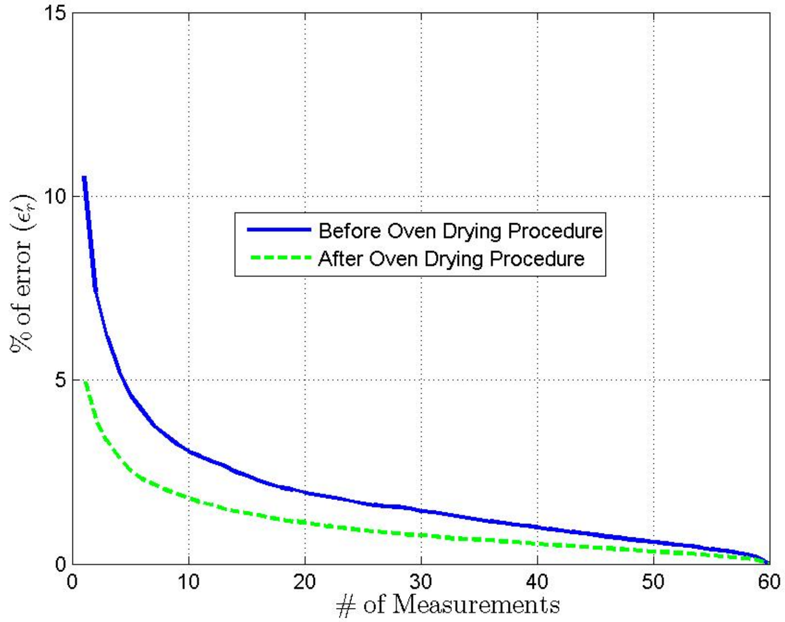


Figure 6-17: The expected error for CP35 before and after oven drying

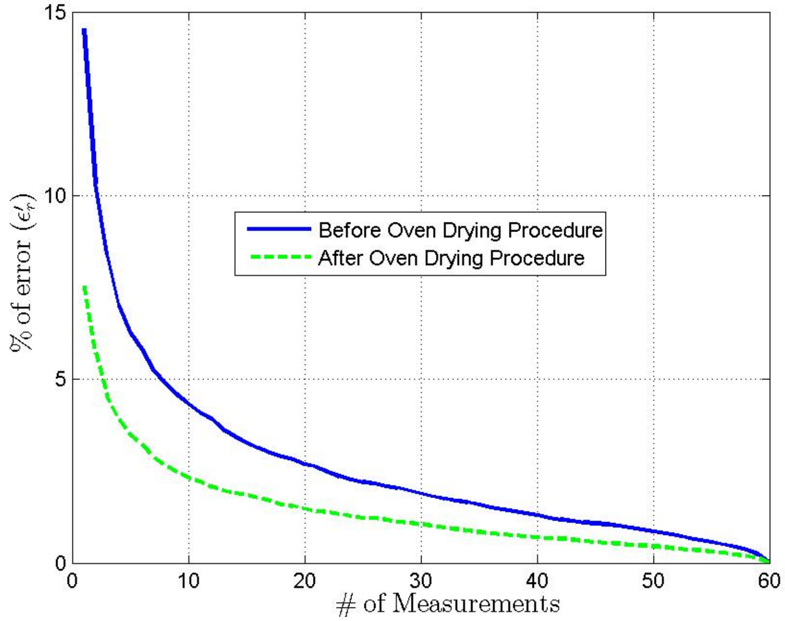


Figure 6-18: The expected error for CP40 before and after oven drying

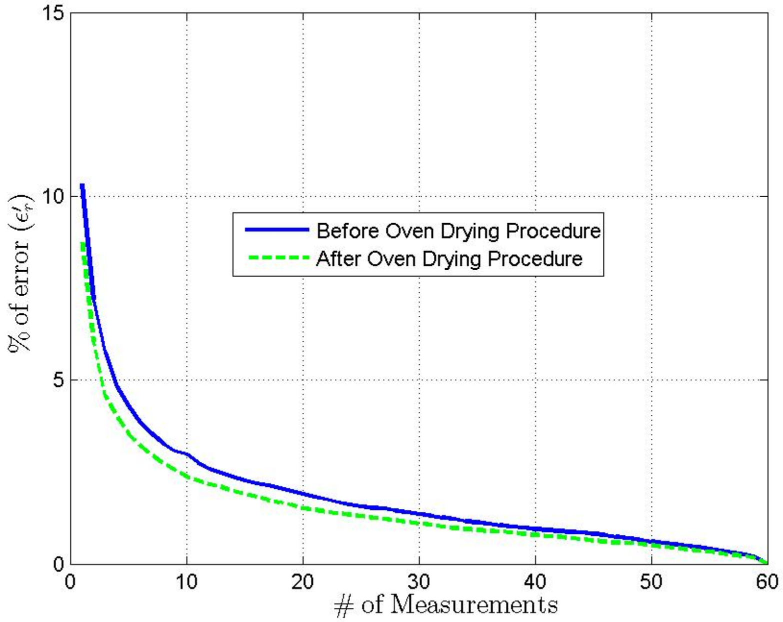


Figure 6-19: The expected error for CP42 before and after oven drying

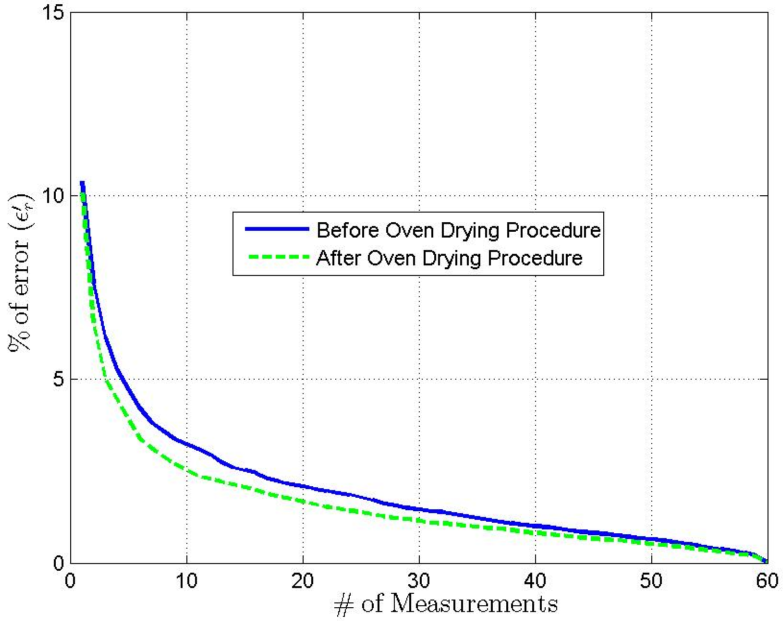


Figure 6-20: The expected error for CP45 before and after oven drying

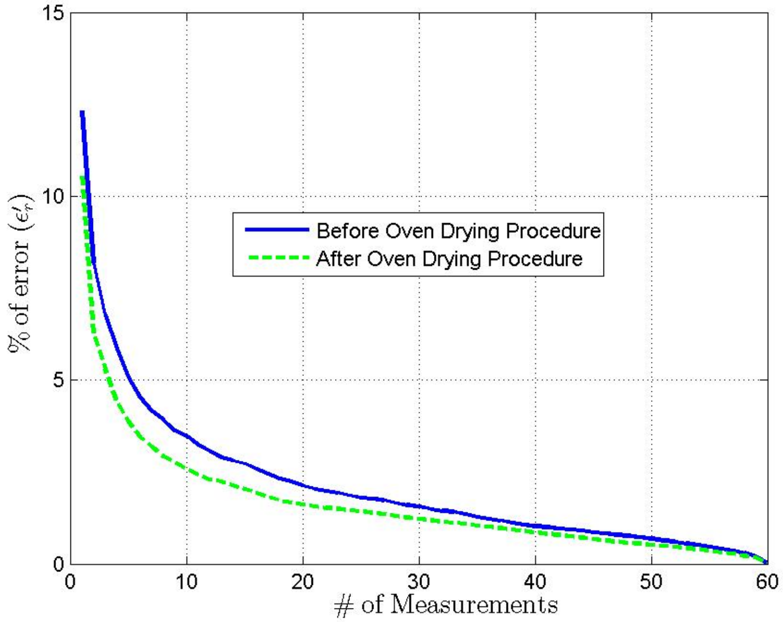


Figure 6-21: The expected error for CP50 before and after oven drying

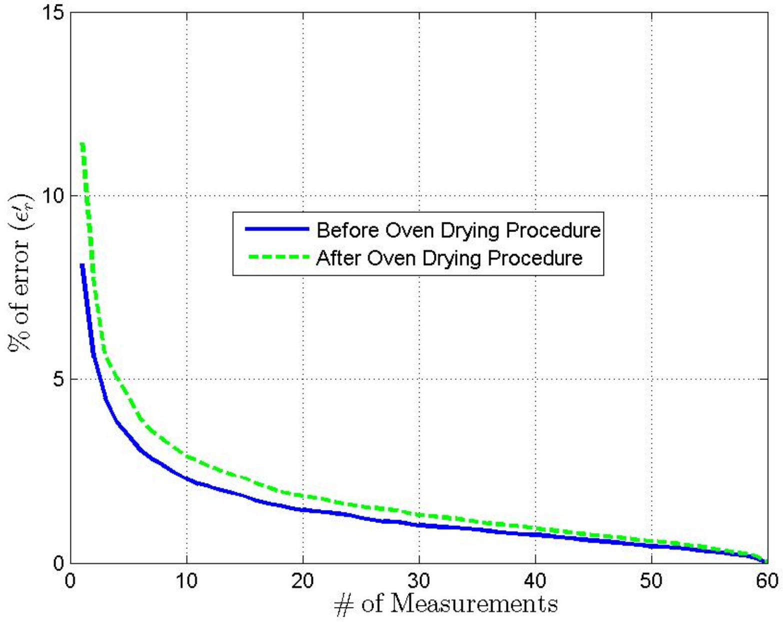


Figure 6-22: The expected error for CP55 before and after oven drying

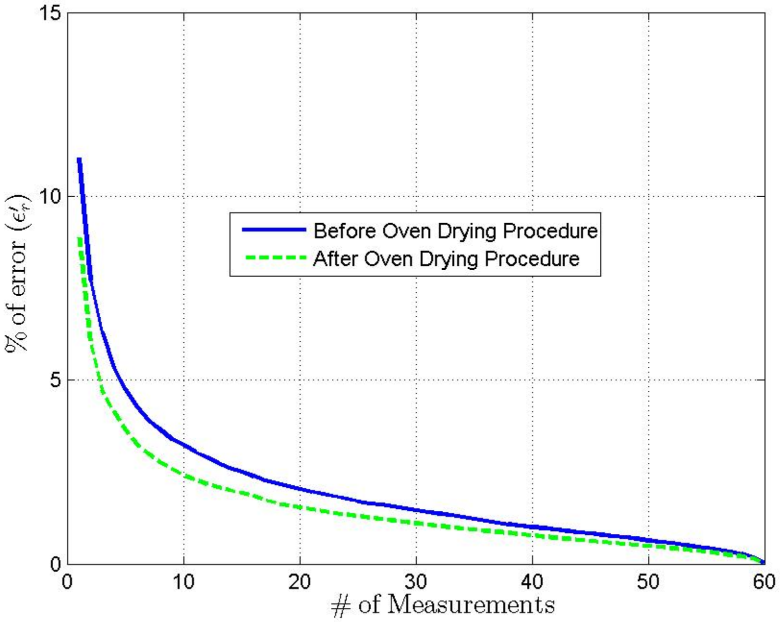


Figure 6-23: Effect of the removal of evaporable water on error for Cement Paste

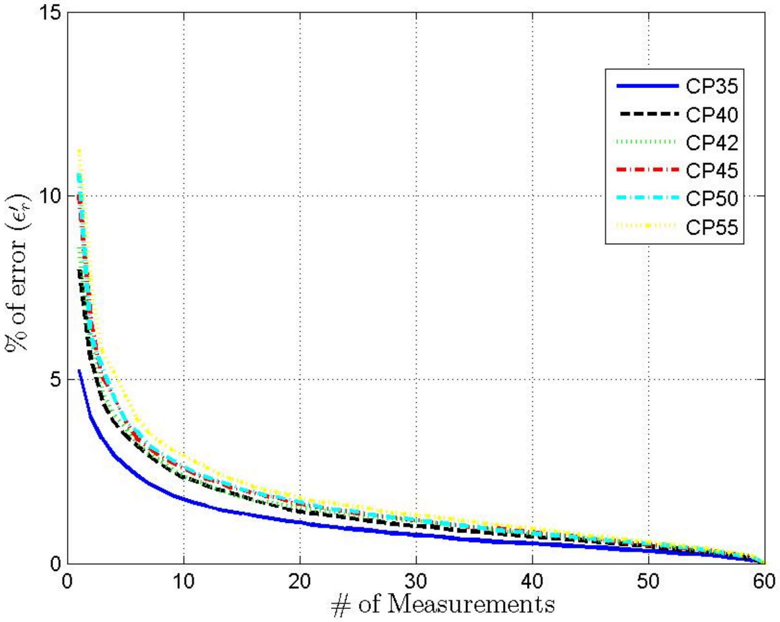


Figure 6-24: w/c ratio dependency of error after the removal of evaporable water at 1 GHz

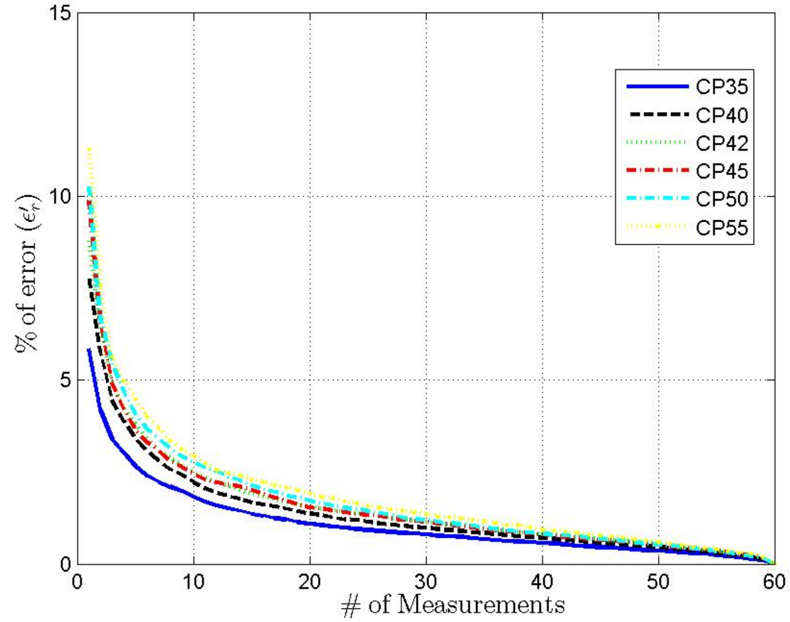


Figure 6-25: w/c ratio dependency of error after the removal of evaporable water at 4 GHz

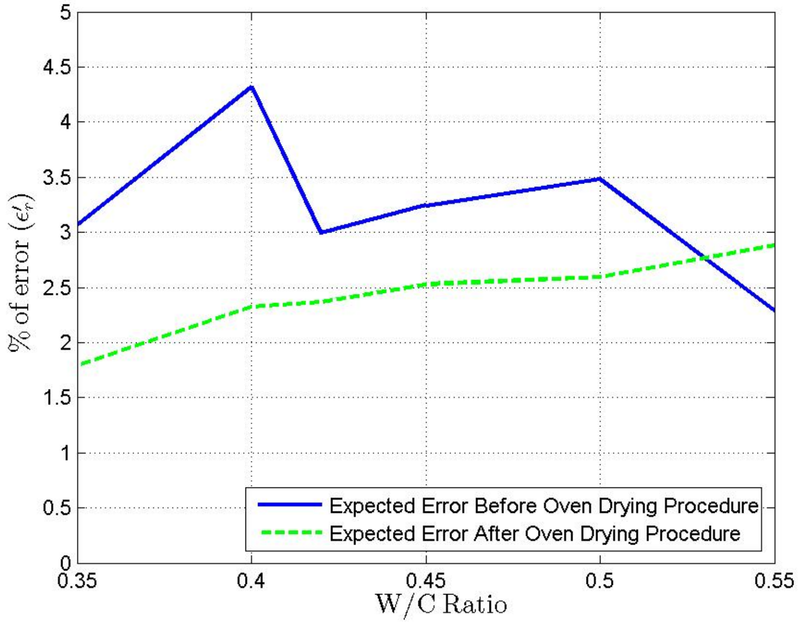


Figure 6-26: The expected error decreases as the w/c ratio increases

6.3 Modeling of Percentage of Error

We decided to use power equation to model the expected percentage of error for cement paste samples. Since the frequency of the measurements and the w/c ratio has no or negligible effect on the expected error we used the average expected error of all six samples at 2 GHz frequency. The model fits fine to the measured data as seen in Figure 6-27. The calculated coefficients are shown below where y is the percentage of error, and x is the number of measurements.

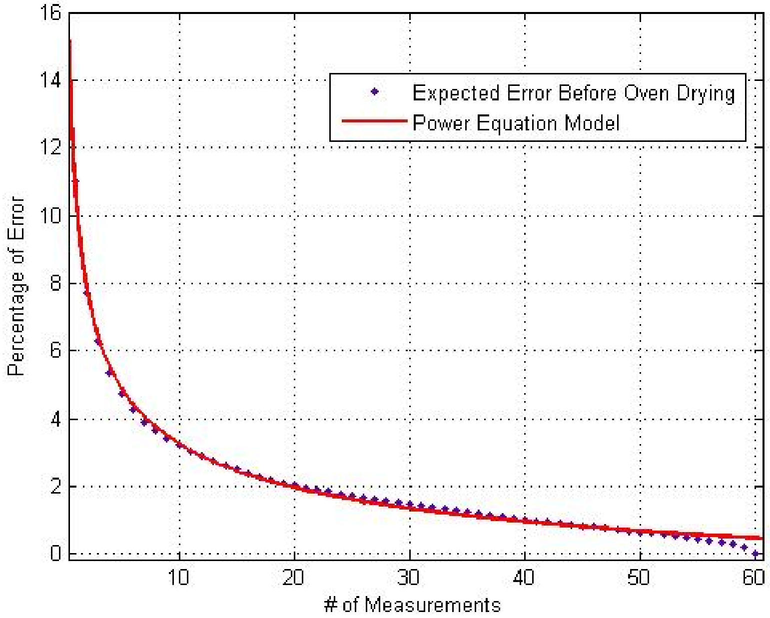


Figure 6-27: The estimated error is modeled by using a power fit before oven drying

$$y = a_1 \times x^{b_1} + c_1 \tag{6.7}$$

where the coefficients are;

a_1 = 13.420, b_1 = - 0.359, c_1 = - 2.621

Since the expected error is going to be less after the oven drying procedure we have also modeled the case after oven drying. Although the expected error is w/c ratio dependant the effect is not very crucial so the average of six samples is used again. The proposed model is shown on Figure 6-27. The calculated coefficients are shown below where y is the percentage of error, and x is the number of measurements.

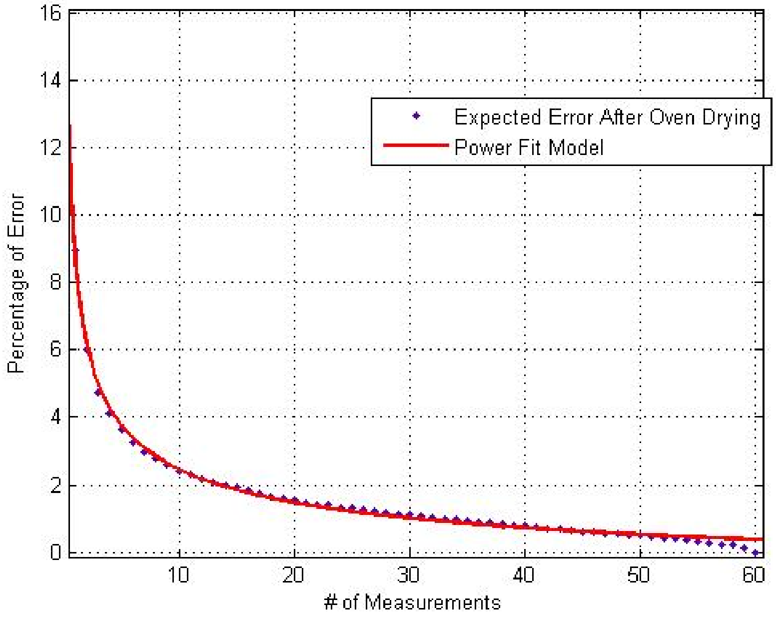


Figure 6-28: The estimated error is modeled by using a power fit after oven drying

$$y = a_2 \times x^{b_2} + c_2 \tag{6.8}$$

where the coefficients are; $a_2 = 10.26$, $b_2 = -0.410$, $c_2 = -1.536$

6.4 Summary

Dielectric constant of cement paste panels have been calculated using coaxial contact probe. It has been shown that a single measurement is not enough to approximate the dielectric constant of cement paste. The relation between the error and number of measurements is modeled using power equation. The required number of measurements can be found for desired error percentage using the model. The frequency of the measurement and w/c ratio of the cement paste being measured has very little effect on the reliability of the measurements, so can be neglected. But when the evaporable water is removed by oven drying, more reliable results will be obtained even with the same number of measurements compared to not oven dried samples.

Chapter 7

Conclusions

In this thesis, dielectric properties of cementitious materials are studied. The effect of water, air and sand on the dielectric properties of cementitious composites are studied. Agilent Technologies E5071C ENA Series Network Analyzer was used with a coaxial contact probe. Cement paste and cement mortar samples with varying w/c ratios from 0.35 to 0.5 were cast and cured for seven days in water. After three months of conditioning in room conditions, measurements were collected. Sixty measurements were collected from different regions of each sample and the average of sixty measurements was used. Research findings and future work are provided in this chapter.

7.1 Research Findings

- **Cement Paste**

  As the w/c ratio increases the porosity of cement paste increases. Cement paste consists of hydrated cement, voids, and the voids are partially filled with water and air (depending on the humidity of the environment). At room conditions we observed that the dielectric constant decreases as the w/c ratio increases. This behavior is due to the low dielectric constant of the air in voids and high volume occupied by voids for high w/c ratio samples. Also, when the water within the voids is removed by the oven drying procedure, the dielectric constant of a cement paste sample decreases.

- **Cement Mortar**

  Cement mortar has sand in addition to all other ingredients in cement paste. The dielectric constant of sand is lower than the one of cement paste. Therefore, cement mortar has a lower dielectric constant, compared to the cement paste of same w/c ratios. Also, as observed on cement paste, the dielectric constant of cement mortar decreases as the w/c ratio increases. The quantity of the sand used in design affects the reduction rate of the dielectric constant of cement mortar. More sand used in design will lead to further reduction in the dielectric constant.

- **Reliability of the measurements**

  In view of the heterogenous structure of cement paste, sufficient data points are needed to collected from a sample in order to obtain a reliable value representing the whole sample. Since the proportion of water, air and hydrated cement is not same at each point, an average value collected from many points will increase the accuracy of the value. Monte Carlo simulations were used to relate the error with the number of measurements. A power equation model is proposed to relate the number of data points with the expected error. After the oven drying of the samples, the measurements collected from a sample seem to fluctuate less at different points, compared to the measurements before oven drying. This indicates that the removal of water provides a more dielectrically homogenous structure.

7.2 Future Work

In this thesis, cement paste and cement mortar samples with no anomalies (reinforced concrete, introduced cracks) were considered. By introducing anomalies, conditions that is expected on field can be better understood. Future measurements by using the contact coaxial probe method can be conducted on concrete with introduced cracks, reinforcing bars, corroded reinforcing bars and different types of aggregates. Also, when the reinforcing bars corrode, rust can penetrate through the cracks in concrete. Detecting the change in dielectric constant due to rust penetration can provide valuable information for the condition assessment of reinforced concrete structure. In addition, comparing field measurements (collected from old, demolished structures or structures in service) and laboratory measurement can provide a better understanding of the effects of environmental conditions and aging of cementitious composites on the dielectric properties of cementitious composites. Building a free space measurement system can allow us to compare the data obtained by free space method with the data obtained by the contact coaxial probe method. This way the relation between local measurements can be compared with global measurements.

Bibliography

[1] Agilent. Application note: Agilent basics of measuring the dielectric properties of materials. *Agilent Literature Number 5989-2589EN*, June 2006.

[2] I. L. Al-Qadi, O. A. Hazim, W. Su, and S. M. Riad. Dielectric properties of portland cement concrete at low radio frequencies. *Journal of Materials in Civil Engineering*, 7(3):192–8, 8 1995.

[3] X. Z. Ding, X. Zhang, C. K. Ong, B. T. G. Tan, and J. Yang. Study of dielectric and electrical properties of mortar in the early hydration period at microwave frequencies. *Journal of Materials Science*, 31:5339–45, 1996.

[4] S. J. Ford, J. H. Hwang, J. D. Shane, R. A. Olson, G. M. Moss, H. M. Jennings, and T. O. Mason. Dielectric amplification in cement pastes. *Advn. Cem. Bas. Mat.*, 5:41–8, 1997.

[5] J. E. Gentle. *Random Number Generation and Monte Carlo Methods*. Springer, Berlin, Germany, 2003.

[6] D. K. Ghodgaonkar, V. V. Varadan, and V. K. Varadan. A free-space method for measurements of dielectric constants and loss tangents at microwave frequencies. *Transactions on Instr. and Meas.*, 37(3):789–93, June 1989.

[7] T. T. Grove, M. F. Masters, and R. E. Miers. Determining dielectric constants using a parallel plate capacitor. *Am. J. Phys.*, 73(1):52–6, January 2005.

[8] Y. Hafiane, A. Smith, P. Abelard, J. P. Bonnet, and P. Blanchart. Dielectric characterization at high frequency (1 mhz - 1.8 ghz) of a portland cement at early stages of hydration. *Ceramics*, 43(2):48–51, 12 1999.

[9] Y. Hafiane, A. Smith, J. P. Bonnet, P. Abelard, and P. Blanchart. Electrical characterization of aluminous cement at the early age in the 10 hz - 1 ghz frequency range. *Cement and Concrete Research*, 30:1057–62, 4 2000.

[10] N. E. Hager and R. C. Domszy. Monitoring of cement hydration by broadband time-domain-refloctometry dielectric spectroscopy. *Jounal of Applied Physics*, 96:5117–28, 9 2004.

[11] J. B. Hasted and M. A. Shah. Microwave absorption by water in building materials. *Brit. J. Appl. Phys.*, 15:825–36, 2 1964.

[12] A. Hu, Y. Fang, J. F. Young, and Y. Oh. Humidity dependence of apparent dielectric constant for dsp cement based materials at high frequencies. *J. Am. Ceram. Soc.*, 82(7):1741–7, 1999.

[13] V. Janoo, C. Korhonen, and M. Hovan. Measurement of water content in portland cement concrete. *Journal of Transportation Engineering*, 125(3):245–9, June 1999.

[14] A. W. Kraszewski and S. O. Nelson. Resonant cavity perturbating - some new applications of an old measuring technique. *Journal of Microwave Power and Electromagnetic Energy*, 31(3):178–87, 1994.

[15] G. P. D. Loor. The effect of moisture on the dielectric constant of hardened portland cement paste. *Appl. Sci. Res.*, 9:297–308, 1961.

[16] N. Makul, P. Keangin, P. Rattanadecho, B. Chatveera, and D. K. Agrawal. Microwave-assisted heating of cementitious materials: Relative dielectric properties, mechanical property, and experimental and numerical heat transfer characteristics. *International Communications in Heat and Mass Transfer*, 37:1096–105, 2010.

[17] J. Maxwell. *A Treatise on Electricity and Magnetism*. Dover Publication, New York, NY, 1954.

[18] W. J. McCarter, G. Starrs, and T. M. Chrisp. The complex impedance response of fly-ash cements revisited. *Cement and Concrete Research*, 34:1837–43, 2004.

[19] N. Miura, N. Shinyashiki, S. Yagihara, and M. Shiotsubo. Microwave dielectric study of water structure in the hydration process of cement paste. *J. Am. Ceram. Soc*, 81(1):213–6, 1998.

[20] M. Moukwa, M. Brodwin, S. Christo, J. Chang, and S. P. Shah. The influence of the hydration process upon microwave properties of cements. *Cement and Concrete Research*, 21:863–72, 6 1991.

[21] K. Mubarak, K. J. Bois, and R. Zoughi. A simple, robust, and on-site microwave technique for determining water-to-cement ratio (w/c) of fresh portland cement-based materials. *IEEE Transactions on the Instrumentation and Measurement*, 50(5):1255–63, 10 2001.

[22] K. Olp, G. Otto, W. C. Chew, and J. F. Young. Electromagnetic properties of mortars over a broad frequency range and different curing times. *Journal of Materials Science*, 26:2978–84, 1991.

[23] S. Peer, K. E. Kurtis, and R. Zoughi. An electromagnetic model for evaluating temporal water content distribution and movement in cyclically soaked mortar. *IEEE Transactions on Instrumentation and Measurement*, 53(2):406–15, April 2004.

[24] S. Peer and R. Zoughi. Comparison of water and saltwater movement in mortar based on a semiempirical electromagnetic model. *IEEE Transactions on Instrumentation and Measurement*, 53(4):1218–23, August 2004.

[25] R. Rianyoi, R. Potong, N. Jaitanong, R. Yimnirun, A. Ngamjarurojana, and A. Chaipanich. Dielectric and ferroelectric properties of 1-3 barium titanateportland cement composites. *Current Applied Physics*, pages 1–4, 2011.

[26] F. Sagnard and G. E. Zein. In-situ characterization of building materials for propagation modeling: frequency and time responses. *IEEE Transactions on Antennas and Propagation*, 53(10):3166–73, October 2005.

[27] W. Shalaby and R. Zoughi. Microwave compressive strength estimation of cement paste using monopole probes. *Research in Nondestructive Evaluation*, 7:101–115, 6 1995.

[28] A. Smith, P. Abelard, F. Thummen, and A. Allemand. Electrical characterisation as a function of frequrncy: application to aluminous cement during early hydration. *Cement and Concrete Composites*, 24:477–84, 2002.

[29] J. M. Tobio. A study of the setting process. dielectric behaviour of several spanish cements. *Silicates Industrials*, 24:30–5, 1 1959.

[30] C. Tsonos, I. Stavrakas, C. Anastasiadis, A. Kyriazopoulos, A. Kanaspitsas, and D. Triantis. Probing the microstructure of cement mortars through dielectric parameters variation. *Journal of Physics and Chemistry of Solids*, 70:576–83, 2009.

[31] M. S. Venkatesh. Cavity perturbating technique for measurements of dielectric properties of some agri-food materials. Master's thesis, McGill University, Montreal, 1996.

[32] S. Wen and D. D. L. Chung. Effect of admixtures on the dielectric constant of cement paste. *Cement and Concrete Research*, 31:673–7, 2 2001.

[33] S. Wen and D. D. L. Chung. Cement-based materials for stress sensing by dielectric measurement. *Cement and Concrete Research*, 32:1429–33, 3 2002.

[34] F. H. Wittmann and F. Schlude. Microwave absorption of hardened cement paste. *Cement and Concrete Research*, 5:63–71, 1 1975.

[35] F. Xing, B. Dong, and Z. Li. Dielectric, piezoelectric, and elastic properties of cement-based piezoelectric ceramic composites. *J. Am. Ceram. Soc.*, 91:2886–91, 9 2008.

[36] S. S. Yoon, H. C. Kim, and R. M. Hill. The dielectric response of hydrating porous cement paste. *J. Phys. D: Appl. Phys.*, 29:869–755, 11 1996.

[37] T.-Y. Yu. *Damage Detection of GFRP-Concrete Systems Using Electromagnetic Waves*. Lambert Academic Publishing, Koln, Germany, 2009.

[38] X. Zhang, X. Z. Ding, T. H. Lim, C. K. Ong, and B. T. G. Tan. Microwave study of hydration of slag cement blends in early period. *Research in Nondestructive Evaluation*, 25:1086–94, 5 1995.