Structural Engineering Research Group
(SERG)
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Determination of Dielectric Constant of Hydrated Cement Paste and Cement Mortar Using Contact Coaxial Probe

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Outline

- Introduction
- Objective
- Literature Review
- Experimental Setup
- Specimen Preparation and Data Collection
- Measurements on Cement Paste
- Measurements on Cement Mortar
- Reliability of the Measurements
- Conclusion
- Future Work
- References

Introduction

- **Why Non Destructive Testing/Evaluation (NDT-NDE) is necessary**
  - To extend the service life of the structure
  - Public Safety
  - Feasibility
- **Common NDT Methods– Type of Wave/Field**
  - Optical Methods - *EM (Electromagnetic) waves in optical spectrum*
  - Acoustic Methods - *Mechanical Waves*
  - Thermal Methods - *EM waves in thermal radiation spectrum*
  - Radiographic Methods - *X-ray, Gamma rays and Neutrons*
  - Magnetic and Electric Methods - *Magnetic field and electrical field*
  - Microwave / Radar Methods - *EM waves in microwave spectrum and radio frequency range*

Introduction

- **Microwave / Radar Methods**

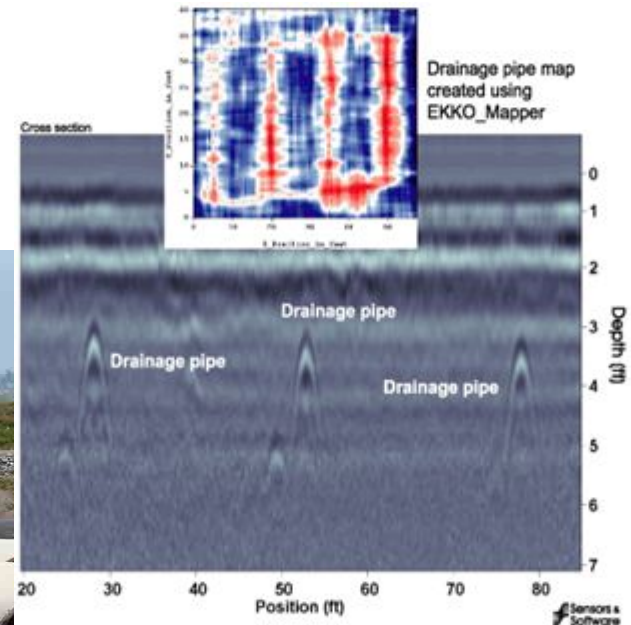
- **Dielectric constant** is the ratio of the stored energy in a material under electrical field, relative to that stored in a vacuum.
- Dielectric materials (e.g., concrete) allow EM waves to propagate inside – Subsurface analysis can be performed.
- Anomalies inside a medium can be detected. For reliable and accurate determination of the size and the depth of anomalies the speed of EM wave propagation is needed, which depends on the dielectric constant of that medium.
- A comprehensive study is needed for the material characterization of concrete, cement paste and cement mortar. Dielectric constant of these materials depends on many factors including **design values of the cementitious composites** (e.g, water-to-cement ratio (w/c), sand-to-cement ratio (s/c)), and **environmental factors** (e.g., relative humidity, temperature).

Introduction

Microwave / Radar Method Applications (300 MHz ~ 300 GHz)

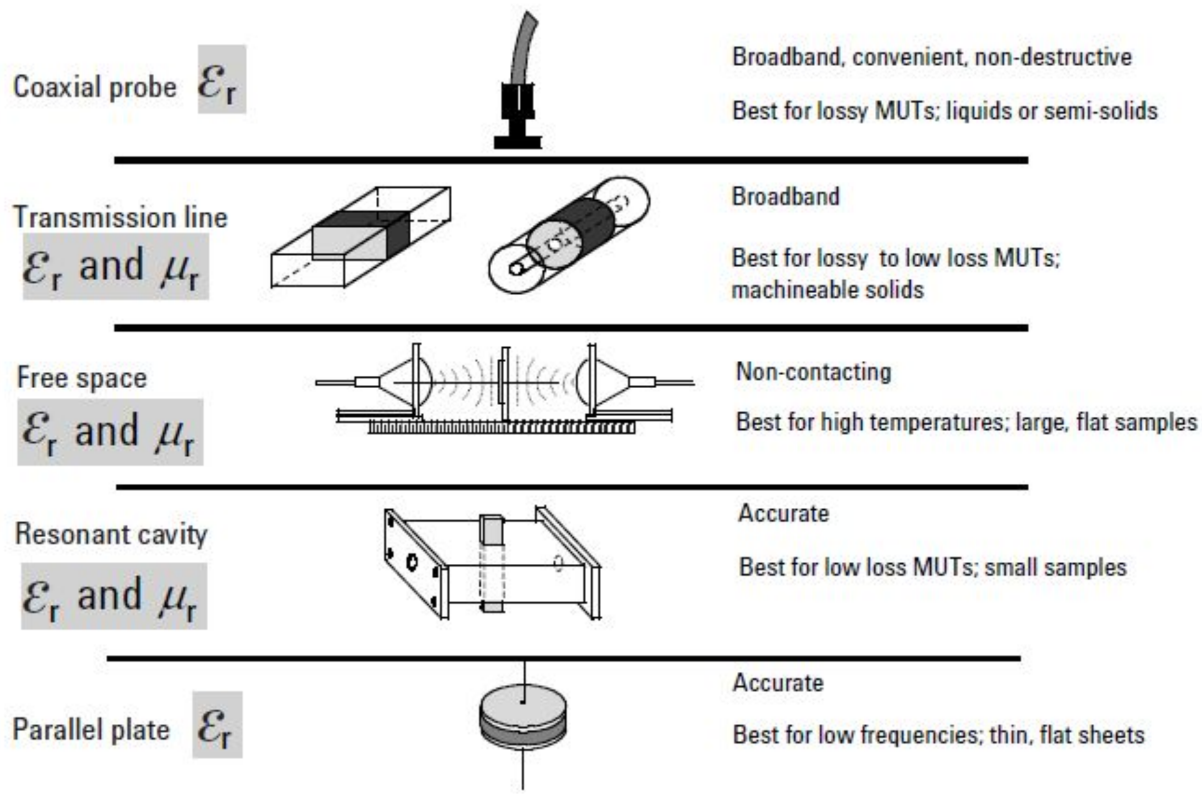
- Underground Object Detection (e.g., rebar, void, crack, pipe line)
- Ground Water Detection
- Material Characterization

Ground Penetrating Radar (GPR)



Introduction

Measurement Methods for Dielectric Constant Determination



(Source: Agilent® Technologies)

Objective

- Determination of the relationship between the dielectric constant, measurement frequency and the water-to-cement (w/c) ratio of cement paste and cement mortar specimens.
- Observations on the effect of evaporable water on the dielectric constant of cement paste specimens.
- Determination of the relationship between the dielectric constant and the sand-to-cement (s/c) ratio of cement mortar specimens.
- Error analysis of the collected data with coaxial contact method.
- Determination of the dielectric heterogeneity of cement paste specimens.

Literature Review

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.*

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.*

Cement Mortar		
Free Space	8 GHz - 12.5 GHz	Sagnard *et. al.*
Waveguide	8.2 GHz - 12.4 GHz 3 GHz	Ding *et. al.* 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.*

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Experimental Setup

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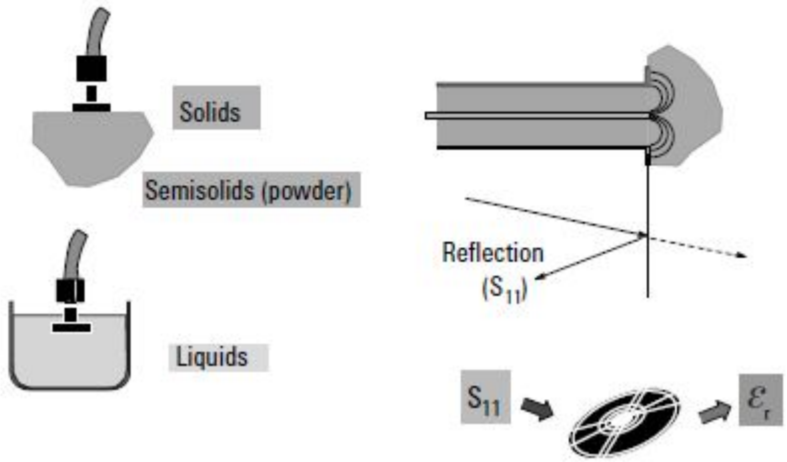
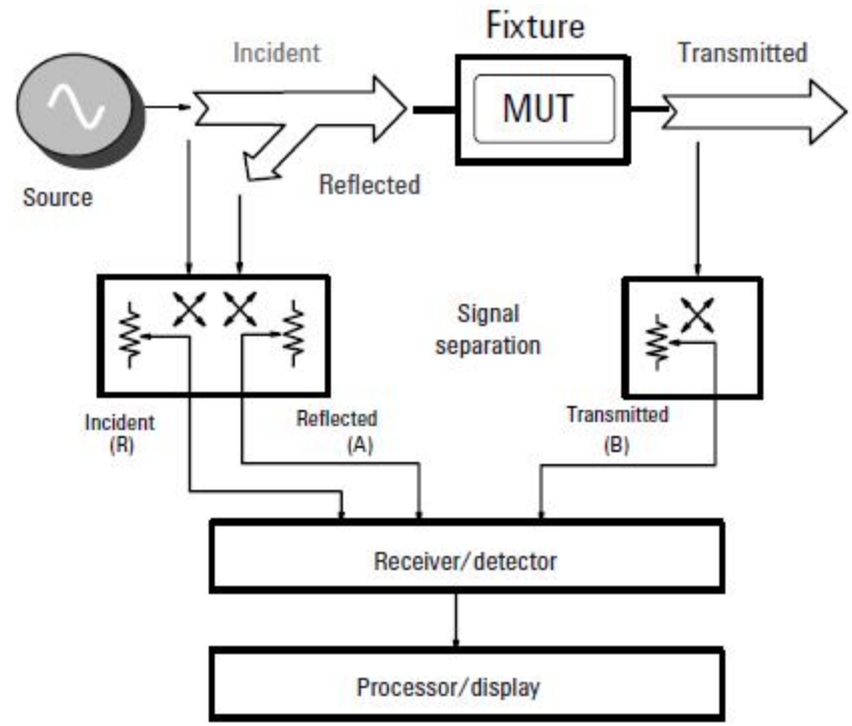
- Agilent® E5071C ENA Series Network Analyzer (0.0001 GHz – 4.5 GHz)
- Agilent® 85070E Performance Coaxial Probe (0.5 GHz – 50 GHz)

Experimental Setup

Measurement Mechanism



(Source: Agilent® Technologies)



(Source: Agilent® Technologies)

S Parameters = Scattering Parameters

S_{11}= Reflection Coefficient

The EM fields at the probe end penetrate into the material and change as they come into contact with the specimen. The reflected signal (S_{11}) can be measured and related to the dielectric constant of the Material Under Testing (MUT).

Experimental Setup

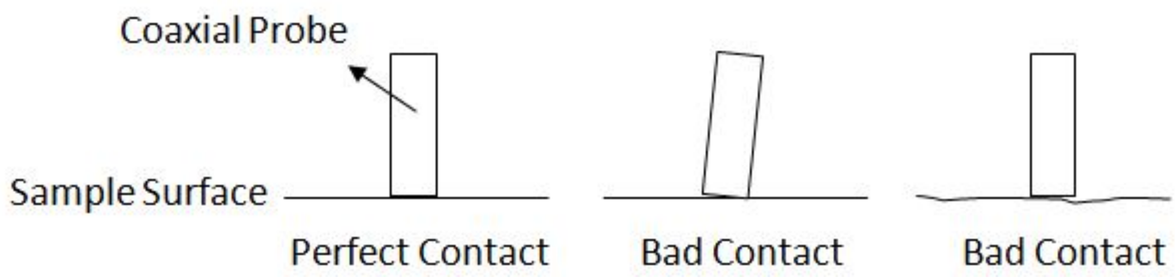
Measurement Calibration

(Source: Agilent® Technologies)

- Before measuring, calibration at the tip of the probe must be performed.
- Three known standards are used to calibrate the device which are water, air and shorting kit.
- Three main sources of error that can affect measurement accuracy are;
  - Cable Stability
  - Specimen Thickness
  - Air Gaps

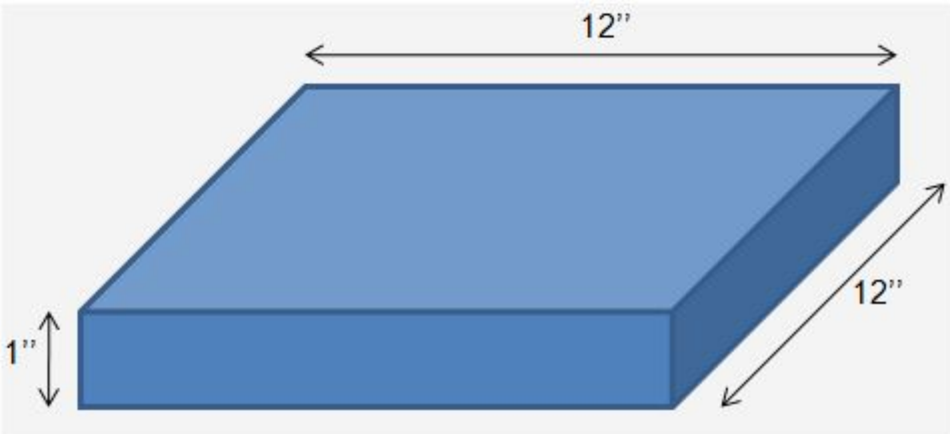
Experimental Setup

Measurement Accuracy



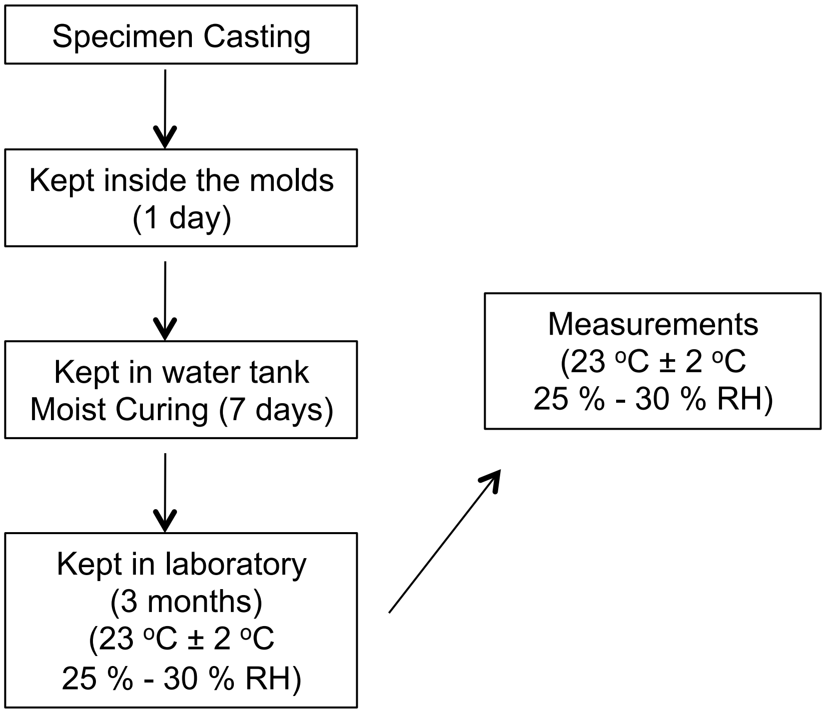
- During the measurements the probe end has to be in perfect contact with the specimen surface. Any air gap between the probe and the specimen surface leads to inaccurate measurements.
- The surface smoothness needs to provided during the manufacturing phase of the specimens.

Specimen Preparation



- Dimensions of the specimens that have been used for the experiment are 1 in.-by-1 ft.-by-1 ft. The dimensions are chosen to provide enough surface area and thickness.
- The inner sides of the molds have been covered with plexiglass to provide desired surface smoothness.

Specimen Preparation

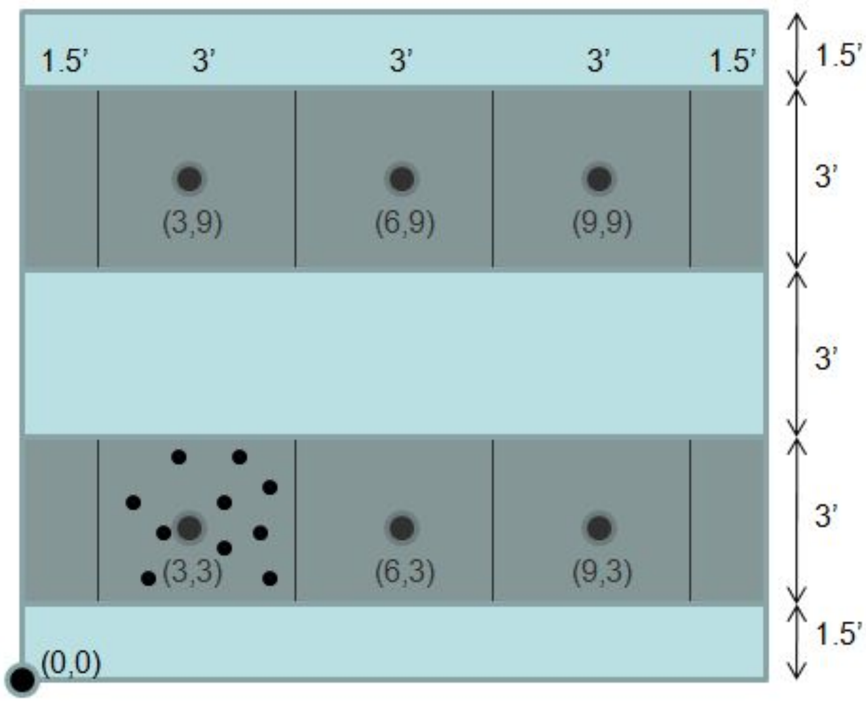


Cement Mortar		
S/C	W/C	Sample
2.53	0.35	CM35
	0.40	CM40
	0.42	CM42
	0.45	CM45
	0.50	CM50
	0.55	CM55
1.90	0.50	CM50S

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

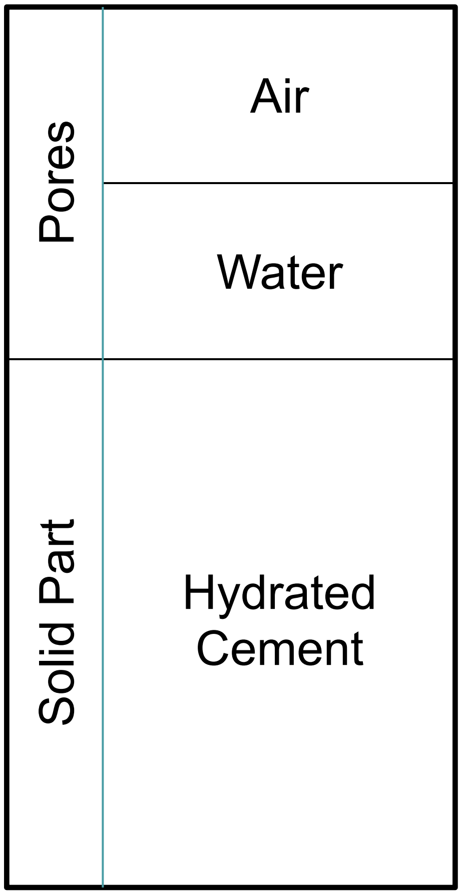
- Six cement paste specimens were prepared with various w/c ratios (0.35, 0.40, 0.42, 0.45, 0.50, 0.55)
- Seven cement mortar specimens had been prepared with various w/c ratios and s/c ratios.

Data Collection



- Since the contact coaxial measurements were collected from single points of the specimens, measurements need to be collected from different locations of a specimen in order to obtain a representative average value.
- Sixty measurements were collected in total from six main regions of each specimen.
- In this research work, the average value of sixty measurements was in data analysis and interpretation.

Measurements on Cement Paste

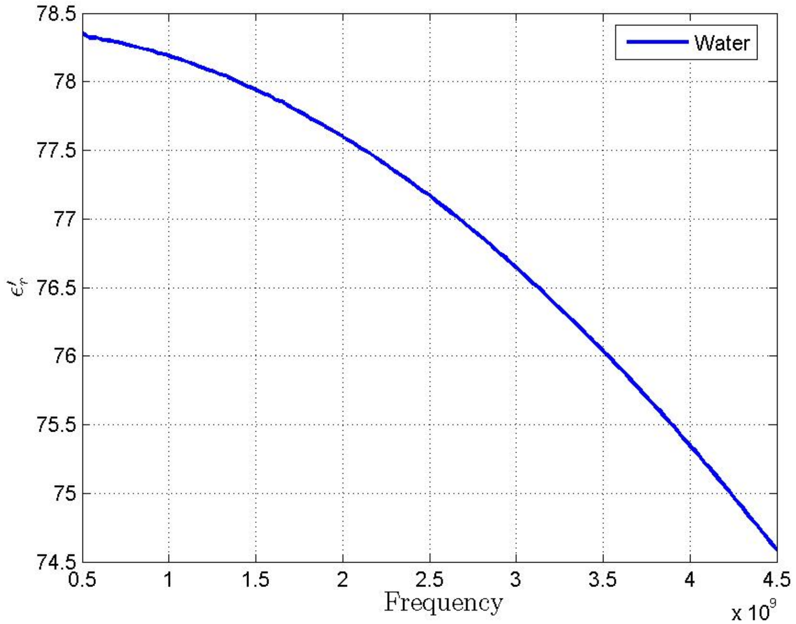


- The dielectric constant of cement paste depends on the dielectric constants of three major contents in its structure which are hydrated cement, air and evaporable water.
- The total volume of the pores inside a cement paste specimen is related to the w/c ratio.
- The proportion of water and air within the pores is related to the relative humidity of the environment.
- Measurement Conditions:

  Temperature: 23 °C ± 2 °C

  Relative Humidity: 25 % - 30 % Relative Humidity

Measurements on Cement Paste

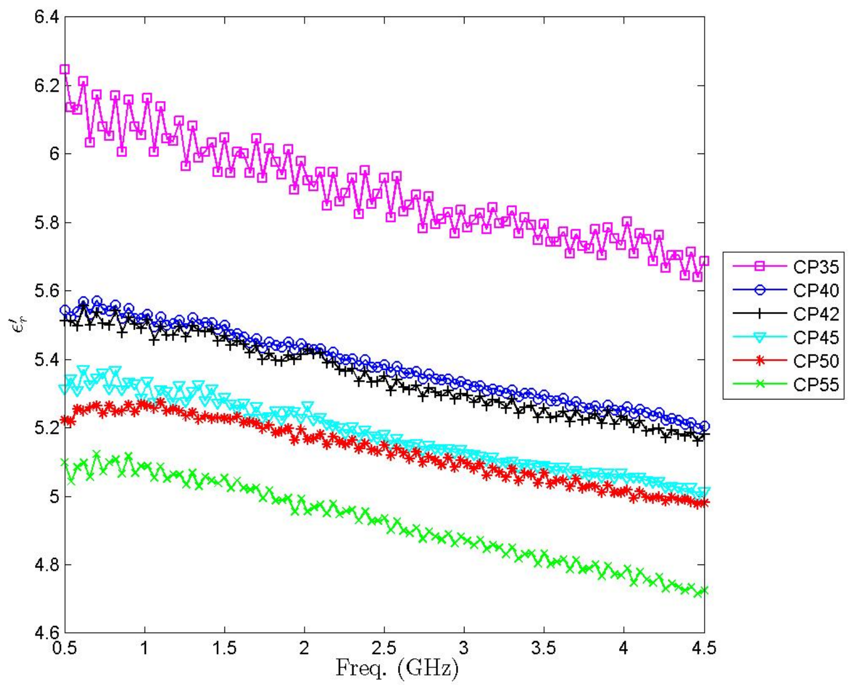


- The influence of water on the dielectric constant of cement paste is very important due to its high dielectric constant compared to other components.

- Variations in the quantity of water leads to high variations on the dielectric constant of cement paste specimens.

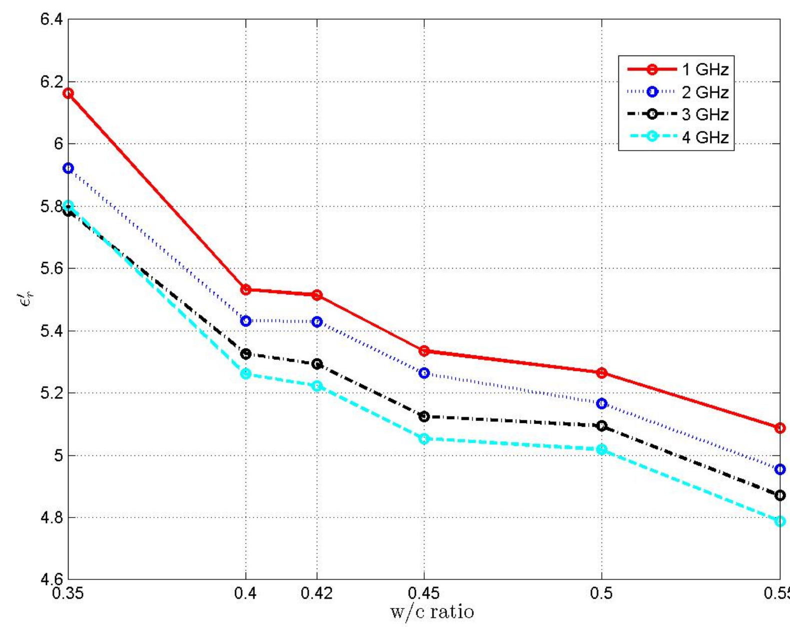
- Dielectric Constant of Air → 1
- Dielectric Constant of Hydrated Cement → 3 – 5

Measurements on Cement Paste

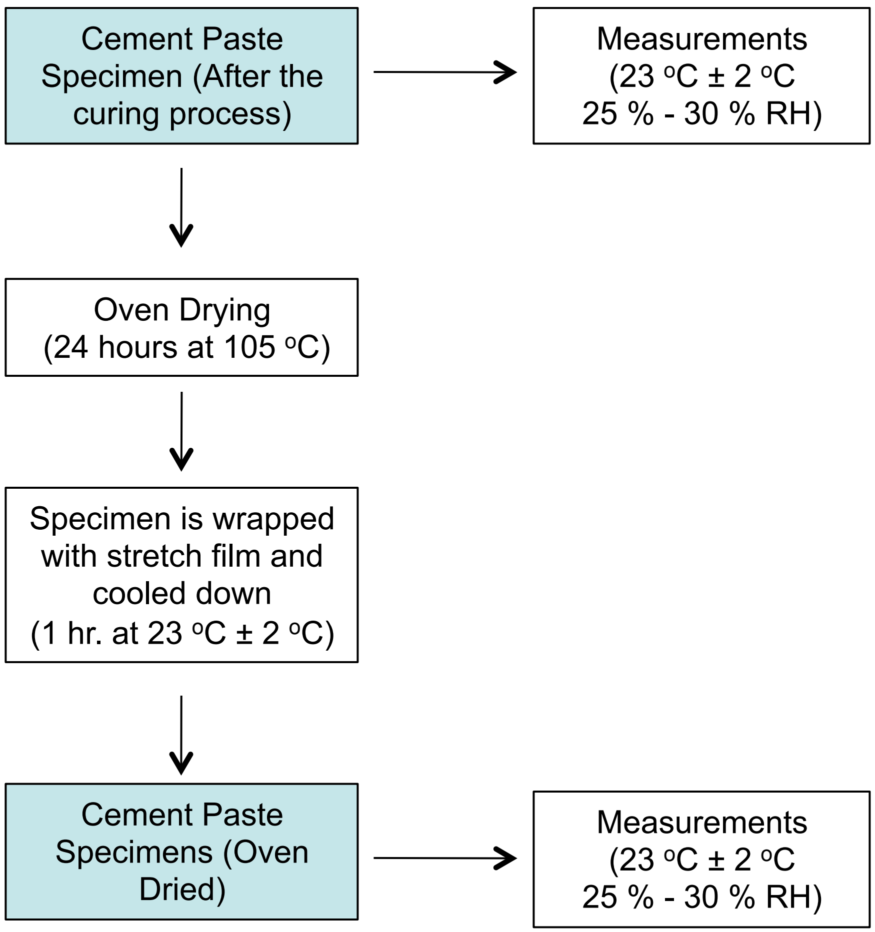


➢ Dielectric constant decreases at higher measurement frequencies.

➢ Dielectric constant decreases as the w/c ratio increases.



Measurements on Cement Paste



- Cement paste specimen was kept in an oven at 105 ^{o}C for twenty four hours and the evaporable water was removed. After oven drying it was covered with stretch film to prevent the cement paste specimen from absorbing the water in the air until its temperature decreases to 23 ^{o}C ± 2 ^{o}C. When the temperature drops down to room temperature, the measurements were collected.

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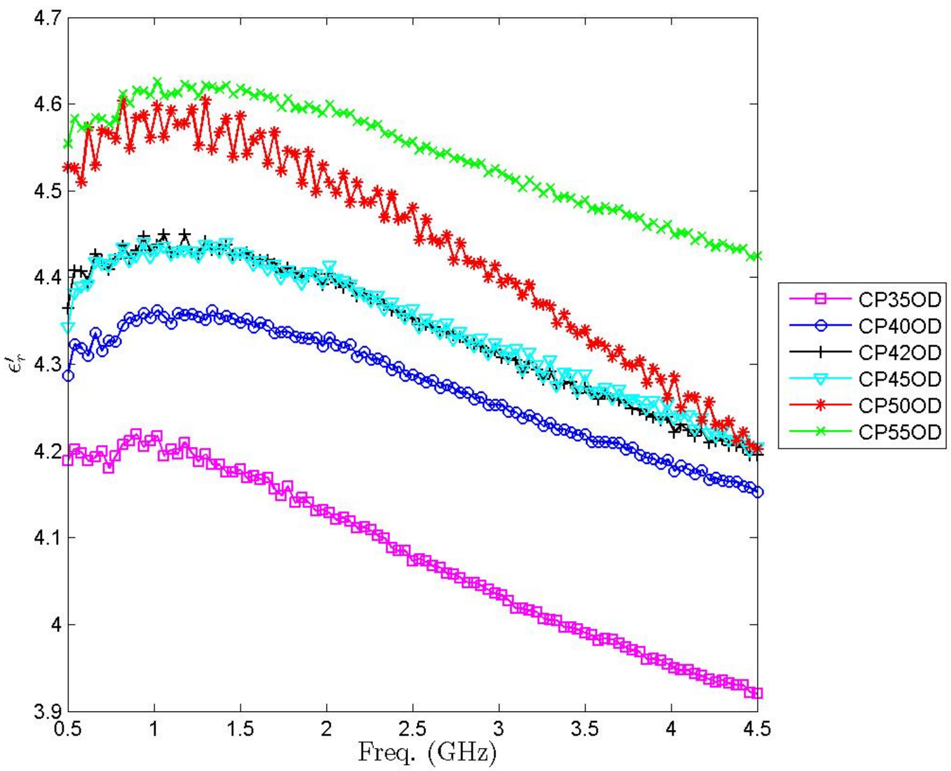
Measurements on Cement Paste



- After cement paste specimens were oven-dried, dielectric constant increased as the w/c ratio increased.

- Dielectric constant of oven dried cement paste specimens decrease as the measurement frequency increases.

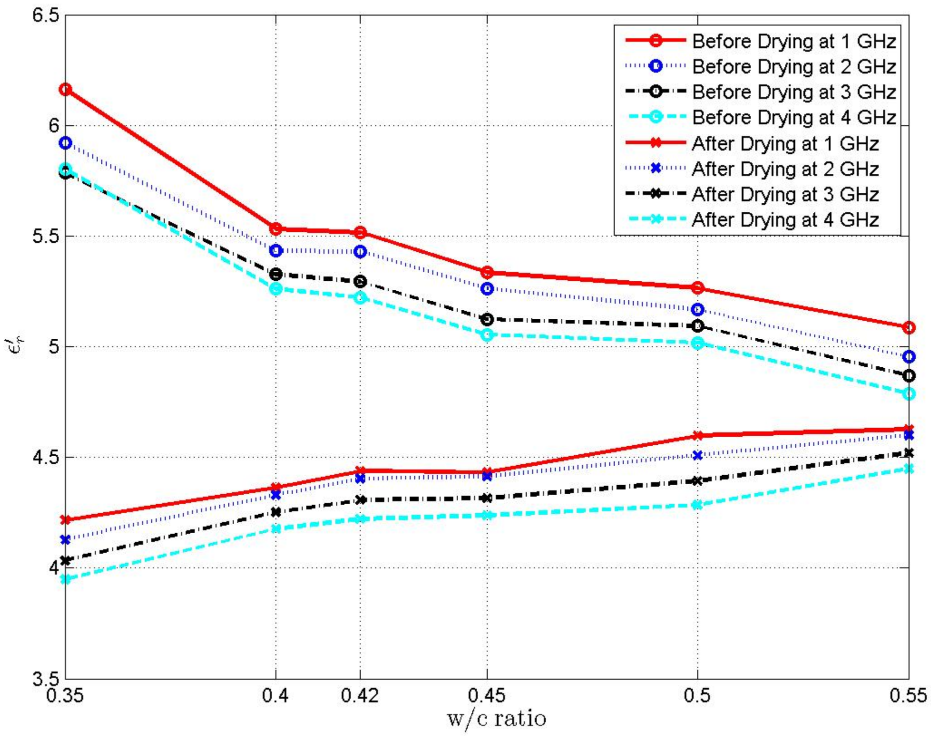
Measurements on Cement Paste

- After the oven drying procedure the water loss was not the same for all cement paste specimens. It was found that, as the w/c ratio increases, the water loss increases.

- Due to high water loss, high reduction of dielectric constant is expected for cement paste specimens with higher w/c ratios.

Sample	Weight Before OD	Weight After OD	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

Measurements on Cement Paste

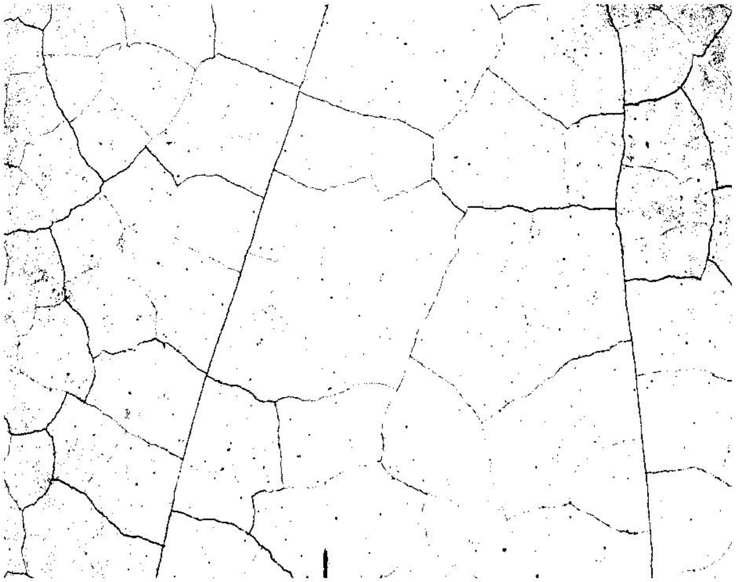


- Reduction of dielectric constant was lower for specimens with higher w/c ratios, although the water loss was higher in these specimens.

- Micro cracks formed on the surface of cement paste specimens when the specimens were oven dried.

Measurements on Cement Paste

CP55OD



With image processing, the background was removed and the cracks were emphasized.

Each photo represents an area approximately 2.5 in.-by-3 in..

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Measurements on Cement Paste

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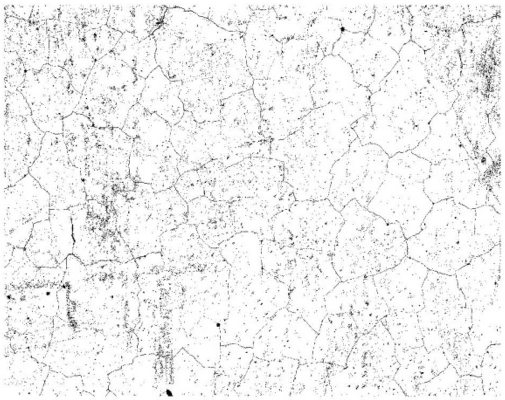
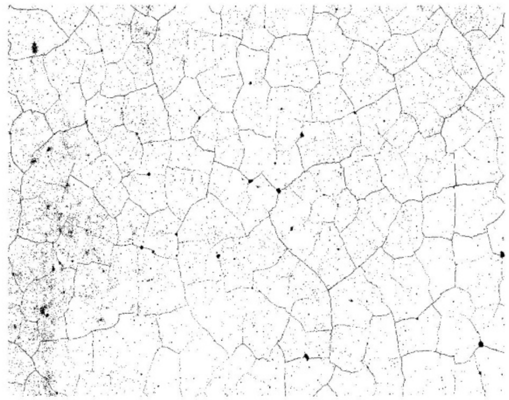
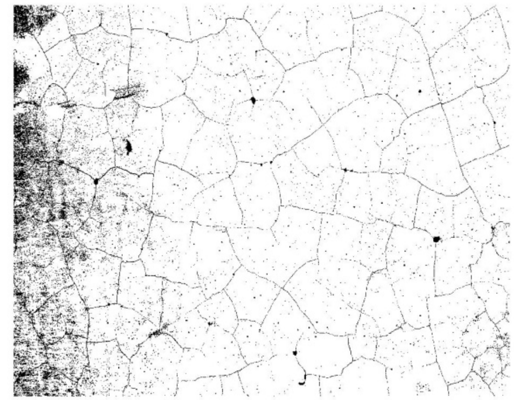
CP35OD

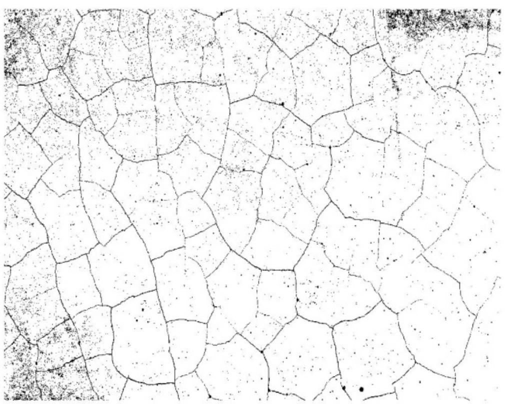


CP40OD

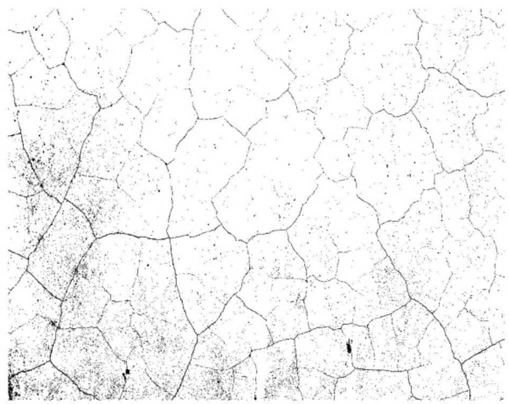


CP42OD

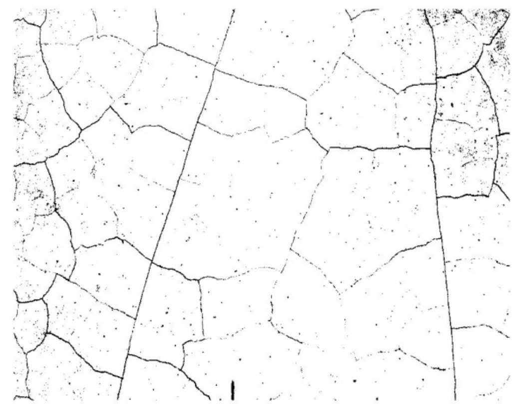




CP45OD



CP50OD



CP55OD

Measurements on Cement Paste

Measured Dielectric Constant ← Cement Paste specimen (Before Oven Drying)

↓ By applying Yu's Model for dried cement paste

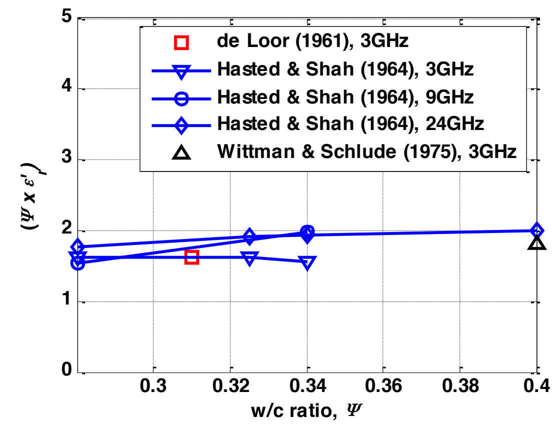
Estimated Dielectric Constant after OD (Without Cracks)

↓ By applying Maxwell's Dielectric Mixture Model

Measured Dielectric Constant ← Cement Paste specimens (Oven Dried)

- **Yu's Model (2009)**;

Product of the w/c ratio and the dielectric constant is approximately constant when there is no moisture present in the specimen.



- **Maxwell's Dielectric Mixture Model (1891)**;

$$\epsilon = \epsilon_h + \frac{3 v_i \epsilon_h}{\frac{\epsilon_i + 2\epsilon_h}{\epsilon_i - \epsilon_h} - v_i}$$

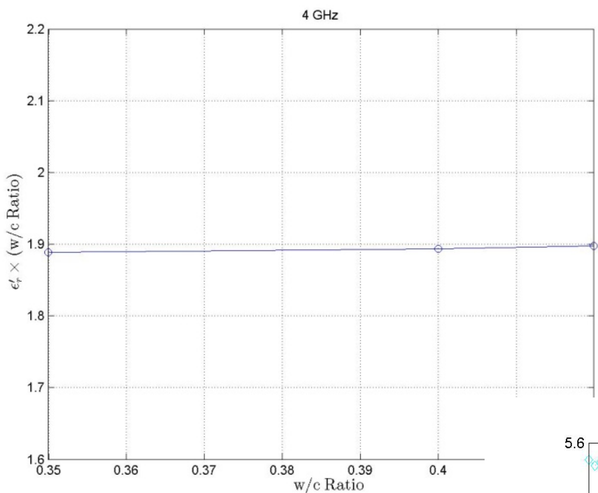
ε = the effective complex permittivity of the mixture

ε_h = the complex permittivity of the host dielectric

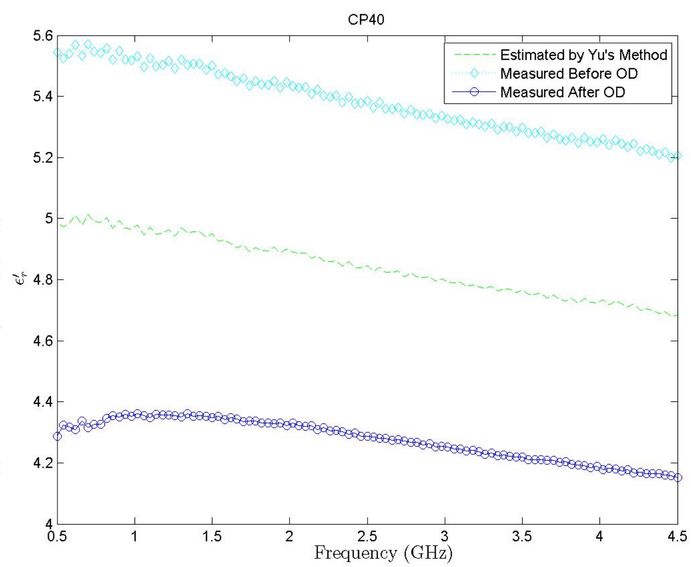
ε_i = the complex permittivity of the inclusion dielectric

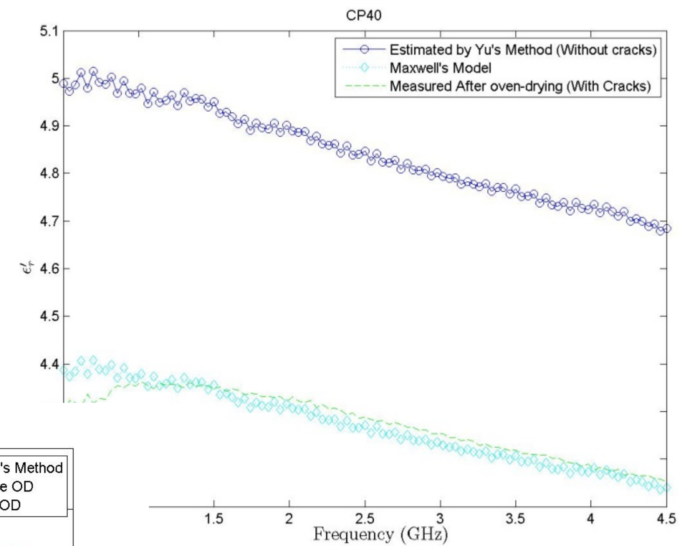
v_i = the volumetric fraction of the inclusion dielectric

Measurements on Cement Paste



Cement Paste	
Sample	Reduction Factor
CP35	0.93
CP40	0.90
CP42	0.87





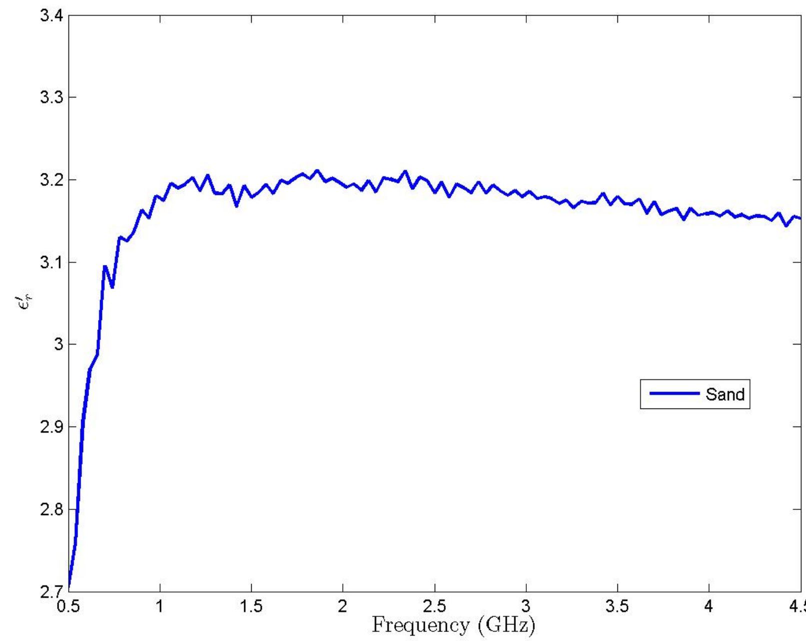
Cement Paste	
Sample	Volume Fraction
CP35	0.14
CP40	0.07
CP42	0.04

Measurements on Cement Paste

- Dielectric constant of cement paste decreases as the w/c ratio increases and measurement frequency increases.
- Dielectric constant decreases when the evaporable water is removed by the oven drying procedure. The reduction rate of dielectric constant is higher for cement paste specimens with lower w/c ratios due to the formation of micro cracks on the surface of oven-dried cement paste specimens.
- By applying Yu's model for oven dried cement paste specimens and Maxwell's dielectric mixture model the volume of the surface cracks on the surface is calculated. The volume of surface cracks is higher for the cement paste specimens with lower w/c ratios.

Measurements on Cement Mortar

Pores	Air
	Water
Solid Part	Sand
	Hydrated Cement



- Sand-to-cement (s/c) ratio was 2.53 (by weight).
- The dielectric constant of sand is lower than the dielectric constant of cement paste. A reduction in the dielectric constant is expected when sand is introduced to cement paste.

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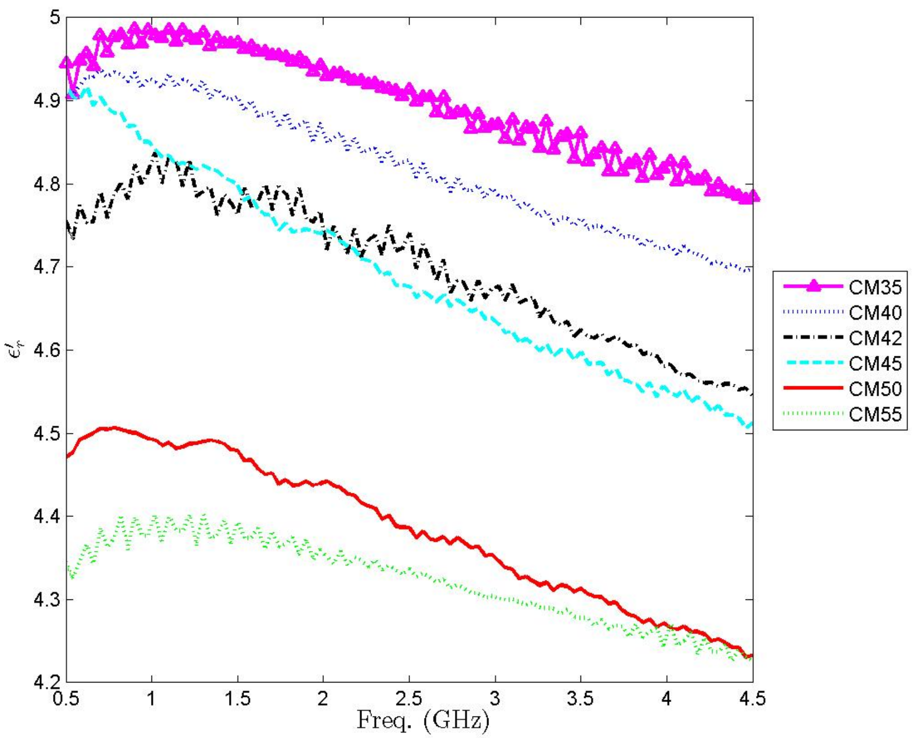
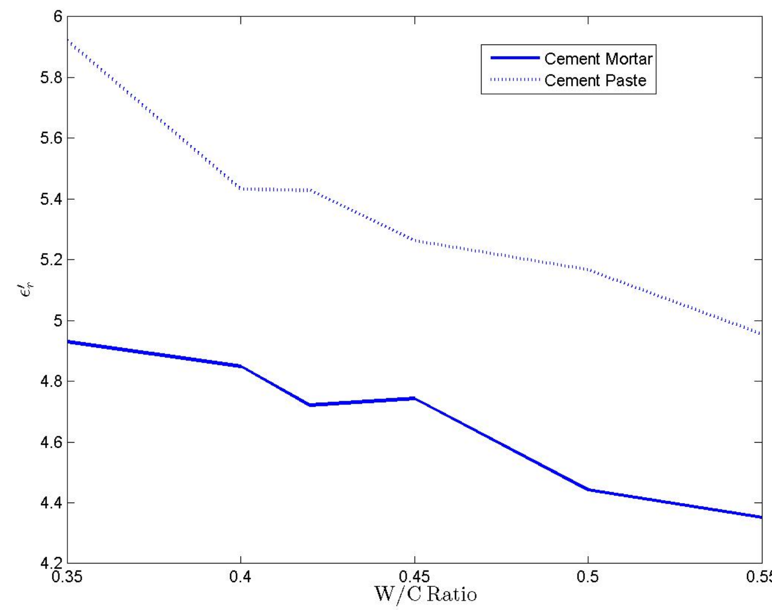
Measurements on Cement Mortar



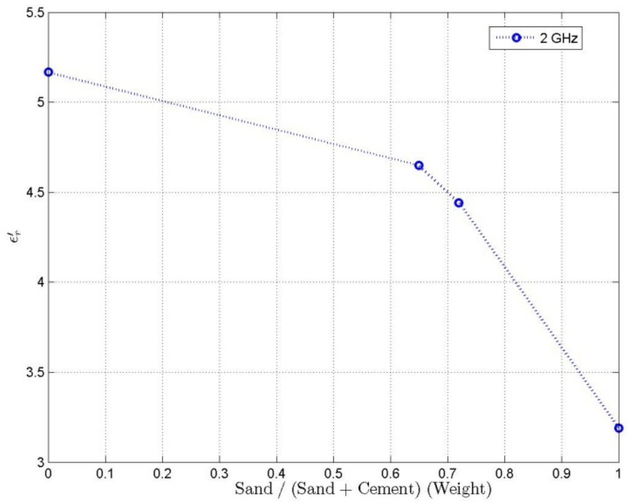
- Dielectric constant decreases at higher measurement frequencies.

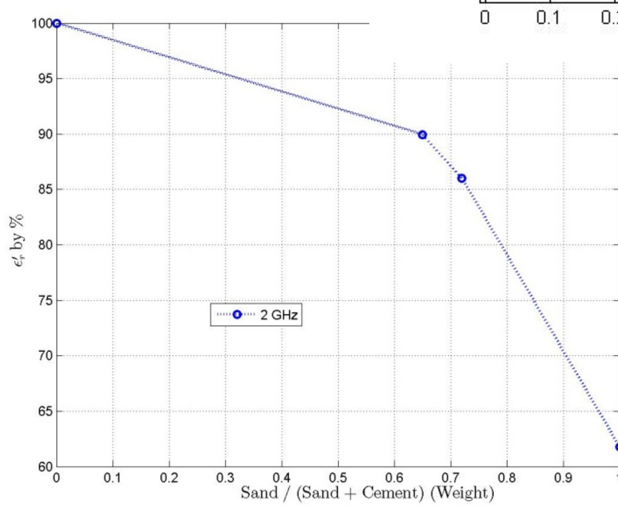
- Dielectric constant decreases as the w/c ratio increases.
- Dielectric constant of cement paste is higher than the dielectric constant of cement mortar with the same w/c ratio.

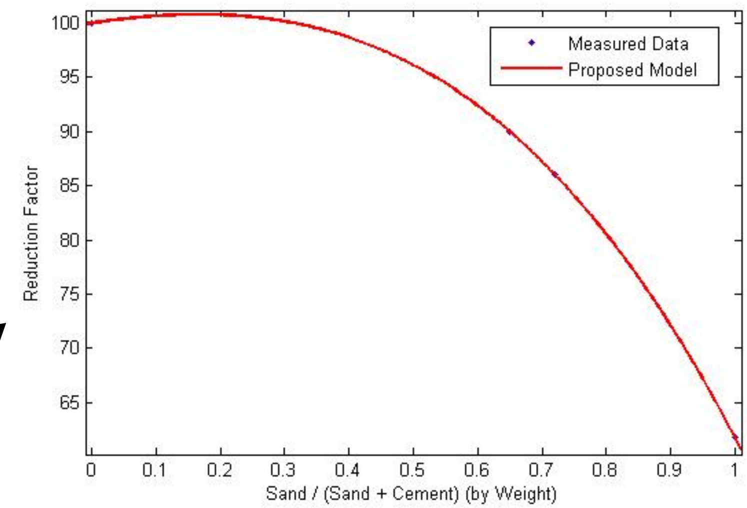


Measurements on Cement Mortar

w/c = 0.50







Measurements on Cement Mortar

Dielectric Constant of Cement Paste (w/c = a)

↓ By applying proposed Power Equation Model

Dielectric Constant of Cement Mortar (s/c = x) (w/c = a)

- Proposed Power Equation Model for the reduction factor;

  $y(x) = ax^3 + bx^2 + cx + d$,

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

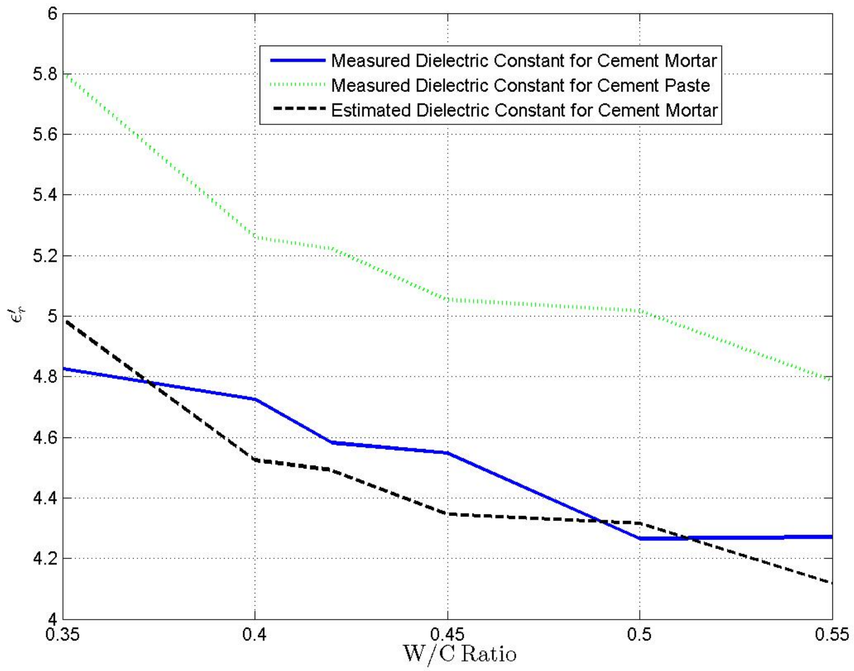
  y = reduction factor, $x = s / (s+c)$

  correlation coefficient (R) ≈ 0.99

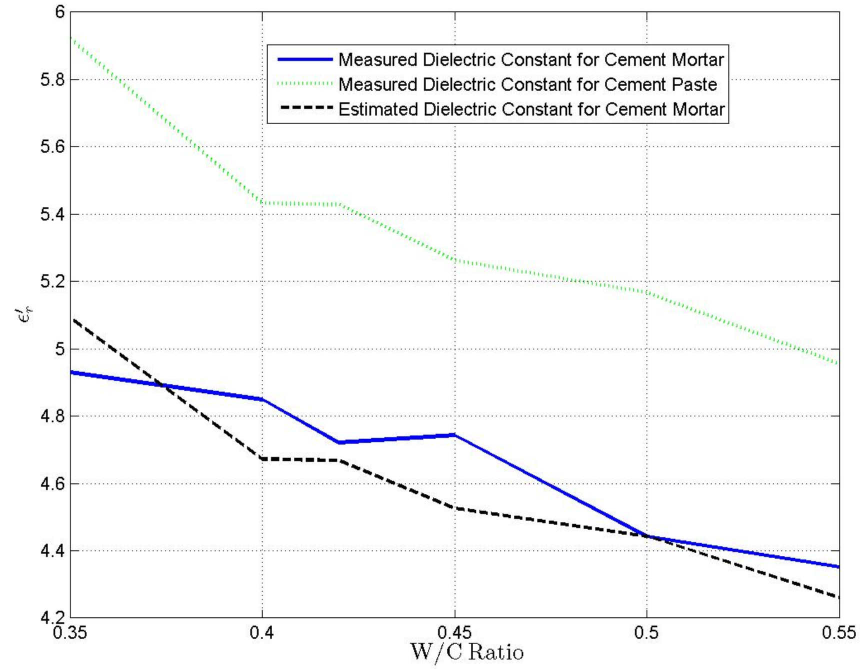
- With the proposed model, dielectric constant of cement mortar can be obtained for any s/c ratio in the range of 1.9 to 2.53, by using the dielectric constant of cement paste with a given w/c ratio.
- The model is applicable in the frequency range of 0.5 GHz to 4.5 GHz, and in the w/c ratio range of 0.35 to 0.55.

Measurements on Cement Mortar

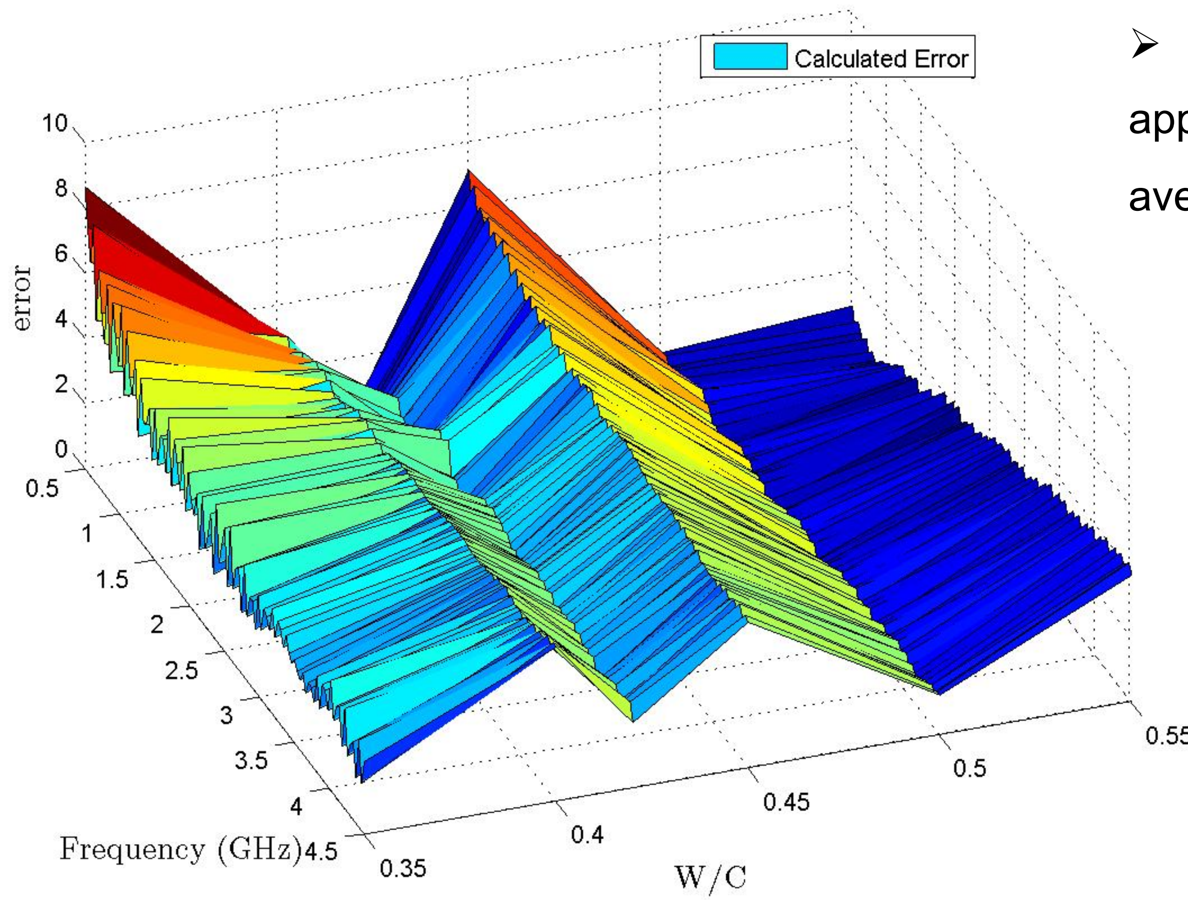
2 GHz



4 GHz



Measurements on Cement Mortar



- The highest error is approximately 8 %, and the average error is approximately 4 %

Measurements on Cement Mortar

- Dielectric constant of cement mortar decreases as the w/c ratio increases and measurement frequency increases.
- Dielectric constant of cement mortar is less than the dielectric constant of cement paste, due to low dielectric constant of sand.
- As the s/c ratio of cement mortar increases, the dielectric constant of cement mortar decreases. By using the proposed model, dielectric constant of cement mortar can be estimated when the dielectric constant of cement paste, and it's w/c ratio are provided.

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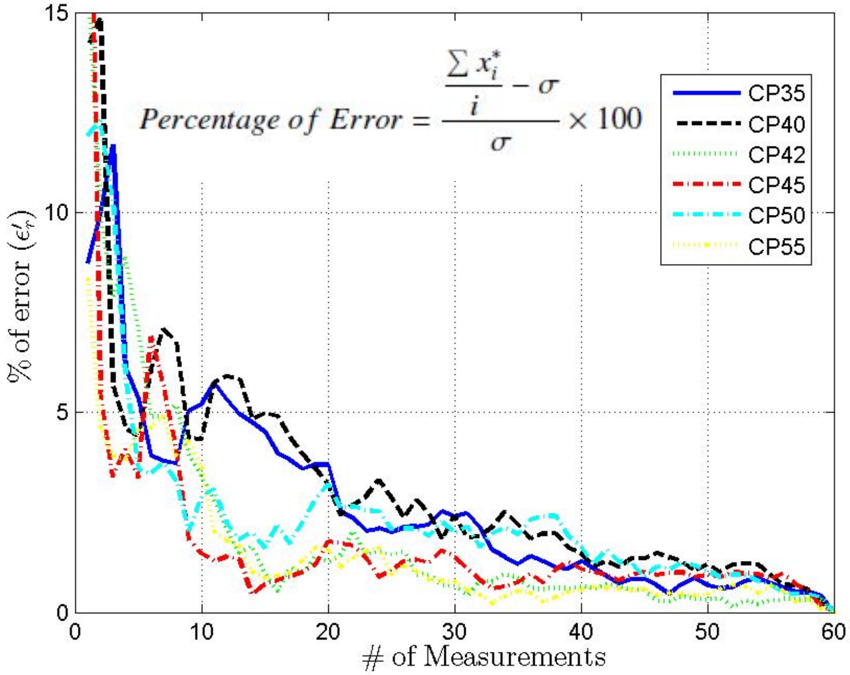
Reliability of the Measurements

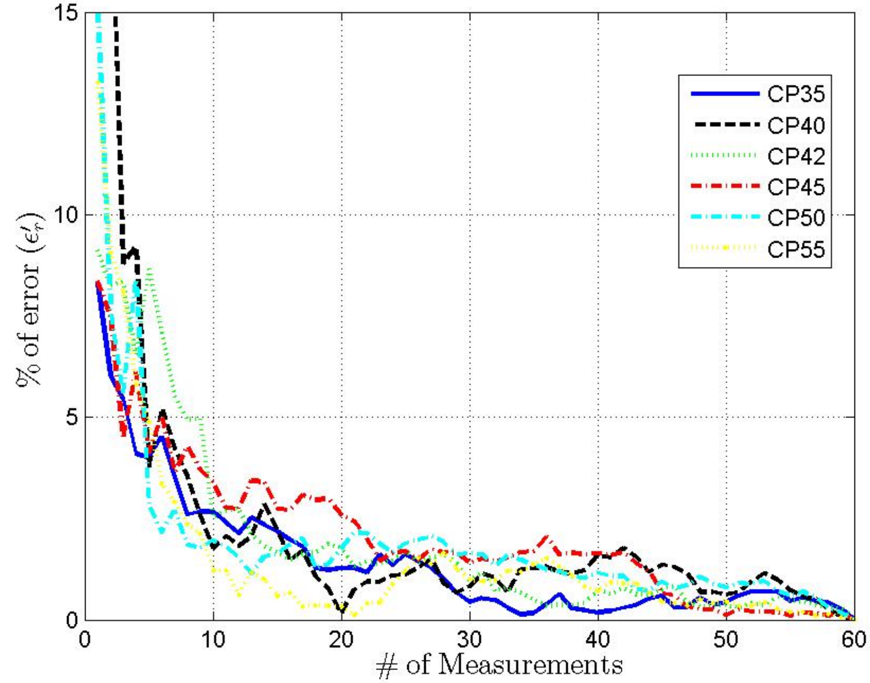
- Cement paste has a heterogeneous structure that consists of hydrated cement, and voids which are partially filled with water and air.
- Individual point readings on the surface of a cement paste specimen do not represent the overall property therefore, multiple points are required to obtain a reliable average value.

Reliability of the Measurements

1 GHz



4 GHz



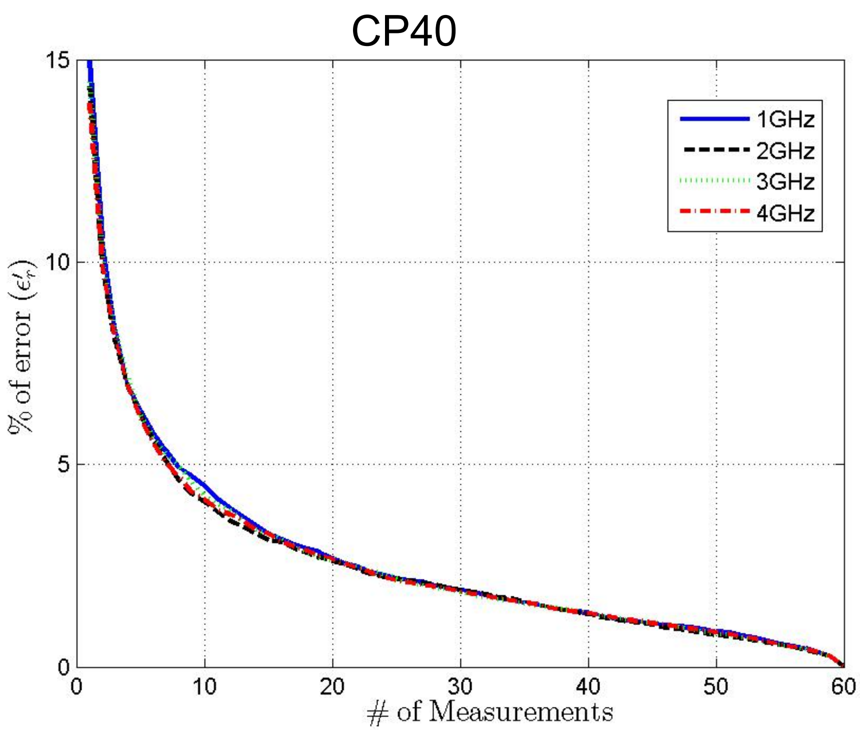
- As the number of collected measurements increases, a reliable /representative average value is obtained that.
- Statistically, we have calculated that the average of sixty measurements has an error less than 3 % at a 95 % confidence level.

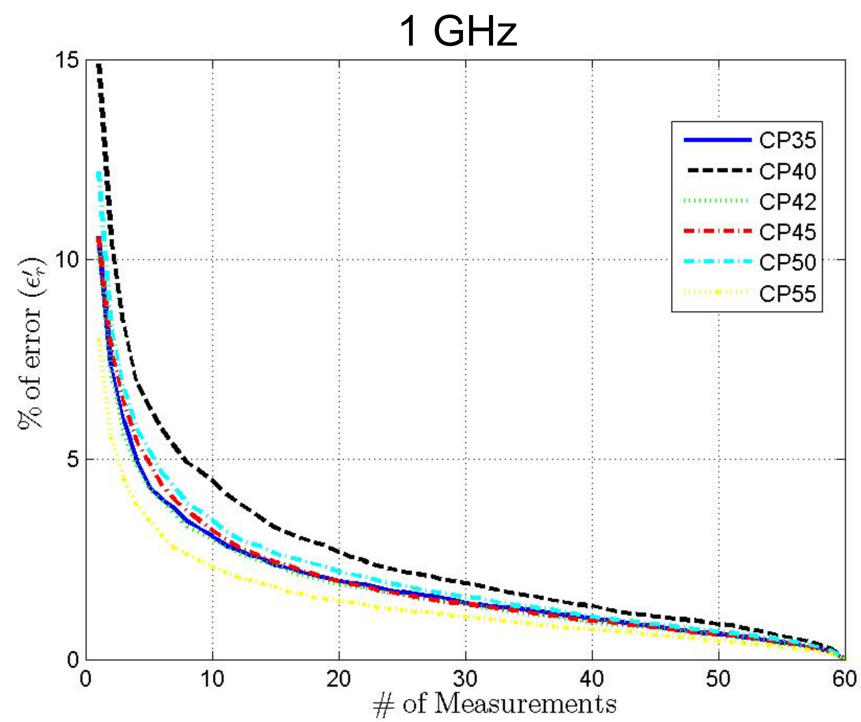
Monte Carlo Simulations

- If someone needs to test the mean of a distribution, the type of the distribution is needed.
- In cases where the properties of the distribution are very difficult to obtain analytically or not known, Monte Carlo methods may be used.
- Monte Carlo methods are a class of computational algorithms that rely on repeated use of random sampling to simulate systems with many degrees of freedom.
- Steps followed for Monte Carlo Simulations;

- Definition of Inputs; $y = f(x_1, x_2, \ldots, x_{60})$
- Generation of Inputs Randomly $y_i = (x_{i1}, x_{i2}, \ldots, x_{i60})$
- Generation of Random Sets $i = 1$ to 1000
- Generation of a Single Set $x_i = \sum x_{i1} / 1000$, $I = 1$ to 1000
- Analysis of the Results

Reliability of the Measurements





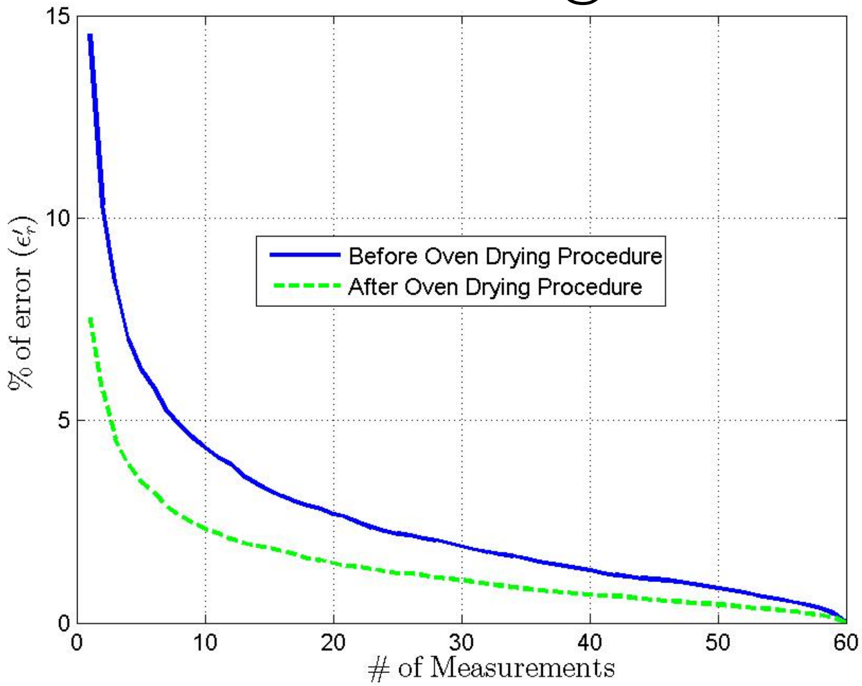
- A smooth curve was obtained by running 1000 simulations and calculating their average.
- It was observed that the measurement frequency and the w/c ratio have a negligible effect on the percentage of error.

SERG
Microwave Material Characterization Lab

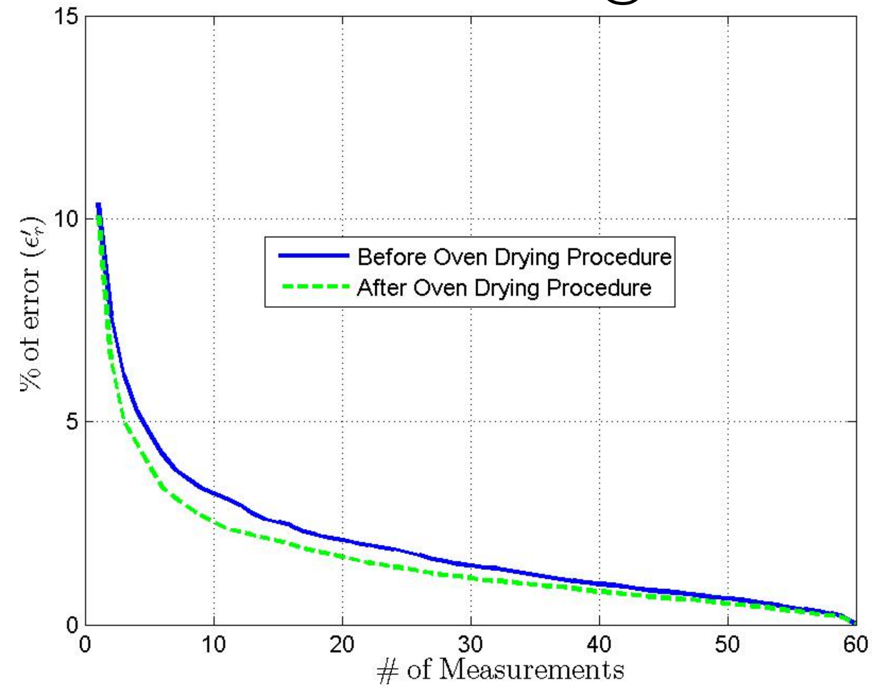
Reliability of the Measurements

UMASS LOWELL

CP40 and CP40OD @ 1 GHz



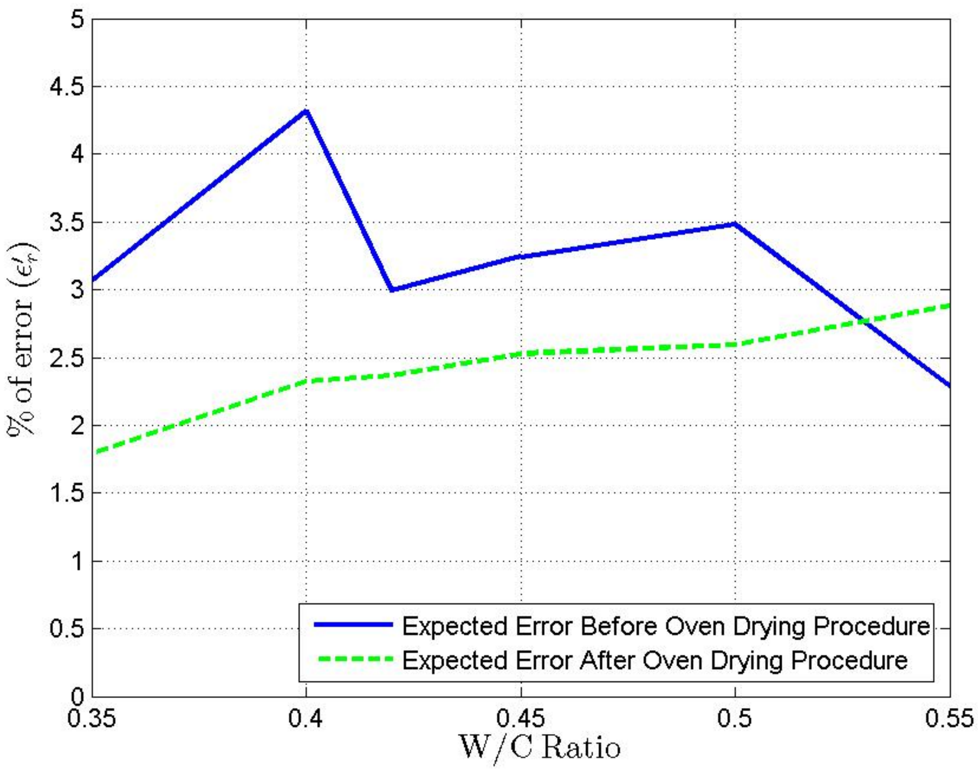
CP45 and CP45OD @ 1 GHz



When we considered the measurements after the oven drying procedure, we saw a decreasing percentage of error. This is due to the removal of water which has a high dielectric constant compared to other ingredients. When the water is removed, a relatively homogenous dielectric constant distribution is observed.

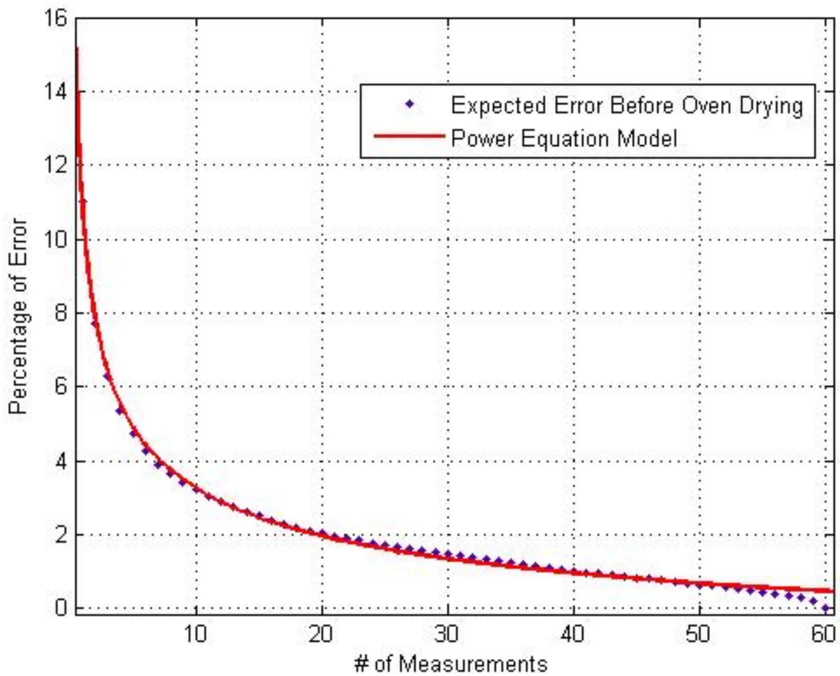
Reliability of the Measurements

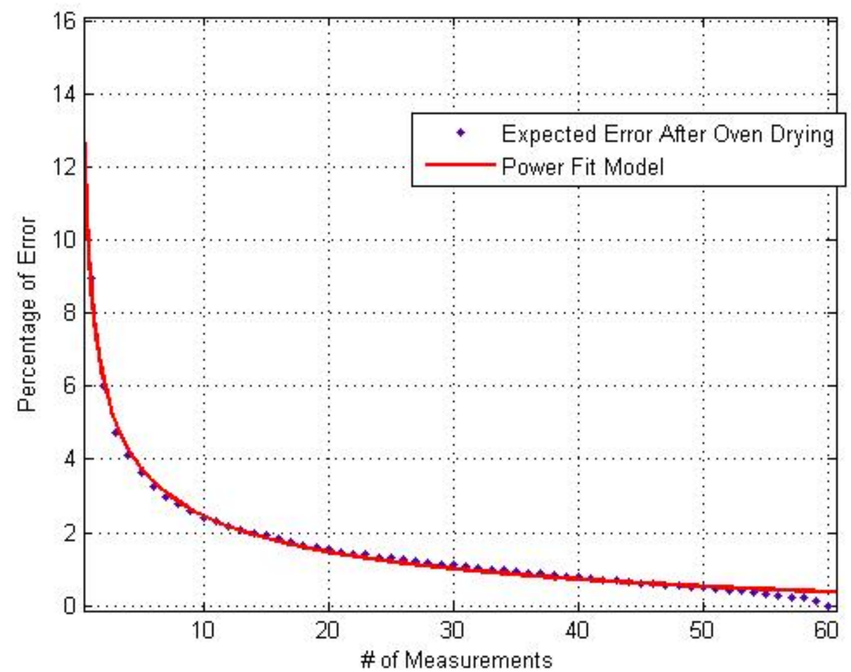
Expected percentage of error when ten measurements are randomly collected from each specimen



After the removal of evaporable water, it was observed that the percentage of error is less for cement paste specimens with a lower w/c ratio, suggesting that low w/c ratio cement paste specimens have a relatively homogenous composition in terms of dielectric constant.

Reliability of the Measurements





$$y(x) = a\,x^{b} + c$$

a = 13.420, b = -0.359, c = -2.621

a = 10.260, b = -0.410, c = -1.536

y = percentage of error, x = number of measurements

Conclusions and Contributions

- Dielectric constant of cement paste and cement mortar both decreases as the w/c ratio increases and measurement frequency increases.
- When the evaporable water is removed from cement paste the dielectric constant decreases. Due to the formation of surface micro cracks on cement paste specimens after oven drying, the reduction rate of dielectric constant is higher for low w/c ratio specimens.
- As the s/c ratio increases, dielectric constant of cement mortar decreases. A model for determining the reduction rate is proposed.
- Error analysis for contact coaxial measurement method was performed. The expected error is related to the number of measured points and modeled for cement paste specimens before and after the oven drying procedure.
- Dielectric heterogeneity of cement paste specimens is observed. Heterogeneity decreases when the evaporable water is removed from cement paste specimens. After the removal of evaporable water, we observed that low w/c ratio specimens become relatively homogenous.

Future Work

- Contact Coaxial Measurements on concrete with introduced cracks, reinforcing bars, corroded reinforcing bars, different types of aggregates. Determination of the effect of rust penetration to concrete on the dielectric constant of concrete.
- Building a free space method system, to compare the data collected with free space method with the data obtained by the contact coaxial probe. The relation between local measurements can be compared with global measurements this way.
- Collecting measurements from structures in use or demolished structures for comparisons with the data collected in laboratory.

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