

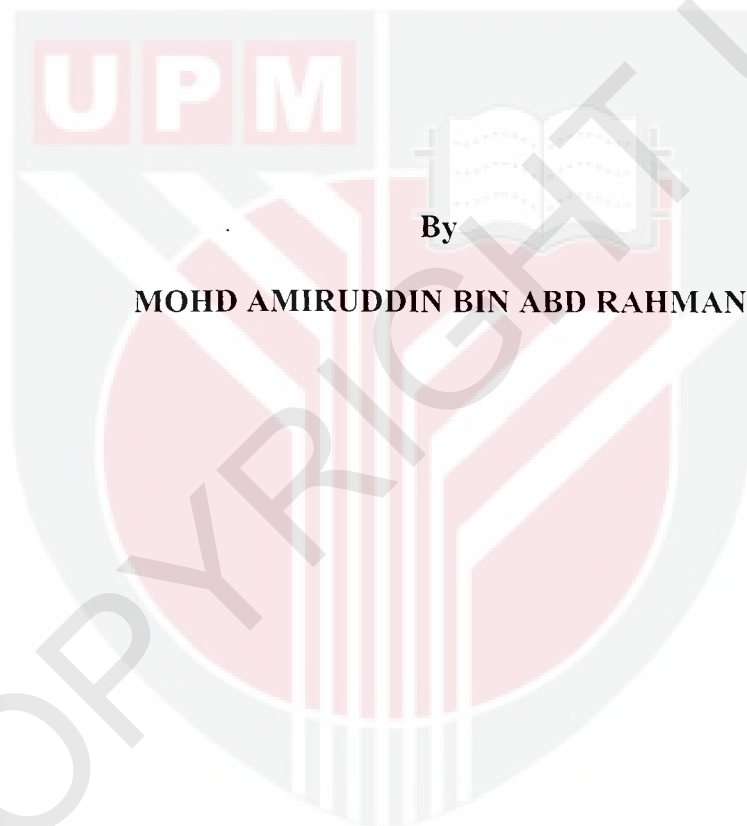


***DETECTION OF IONIC CONDUCTIVITY AND MOISTURE CONTENT OF
VARIOUS LIQUIDS USING MICROWAVE TECHNIQUE***

MOHD AMIRUDDIN BIN ABD RAHMAN

FS 2011 99

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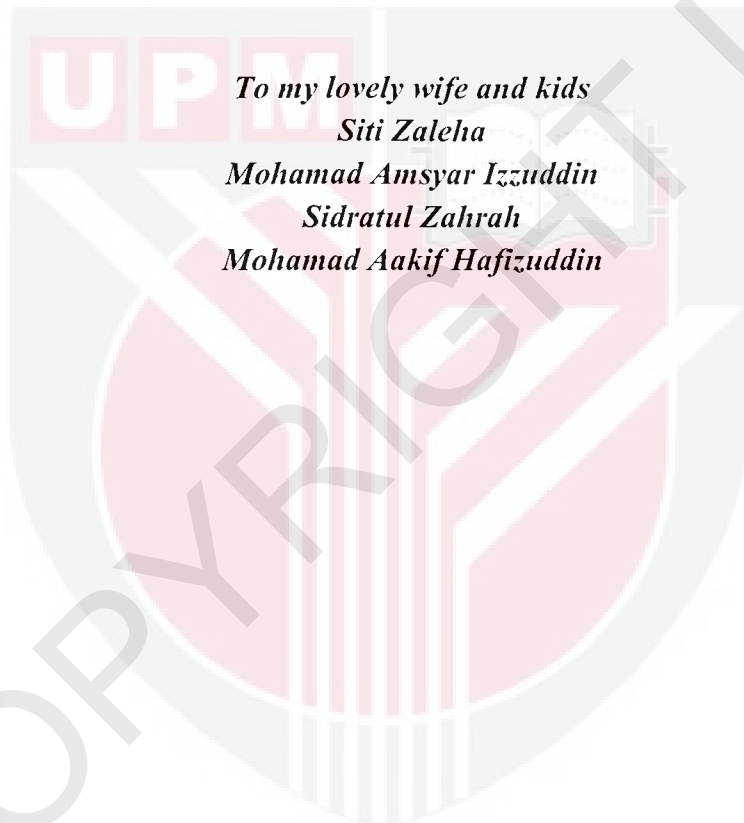


By

MOHD AMIRUDDIN BIN ABD RAHMAN

© Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfilment of the Requirement for the Degree of Master of Science

April 2011



To my lovely wife and kids
Siti Zaleha
Mohamad Amsyar Izzuddin
Sidratul Zahrah
Mohamad Aakif Hafizuddin

Abstract of thesis submitted to the Senate of Universiti Putra Malaysia in fulfilment
of the requirement for the degree of Master of Science

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April 2011

Chairman: Professor Kaida Khalid, PhD

Faculty: Science

Measurement of ionic conductivity and moisture content of liquid is important because such information give indication about quality and also composition of elements of liquid. Previously microwave sensors are mainly used to determine moisture content in liquid. However, the microwave sensor may also be used to measure the conductivity at lower microwave frequency range. At present, detection of conductivity is done at low frequency and no previous efforts using microwave technique is considered because of the cost. However, a microwave system which could do simultaneous detection could overcome that problem.

A quasi-transverse electromagnetic technique employing variational method is used to simulate the behaviour of attenuated wave of the sensing element of the microwave microstrip sensor. Three different operating frequencies at 300 MHz, 2 GHz and 5 GHz is chosen particularly for detection of the attenuation of the sensor

where the first two is for conductivity detection and the last is for moisture content detection. The calculated results demonstrate that the attenuation is a function of permittivity of liquid. Also, permittivity is related to the conductivity and moisture content of liquid at low and high frequency respectively. A computer program using Fortran programming language is used for the calculation.

The U-shaped microwave microstrip system is based on the transmission coefficient of the microstrip sensors. The system utilises two oscillators that act as the microwave source which operates at above mentioned frequencies. Two crystal detectors are placed in the system to measure input and output voltage so that total attenuation of the system can be determined. Conductivity and moisture content standard measurement are done using a commercial conductivity meter and oven drying method respectively.

Comparison of the insertion loss of the microstrip sensor system from calculation with attenuation of microstrip sensor alone shows excellent correlation of 0.9945. Established empirical relationship between insertion loss and conductivity at 300 MHz and 2 GHz gives correlation coefficient of 0.6334 and 0.9431 respectively. Another empirical relationship of insertion loss and moisture content at 5 GHz shows good fit at 0.9688. Evaluation of the measured conductivity and moisture content using developed system with standard measurement gives correlation coefficient of 0.3282 (300 MHz), 0.9435 (2 GHz), and 0.9781 (5 GHz). The main limitation of the developed system is in terms of measurement of conductivity of fresh latex samples

at 300MHz since the variation of conductivity is less than 0.2 S/m. The moisture content measurement shows very good sensitivity at 1% change of moisture content.

The results present that the developed microwave system is possible to be used for simultaneous measurement of conductivity and moisture content of liquids. Further enhancement may be made to improve the resolution of conductivity measurement at low frequency. The system could be made portable which reliable for in-situ field-measurement.



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PENGESANAN SERENTAK KEBERALIRAN ION DAN KANDUNGAN LEMBAPAN PELBAGAI CECAIR MENGGUNAKAN TEKNIK GELOMBANG MIKRO

Oleh

MOHD AMIRUDDIN BIN ABD RAHMAN

April 2011

Pengerusi: Profesor Kaida Khalid, PhD

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Pengukuran keberaliran ion dan kandungan lembapan penting kerana maklumat berkenaan memberikan petunjuk kepada kualiti dan kandungan unsur dalam cecair. Sebelum ini sensor gelombang mikro hanya digunakan kebanyakannya untuk menentukan kadar lembapan dalam cecair. Namun, sensor gelombang mikro boleh juga digunakan untuk mengukur keberaliran pada julat frekuensi rendah. Pada masa sekarang, keberaliran biasanya diukur pada julat frekuensi rendah gelombang mikro dan tiada usaha pada masa lalu menggunakan gelombang mikro untuk mengesan kekondusian ion disebabkan oleh kos. Walaubagaimanapun, satu sistem gelombang mikro yang boleh melakukan pengesanan serentak mampu mengatasi masalah tersebut.

Satu teknik kuasi-elektromagnet lintang menggunakan kaedah bervariasi digunakan untuk menentukan pengecilan gelombang pada sensor mikrostrip. Tiga frekuensi pengendalian berbeza pada 300 MHz, 2 GHz, dan 5 GHz dipilih terutamanya untuk pengesanan pengecilan pada sensor dengan dua frekuensi pertama ialah untuk pengesanan keberaliran dan yang terakhir untuk pengesanan kandungan lembapan. Hasil pengiraan menunjukkan pengecilan gelombang berdasarkan kepada ketelusan yang berkait dengan keberaliran cecair pada frekuensi rendah dan kadar lembapan cecair pada frekuensi tinggi. Satu program komputer menggunakan bahasa pengaturcaraan Fortran digunakan untuk pengiraan.

Sistem sensor mikrostrip berbentuk U adalah berdasarkan kepada pekali penghantaran sensor. Sistem berkenaan menggunakan dua pengayun yang berfungsi sebagai sumber gelombang mikro beroperasi pada frekuensi yang dinyatakan di atas. Dua pengesan hablur diletakkan pada sistem untuk mengukur voltan input dan output yang membolehkan jumlah pengecilan sistem boleh ditentukan. Pengukuran keberaliran dan kandungan lembapan piawaian masing-masing dilakukan menggunakan meter keberaliran dan kaedah pengeringan ketuhar.

Perbandingan kehilangan sisipan sistem sensor mikrostrip dari pengiraan dengan nilai pengukuran pengecilan sensor tersebut menunjukkan korelasi yang sangat baik iaitu pada 0.9945. Hubungan emperik yang diuji di antara kehilangan sisipan dan keberaliran pada 300 MHz dan 2 GHz masing-masing memberikan pekali korelasi 0.6334 dan 0.9431. Seterusnya hubungan emperik di antara kehilangan sisipan dan

kadar lembapan pada 5 GHz menunjukkan padanan yang baik pada 0.9688. Penilaian di antara keberaliran dan kadar lembapan yang diukur menggunakan sistem yang dibangunkan dengan pengukuran piawaian memberikan pekali korelasi 0.3282 (300 MHz), 0.9435 (2 GHz), dan 0.9781 (5 GHz). Kekangan utama menggunakan sistem yang dibangunkan ialah dari segi pengukuran keberaliran sampel susu getah segar pada frekuensi 300 MHz kerana perubahan keberaliran kurang daripada 0.2 S/m. Pengukuran kandungan lembapan menunjukkan kepekaan yang sangat baik iaitu pada 1% perubahan kandungan lembapan.

Hasil pengukuran menunjukkan sistem gelombang mikro yang dibangunkan sesuai untuk digunakan untuk pengukuran serentak keberaliran dan kadar lembapan cecair. Pembaikan seterusnya adalah dengan memperbaiki kejituan pengukuran keberaliran pada frekuensi rendah. Dengan ini, suatu sistem mudah alih boleh dibangunkan untuk pengukuran di lapangan.

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LIST OF ABBREVIATIONS

TEM	Transverse Electromagnetic
PNA	Personal Network Analyzer
MIT	Massachusetts Institute of Technology
Q-factor	Quality Factor
RF	Radio Frequency
MRR	Microstrip Ring Resonator
VNA	Vector Network Analyzer
GPR	Ground Penetrating Radar
MC	Moisture Content
SMA	SubMiniature Version A
MUT	Material Under Test
ISO	International Organization of Standardization
ASTM	American Society for Testing and Materials
PNA	Personal Network Analyzer
PVC	Polyvinyl Chloride
VSWR	Voltage Signal Wave Ratio
DRO	Dielectric Resonator Oscillator

VCO

Voltage Controlled Oscillator

LCD

Liquid Crystal Display



CHAPTER 1

INTRODUCTION

1.1 Motivation of Project

Measurement of moisture content and electrical conductivity give information about water percentage and salt or ion content inside a material respectively. Quantifying water and salt content is a critical parameter in order to determine productivity, market value and quality indication of a product especially in agriculture industry. As example, most natural substances commercial values depending on their weight, hence the exact determination of moisture content will help to fix proper value of the product such as hevea rubber latex, milk, and grain. During transportation and storage of the product where a long term storage is free from microbial degradation. However, over drying grain can decrease its nutritional and reproductive values and contribute to the increased breakage during handling (Khalid, 2004). In terms of conductivity, it gives indication of water quality. In fruits production, conductivity is important tool to determine fruits maturity level (Guo et al., 2007). When crops are failing, conductivity relationship also may be studied to improve production.

Standard method to quantify conductivity and moisture content is using conductivity meter and oven drying method respectively. Currently, standard method to find conductivity is rapid. However, standard method to determine moisture content is time consuming. In addition to that, measurement of conductivity and moisture content simultaneously is impossible since the cost is high and the size of the

hardware is not suitable for remote sensing. Hence there is a need for a device which is portable and has the ability to perform simultaneous sensing of conductivity and moisture content of materials.

In microwave region, electromagnetic wave is sensitive to ionic conductivity at its lower frequency counterpart and supplementary very strongly interacts with water molecules as frequency gets higher. As a result, dielectric properties of moist materials should have a good relationship with ionic conductivity at lower microwave frequency and moisture content at higher microwave frequency. Therefore microwave measurement technique could offer an attractive means to overcome above issue.

This research project is focused on providing answer whether simultaneous in-situ measurement of ionic conductivity and moisture content could be realized by implementing microwave sensor system.

1.2 Background of Project

The basic principle of microwave moisture measurement is interaction of biological materials with electromagnetic fields. At microwave frequency, when an electromagnetic wave passes a materials the wave is altered by the materials properties. Such properties include density, temperature, concentration, composition

and microstructure of the material. The material properties obtained by this interaction is strictly described by two parameters namely, dielectric permittivity or also called dielectric properties, ϵ , and magnetic permeability, μ .

In this project, only non-magnetic liquid materials are tested which are saline water, seawater, concentrated latex and fresh latex. Indeed, most of the liquid substance on earth is also nonmagnetic. Thus microwave moisture measurement in this thesis will only be exploited by dielectric permittivity of materials.

Dielectric permittivity can be described in terms of dielectric constant and loss factor which is the real and imaginary part of complex permittivity respectively. Complex permittivity is generally expressed as following equation:

$$\epsilon^* = \epsilon' - j\epsilon'' \quad (1.1)$$

where ϵ^* is complex permittivity, ϵ' is dielectric constant, and ϵ'' is loss factor. Therefore, the term 'permittivity' and 'complex permittivity' are used interchangeably in this thesis.

At microwave frequency, only two physical mechanisms dominate materials dielectric behavior that is ionic conductivity and dipolar polarization. The terms mentioned here is further discussed in the next subsection.

Before that, the objective of this project was to design and develop a microwave microstrip sensor to characterize dielectric properties of liquid materials. Using the microstrip sensor configuration which was theoretically analyzed, a relationship of

materials permittivity with both moisture content and conductivity could be experimentally verified. Microstrip is mainly chosen because the cost to produce is cheaper than other microwave sensors and could provide accurate and reliable readings. A brief introduction of microstrip sensor used to characterize the dielectric samples is also discussed in section 1.3.

1.2.1 Polarization

Polarization is defined as a phenomenon when an electric charge is distorted due to the force from applied electric field causing the charge slightly moved from its equilibrium position. Polarization can be divided into two processes which are called relaxation and resonant. There are four main types of polarization which are electronic polarization, atomic polarization, dipolar or orientation polarization and ionic polarization where the first two belongs to resonant and the latter belongs to relaxation. At different frequencies, the polarization mechanism affects the permittivity of materials differently as shown in Figure 1.1. Since only last two mechanisms are crucial at microwave frequency, the electronic and atomic polarization effect could be neglected since it is crucial only at infrared and optical frequencies respectively and is not discussed further.

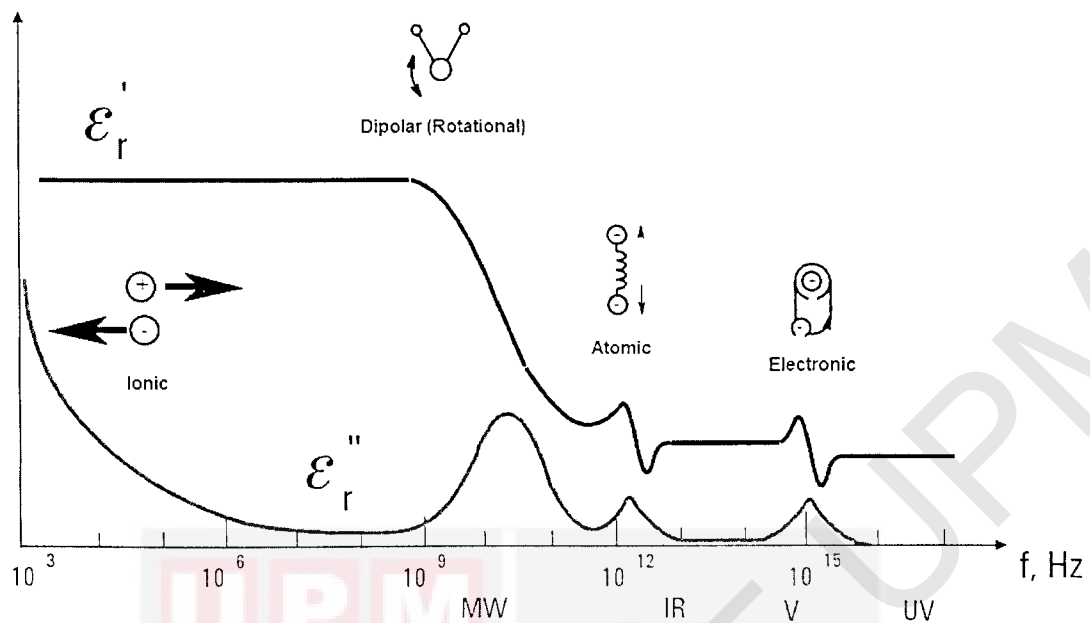


Figure 1.1. Polarization mechanisms at different frequency region. MW: Microwave, IR: Infrared, V: Visible, UV: Ultraviolet. (Source: "Basics of Measuring the Dielectric Properties of Materials," 2006)

Ionic polarization describes the ions conductivity in a medium. A material that has free charges suspended in non-conducting medium can be polarized when an electromagnetic field presents. The effect of ionic conductivity can only be seen in dielectric loss spectra. Only if heterogeneous materials which contains conducting region that is not in contact with each other presents, the influence of ion activity can be seen at the real part of permittivity, ϵ' (Hasted, 1973). The effect is called Maxwell-Wagner polarization. However, Maxwell-Wagner effect is negligible throughout this thesis since the moist materials used here are all homogeneous. Perhaps, most of macroscopic moist materials are homogeneous. In moist materials, ion conductivity dominates the low region of microwave frequency (usually less than 1GHz) (Kraszewski, 1996).

Dipolar polarization is the dominant polarization at microwave frequency. When an electromagnetic field is applied to the medium, the force from the field makes the dipole molecules experience a torque. Consequently, the molecules rotate in line with the applied field where charge in the molecule is unevenly distributed. This phenomenon causes the molecule having a permanent dipole moment. At microwave frequency this phenomenon occurs at higher frequency range (usually above 1GHz). The whole relaxation process from ionic to dipolar polarizations is called dielectric relaxation. An example of the entire polarization process at microwave frequency region is shown in Figure 1.2.

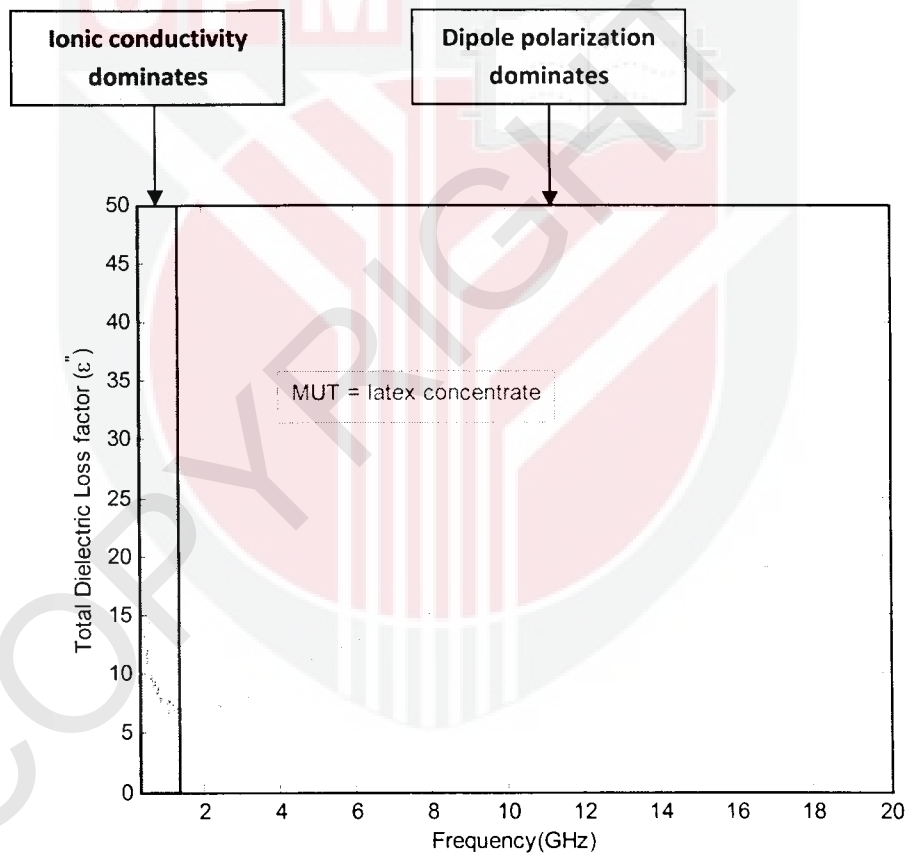


Figure 1.2. Effect of ionic conductivity and dipole polarization domination at microwave frequency region of concentrated latex sample.

Each polarization mechanism affects the loss factor of complex permittivity of materials. For example if dipolar polarization is the only mechanism leading to losses, the complex permittivity could be written as:

$$\varepsilon_d^* = \varepsilon' - j\varepsilon_d'' \quad (1.2)$$

where subscript d refers to loss due to dipolar polarization mechanism. However, overall loss of material can be defined as:

$$\varepsilon'' = \varepsilon_d'' + \varepsilon_e'' + \varepsilon_a'' + \varepsilon_i'' + \varepsilon_c'' \quad (1.3)$$

where the subscript d, e, a and i refer to dipolar, electronic, atomic, and interfacial polarization and ε_c'' is loss due to ionic conductivity which is defined as:

$$\varepsilon_c'' = \frac{\sigma}{2\pi f \varepsilon_0} \quad (1.4)$$

where σ is the ionic conductivity (S/m) and f is the operating frequency (Hz). However at microwave frequency, only ε_d'' , and ε_c'' are considered and all of these losses are dependent of frequency, moisture content, and ionic conductivity. These dependencies are discussed further in the next subsections.

1.2.1.1 Operating frequency

Microwave region seems could be divided into three region of frequency. The first region is the region where the loss is dominated by Maxwell-Wagner polarization and ionic conductivity. In this region material having high ion concentrations will have high 'loss factor' due to those mechanism. The second region is where the effect of dipolar polarization can be seen. Here, water molecules slightly rotate due

to dipolar polarization. At the same time, the mechanism occurs in the first region tend to decrease. Material 'loss factor' will have the lowest value in this region. The last region describes domination of dipolar polarization. At this range, the 'loss factor' due to dipolar polarization showing its peak at intermediate frequency and dielectric constant undergoes dispersion. This effect is called relaxation frequency.

When a measurement is made at certain range of frequencies, the best operating frequency needs to be chosen so that measured material permittivity is according to the objective. In this project, frequency of 300 MHz, 2 GHz and 5 GHz has been chosen as the operating frequencies of the developed microwave sensor system. The first two is used for conductivity detection and the last is for moisture content detection.

1.2.1.2 Moisture Content

Moisture content is defined as amount of water contains in a material. It is usually expressed in percentage. Conventional method that most commonly used is direct method such as oven drying and Karl-Fisher titration method. Oven drying method works by sample first is weighted to know the initial mass. Then the sample is dried in an oven having temperature at 70°C for about 18 to 24 hours to remove the water content before it is reweighted to determine the final mass.

Water in a sample of liquid may present in terms of bound and free water. Bound water has its molecules chemically combine with other molecules presence in the

material or physically absorbed to the surface of material (Kraszewski, 1996). It is a fact that low concentration of liquid sample has high moisture content since there are less tightly bound water molecules. An example of relationship of moisture content with material permittivity is established as shown in Figure 1.3.

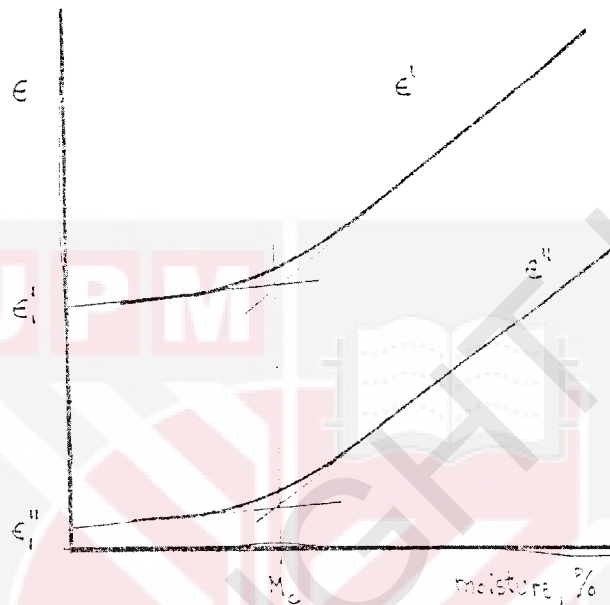


Figure 1.3. Material permittivity as a function of moisture content. (Source: Kraszewski, 1996)

1.2.2 Electrical Conductivity (Salt/Ion content)

Salt or ion content is measurable by electrical conductivity. Conductivity is a measure of how water can conduct electric current. Thus conductivity can only be measured when ions present. Standard method to measure conductivity is by having two parallel plates that is sandwiched within the sample which a low frequency potential is applied across the plates and the current is measured.

Ionic conductivity is extension of loss from imaginary part of complex permittivity function. The effect of conductivity can be seen at few megahertz of microwave frequency where the function of total dielectric loss is alike $1/f$ where f is the frequency of the propagating electromagnetic wave (Nyfors & Vainikainen, 1989). Dielectric loss due to ionic conductivity effect gets smaller and almost could not be seen as frequency gets into gigahertz range. This is because dipolar loss from water dominates the loss factor as frequency increases. Therefore, a good relationship of conductivity with material permittivity could only be established at megahertz range of microwave frequency for many materials.

1.3 Microwave Microstrip Sensor

Microstrip is a microwave planar transmission line having two conductors sandwiching a thin dielectric substrate as the geometry shown in Figure 1.4. The top conductor functions as a strip conducting line to propagate the wave and the bottom conductor acts as a ground. The wave propagation mode of microstrip line is quasi-transverse electromagnetic (quasi-TEM) (Figure 1.5). Microstrip line cannot support fully transverse electromagnetic (TEM) wave as the fields are not contained in single homogenous medium. Indeed, quasi-TEM is proposed because some of the fields radiate in air which the phenomenon is called as fringing field. Since air and dielectric substrate have different dielectric constant, ϵ_r , another term describing dielectric constant for both medium is proposed which is called as effective dielectric

constant, ϵ_{eff} . Information of ϵ_{eff} together with characteristic impedance of the line, Z_0 can be used to determine appropriate characteristic of microstrip geometry such as conductor strip width, w , and thickness of the substrate, d , as shown in Figure 1.4. It is also important to match Z_0 with line impedance to minimize the loss of transmitted signal. Details derivation and information of ϵ_{eff} and Z_0 is discussed in Chapter 3.

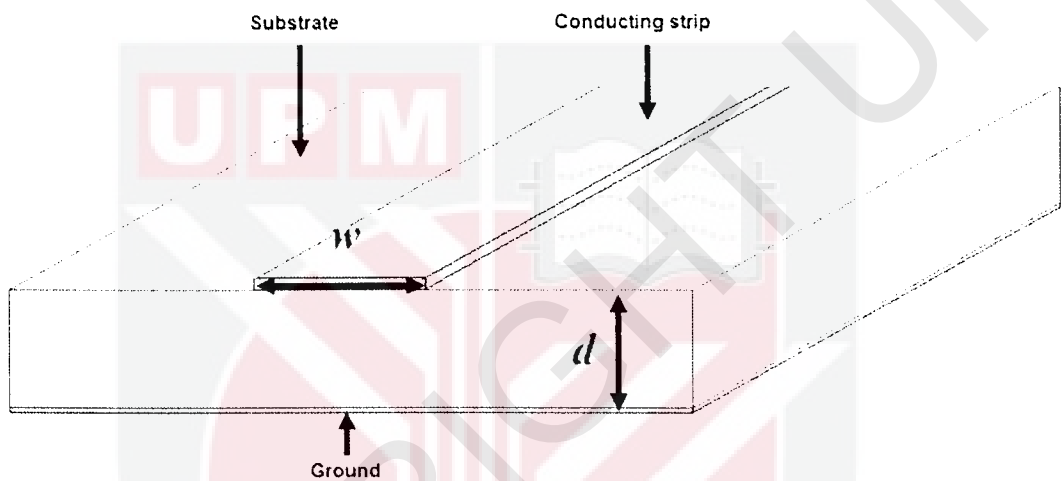


Figure 1.4. Geometry of microstrip transmission line.

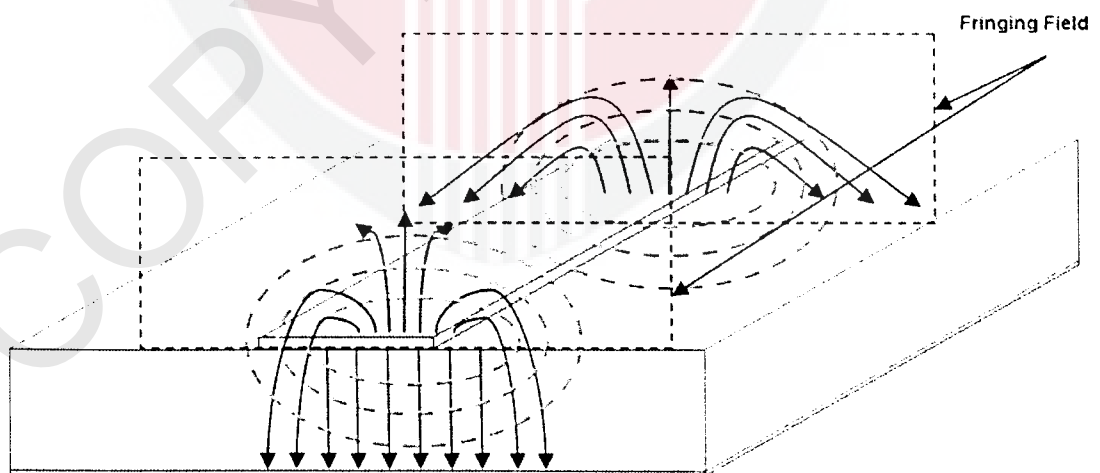


Figure 1.5. Electric (black line with arrow) and magnetic (red dashed line) fields of microstrip line with fringing field are shown.

In material sensing, the sample is usually placed in contact with the conducting strip as shown in Figure 1.6. Previously it is imagined that part of the electromagnetic fields radiate in air, however now the fields radiate into the sample medium. The sample now also has different dielectric properties means that it has different ϵ_r than air which ϵ_r is equals to 1. Accordingly the ϵ_{eff} will always change whenever different moisture level of sample is placed on the microstrip line. Different values of ϵ_{eff} effect values of Z_0 as well. However, values of ϵ_{eff} and Z_0 only do not characterize a microstrip sensor completely, the length of the strip also must also be taken into consideration. As the principle of microwave microstrip sensor is described, it is important to know all of these parameters to design a good microstrip sensor.

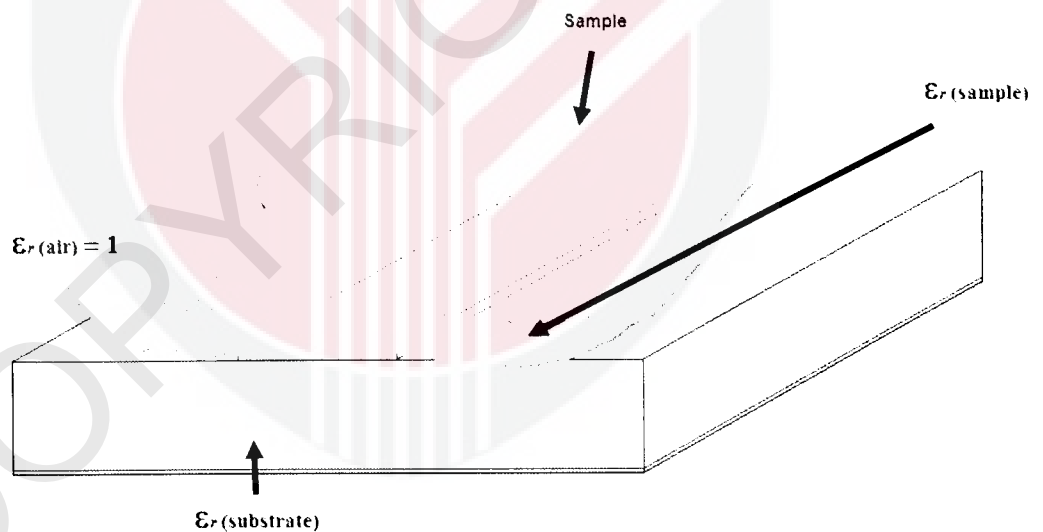


Figure 1.6. Example of measurement of materials with microstrip line sensor.

1.4 Objectives

The main aim of this thesis is to demonstrate a microwave measurement system that could simultaneously measure ionic conductivity and moisture content of various liquid materials.

Specific aims of the project are given as follows:

1. To measure the dielectric properties of various concentration of concentrated latex, fresh latex, sea water and saline water to determine the suitable region for ionic conductivity and moisture content of materials
2. To develop a good approximation of theoretical model of the microstrip sensor and system which allows the dielectric properties of the samples to be deduced from the scattering parameters of the sensor
3. To design and fabricate planar U-shaped microstrip sensors to be integrated in the system and measure scattering parameters of the sensors using tested samples by Personal Network Analyzer (PNA) to determine the best frequency to be used in the microwave system
4. To develop, test and calibrate the microwave measurement system using mentioned samples to verify its accuracy and reliability compared to standard method measurement and also along with theoretical models and establish a

relationship between microwave attenuation together with ionic conductivity and moisture content of the samples.

1.5 Overview of thesis

This thesis is organized into six chapters. Each of these chapters contains an independent introduction of the content and conclusion of the work in the end. The chapters present a complete and coherent story of the project done. First two chapters start with general idea and formulation of the problems. They provide project rationale and background, definitions to some related parameters which will be used extensively in the subsequent chapters and literature survey which address potential contribution in the area of microwave aquametry. The later chapters present thoroughly theoretical analysis of the sensor, development of prototype system and couple of important measurements methods. The last set of chapters then discuss significance of the results from the first two parts of the chapters and conclusions close the thesis.

Overview of the whole thesis is as follows:

Chapter 1 – Introduction

In current chapter, the aims, background and rationale of the research are presented.

This chapter provides the basic impetus of the study and some relative definitions of

microwave-based technique are presented. In addition, contributions made to the knowledge are highlighted.

Chapter 2 –Literature Review

In the second chapter, a general discussion and comparison of microwave aquametry technique and conductivity measurement are presented. Some important examples of previous applications of the technique are reviewed and summarized.

Chapter 3 – Quasi-TEM Analysis

In the third chapter, a thorough numerical model was developed to cater the parameters needed to design a transmission sensor utilizing planar microstrip line which is used in the microwave system. This chapter presents for the first time a numerical solution for the microstrip sensor which relates attenuation simultaneously with moisture content and conductivity.

Chapter 4 – Methodology

In the fourth chapter, the key discussion is about experimental work starting with development of the microwave system. Components build up the system are described in details. The system is the main measurement engine to monitor the liquid samples properties. Measurement of materials using high accuracy PNA to determine its complex permittivity is also mentioned as to support and provide comparison to data obtained from developed system.

Chapter 5 – Results and discussion

In the fifth chapter, comparison and contrast of the theoretical attenuation data with experimental results are done. Limitation and errors of approximation of the chosen model is discussed.

Chapter 6 – Conclusion and Recommendation for Future Research

In the final chapter, a closing discussion and concluding remarks are presented. This section provides an overall discussion of the problem raised in the thesis and points to new issues and questions for future research as result of the thesis.

1.5 Summary of Original Work

The following is the summary of original contributions in this thesis:

1. Development of numerical algorithm for calculating initial estimate of the ionic conductivity of liquid materials.
2. Development of two microstrip sensors to avoid necessity to calibrate the system using water.
3. Analysis of simultaneous measurement of conductivity and moisture content of a microwave sensor system.

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