Integrated Dual Frequency Permittivity Analyzer using Cavity Perturbation Concept

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ABSTRACT

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Optimal utilization of microwave energy requires more basic knowledge of the dielectric properties of the material under investigation. The dielectric properties of materials subjected to microwaves are known to depend on moisture content, temperature and density of the material as well as the frequency of the applied microwave field. This thesis is focussed on the development and evaluation of the new Permittivity Analyser to measure the dielectric properties of agri-food materials at 915 and 2450 MHz using cavity perturbation concept.

In this study, the dielectric properties measuring system was designed and developed using cavity perturbation concept to measure the essential and fundamental parameters of microwave-material interaction; dielectric constant and dielectric loss factor of selected agri-food substances and organic solvents. The TM_{010} mode of electromagnetic energy was selected and configured to operate at the peak resonant condition for both ISM (Industrial, Scientific and Medical) approved frequencies of 915 and 2450 MHz frequencies. The resonant perturbation cavities were designed, fabricated and tested using the network analyser and the permittivity analyser. High Q (ratio of energy supplied to absorbed) values were reported for both frequencies. Basic calibration of the measurement system was performed using standard media such as air, water and alcohol. Necessary mathematical steps and algorithms were written and integrated into a user-friendly software program (Visual basic 6.0) to carry out the entire measurement.

The dielectric properties (ε' & ε'') of materials such as; edible oils - canola, soya and sunflower oils, neem oil /pulp, homogenized milk (1, 2 and 3.25% fat), organic solvents such as ethanol, hexane and their mixtures were determined at various temperatures and frequency (915, 2450 MHz) combinations, using cavity perturbation technique. Linear relationships between the dielectric properties and temperature found in the literature were confirmed to be valid for certain ranges in case of edible oils, organic solvents and milk samples tested with the cavity perturbation method. Repeatibility and variability aspects of the permittivity analyzer at both the frequencies are presented.

Résumé

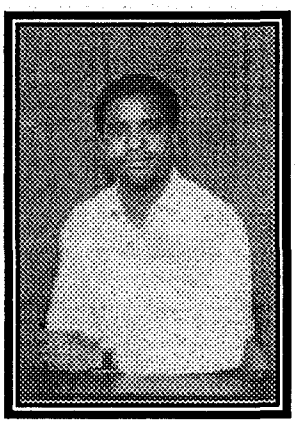
Venkatesh Meda **Ph.D. (Genie Biosystems)**

Analyseur Intégré à double fréquences pour la mesure de la constante diélectrique grâce à la théorie des perturbations en cavité résonante

L'utilisation optimale de l'énergie micro-onde nécessite une compréhension de base accrue des propriétés diélectriques des matériaux concernés. Les propriétés diélectriques des matériaux soumis au microonde sont reconnues dépendre du taux d'humidité, de la température et de la densité du matériau de même que la fréquence du champ micro-onde appliqué. Cette étude se penche sur le développement et l'évaluation d'un nouvel analyseur des propriétés diélectriques de matériau agro-alimentaires aux fréquences de 915 et 2450 MHz, utilisant la théorie des perturbations en cavité résonante.

Le système de mesure des propriétés diélectriques à été conçu et développé en utilisant la théorie des perturbations pour mesurer les paramètres essentiels et fondamentaux des interactions entre les micro-ondes et les matériaux. Cette étude a porté sur la constante diélectrique et le facteur de perte diélectrique d'un nombre sélectionné de substances agro-alimentaires et de solvants organiques. Le mode électromagnétique TM_{010} a été sélectionné et configuré afin d'opérer à la pointe de résonance des deux fréquences de la bande ISM de 915 et 2450 MHz. Les cavités résonantes ont été conçues, fabriquées et testées à l'aide d'un analyseur de réseau et de l'analyseur de permittivité. Un facteur de surtension élevé (mesure de la relation entre l'énergie accumulée et le taux de dissipation) a été noté pour les deux fréquences. Une calibration de base du système de mesure a été effectuée avec des éléments standards comme l'air, l'eau et l'alcool. Des routines et algorithmes mathématiques ont été mis au point et intégrés à un logiciel convivial (VB 6.0) permettant les calculs complets des mesures effectuées.

Les propriétés diélectriques (ε',ε'') des matériaux tels des huiles comestibles - huiles de canola, de soja et de tournesol - de l'huile de neem, du lait (1, 2 et 3.25% matières grasses), des solvants organiques-éthanol, hexane, et différentes combinaisons de ces composantes ont été analysées à différentes températures et différentes fréquences. Les relations linéaires, établies entre les propriétés diélectriques et la température, retrouvées dans divers travaux cités, ont été confirmées valides à certains degrés dans le cas des huiles comestibles, et dans le cas des solvants organiques et les échantillons de lait testés. La fidélité et le coefficient de variabilité de l'analyseur de permittivité aux deux fréquences sont analysés et présentés.



This thesis is dedicated in memory of our beloved father

Mr. Shashikumar Meda

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Nomenclature

ε_o	=	absolute permittivity of vacuum (8.854 X 10^{-12} F/m)
μ_o	=	magnetic permeability of free space (1.26 x 10^{-6} H/m)
ε^*	=	complex permittivity
ε_r'	=	relative dielectric constant (= ε' or eps')
ε_r''	=	loss factor (= ε" or eps")
tanδ	=	loss tangent or dissipation factor
d_p	=	depth of penetration (m)
ω	=	angular frequency (rad/sec)
σ	=	conductivity (S/m)
λ	=	wavelength (m)
E	=	RMS field intensity (V/m)
f	=	frequency (cycles/sec, Hz)
P_{abs}	=	absorbed power (W/cm^3)
P_v	=	energy developed per unit volume (W/cm^3)
TM_{mnk}	=	transverse magnetic; directions : x,y,z
m_1, m_2, m_3	=	constants
e	=	constant dependent on the material
ρ	=	density (g/cm^3)
a, b, c	=	constants
A_o, A_1	=	regression coefficients
r^2	=	coefficient of determination
s.e.	=	standard error of estimate
MHz	=	mega hertz (10^6 Hz or cps)
GHz	=	giga hertz (10^9 Hz or cps)
Q_o	=	quality factor of empty cavity
Q_s	=	quality factor of cavity with sample
V_o	=	volume of empty cavity (mm^3)
V_s	=	volume of cavity with sample (mm^3)
Δf	=	shift in resonant frequency (Hz)
P	=	power loss (dB)

ω_o	=	resonant frequency ($=2\pi f_o$)
c_f	=	center frequency (Hz)
W_{max}	=	energy received by the resonator (Watts)
$\omega_{1,2}$	=	volume fraction
C_p	=	bidirectional coupler
C	=	Capacitance (Farad)
C_o	=	speed of light (m/sec)
A	=	amplifier
A_t	=	attenuator
T	=	relaxation time
dB	=	decibels
v_s	=	sample volume (mm^3)
µl	=	microlitre (10^{-6})
MW	=	Micro-Wave
M.C.	=	Moisture Content, %
a_w	=	water activity
S/N	=	signal to noise ratio
DC	=	direct current
Δf	=	Shift in Resonant Frequency (MHz)

1 INTRODUCTION

1.1 Background

Interest in the electrical properties of agricultural products dates back to more than 80 years. The earliest reference to the dielectric properties of materials appeared in 1929, where Debye (1929) described the dielectric behavior of polar molecules, such as water. It is primarily the response of polar molecules that gives rise to heat generation when materials containing such molecules are subjected to electromagnetic fields in the radio (30 to 300 MHz) and microwave (300 to 3000 MHz) frequency bands. The instantaneous internal heat generation which may be induced by this transfer of radiant energy, has been the basis of a great deal of recent work on alternate thermal processing methods for agricultural and food materials, although most of the work has been restricted to the ISM (Industrial, Scientific, Medical) frequencies of 915 and 2450 MHz to minimize interference with microwave frequencies used for telecommunications, defence and navigation.

Perhaps the greatest incentive for using microwave energy for thermal treatment of agri-food materials is the speed at which processing may be done compared to conduction or convection-based technologies (Metaxas, 1996). Broader issues also come into play, in particular the increased use of hydroelectric power at the detriment of further expansion of the use of fossil fuels for thermal processing (Raghavan et al., 1993). Thus, the development of microwave and RF-based applications can be justified on physical, as well as environmental grounds. Today, research in this area focuses on the measurement of dielectric properties, the development of techniques to measure these properties, and the development of electro-technologies for thermal processing applications.

The degree to which a given material responds to an electromagnetic field depends on the frequency and intensity of the field, on the one hand, and on the characteristics of the material itself. The characteristics of the material itself are also classified into two groups: specific characteristics and bulk characteristics. Specific characteristics are the chemical composition, surface structure, density and moisture content (and to a certain extent moisture profile) of integral particles, whereas bulk characteristics include size, shape and porosity. Another important variable is the temperature, which is initially an environmental factor, but which, during processing, becomes a function of electromagnetic field parameters and the material properties. The dielectric response of a material usually increases with temperature since more of the bound dipoles become free to oscillate as

the overall kinetic energy level increases. Minor and possibly undetectable changes due to changes in chemical composition through heat-induced reactions and volatilization may also occur.

The electrical basis of interaction is described in terms of the dielectric properties - dielectric constant and dielectric loss factor. These respectively represent the proportion of impinging energy that can penetrate the material, and the amount of energy that can be absorbed. These two parameters are functions of the specific characteristics of the material and of temperature, for a given frequency. The actual energy absorbed and dissipated as heat by a given mass of material depends on both the specific and bulk characteristics of the material.

The dielectric properties of a material are usually given as functions of field frequency, material temperature and material moisture content. Several authors have also studied the changes in dielectric properties associated with levels of some chemical components such as fats, proteins (Sun et al., 1995) and dissolved ions (Krazsewski 1996, Tinga & Nelson, 1973; Bengtsson & Risman, 1971), while still others have described "bulk dielectric properties" in terms of bulk density (Nelson; 1984, 1986, 1987), sieve size of ground materials (Nelson, 1984; Venkatesh, 1996), and so on. However, data obtained on the same material have, in several instances, been found to differ significantly. In some cases such differences have been attributed to measurement technique, while in others, compositional differences of the materials have been suspected (Sun et al., 1995).

Measurement of dielectric properties is important from two points of view. First, since dielectric properties are transient during processing, functional relationships describing these changes are needed for process control and optimization. Second, since dielectric properties are intrinsically related to specific and bulk characteristics of materials, the development of rapid measurement techniques can potentially lead to rapid indirect determination of a multitude of material properties. The best known instance of this use is the dielectric determination of moisture content in materials requiring this parameter for quality control (Nelson et al. 1990). Although a great deal of progress has been achieved in the development of instrumentation and in the understanding of the functional relationships between dielectric properties and other parameters, the growing trend towards microwave processing has created a need for more flexible and accessible equipment to quantify the dielectric properties of many agri-food products under a wider

variety of conditions. Existing measurement technology is very expensive and the applicability of the various measurement techniques to certain types of material has been questioned (Sun et al., 1995). Some limitations have been identified in the preliminary work to be described later.

Prior knowledge of these functions is invaluable to process development and control and is the main reason why there has been considerable research into the measurement of the dielectric properties. Although various kinds of apparatus have been used to measure dielectric properties, the task of describing these functions sufficiently accurately at both allowable wavelengths over the range of temperatures and other factors likely to be encountered in a given processing situation, is quite lengthy, regardless of the limitations of the particular technique used. For this reason, after a number of preliminary studies and literature review, it was decided to attempt to develop and integrate the dielectric properties measurement set up using the Cavity Perturbation concept that would permit measurement at both allowable frequencies (915, 2450 MHz) over a wide range of temperatures, and to evaluate measurements against literature data. With the need for development of new sensing devices for the automation and control of various agricultural and food processes, there is a need for better understanding of the usefulness of dielectric properties of materials and methods for measuring these properties.

Thus, the research proposed here has two motivations: (i) to develop a less expensive and more flexible system for determining the dielectric properties of agri-food materials, and (ii) to elaborate the functional relationships for the dielectric properties of specific materials. The data will also be used to evaluate the new system.

1.2 Hypothesis

The research proposed here is based on the hypothesis that it is possible to develop a system to measure the dielectric properties of agri-food products which is less expensive and more flexible than existing equipment, thus permitting more detailed studies of the functional relationships over a wider range of conditions using cavity perturbation technique.

1.3 Objectives

The main objective of this research is to further our understanding of the dielectric properties and behavior of agricultural and food materials subjected to electromagnetic waves (microwaves). The specific objectives are:

- To design and develop equipment permitting measurement of dielectric properties over a wide range of temperatures (-30 to 100° C), at two different ISM approved frequencies (915, 2450 MHz).
- To test and evaluate the performance of the new equipment and compare the data obtained on specific agri-food materials with those obtained by other techniques or systems.
- To study the dielectric responses of agri-food materials at critical conditions, such as phase change or threshold ionic conditions.
- To develop a control software that integrates both the operating frequencies.

II REVIEW OF LITERATURE

2.1 Microwave Processing: Fundamentals And Application

In North America, only four microwave and three radio frequencies are permitted by the Federal Communications Commission (FCC) for dielectric heating applications (Decareau, 1985). The allotted microwave frequencies are 915, 2450, 5800 MHz and the radio frequencies are 13.56, 27.12, and 40.68 MHz. Although 915 MHz is permitted by both North and South America, most of the commercial microwave processing equipment is designed for operation at 2450 MHz. In Europe, other frequencies have been allocated for commercial use in the radio-frequency region of the spectrum.

Dielectric properties of agricultural materials and products are finding increasing application as new technology is adapted for use in agriculture and food related industries. The interest in dielectric properties of materials has historically been associated with the design of electrical equipment, where various dielectrics are used for insulating conductors and other components of electric equipment. During the past century, materials research has provided many new dielectric materials for application in electronics. As the use of higher and higher frequencies came into practice, new materials, suitable for use in the radio frequency, microwave, and millimeter wave regions of the electromagnetic spectrum, have been developed. The dielectric properties of these materials are important in the design of electrical and electronics equipment, and suitable techniques for measuring the dielectric properties of various materials have been developed as they were needed.

The interest in the dielectric properties of agricultural materials and products has been principally for predicting heating rates describing the behavior of materials when subjected to high-frequency or microwave electric fields in dielectric heating applications and as indicators of moisture content in the development of rapid techniques for moisture determination. The influence of the dielectric properties on the heating of materials by absorption of energy through radio-frequency dielectric heating, whether at high frequencies or microwave frequencies, has been well known for a long time, and many potential applications have been investigated (Brown et al. 1947, Thuery, 1992, Metaxas and Meredith, 1983). With the advent of commercial microwave heating and the wide acceptance of microwave ovens for the home, the concepts of dielectric heating have become more popular.

The use of dielectric properties for measuring moisture content of products such as cereal grains has been recognized for at least 75 years and has been in common use for more than 48 years (Nelson, 1977, 1991). However the first dielectric properties for grain were not reported until 45 years ago (Nelson et al., 1953). Since then much data and information on the dielectric properties of grain and other agricultural products have become available (Nelson, 1965, 1973, ASAE Standards, 1988, Tinga and Nelson, 1973, Kent 1987, Datta et al., 1995), and the influence of important variables on these dielectric properties has been evaluated (Nelson, 1981, 1991, Mudgett, 1995).

In the past 20 years, the microwave oven has become an essential appliance in most kitchens. Faster cooking times and energy savings over conventional cooking methods are the primary benefits. Although the use of microwaves for cooking food is widespread, the application of this technology to the processing of materials is a relatively new development. The use of microwave energy for processing materials has the potential to offer similar advantages in reduced processing times and energy savings (Thostenson and Chou, 1999).

In conventional thermal processing, energy is transferred to the material through convection, conduction, and radiation of heat from the surfaces of the material. In contrast, microwave energy is delivered directly to materials through molecular interaction with the electromagnetic field. In conventional methods energy is transferred due to thermal gradients, but microwave heating is the conversion of electromagnetic energy to thermal energy through direct interaction of the incident radiation with the target material's molecules. The difference in the way energy is delivered can result in many potential advantages to using microwaves for processing of materials. Because microwaves can penetrate materials and deposit energy, heat can be generated throughout the volume of the material. The transfer of energy does not rely on diffusion of heat from the surfaces, and it is possible to achieve rapid and uniform heating of relatively thicker materials. In traditional heating, the cycle time is often dominated by slow heating rates that are chosen to minimize steep thermal gradients that result in process-induced stress. For polymers and ceramics, which are materials with low thermal conductivity, this can result in significantly reduced processing times. Thus, there is a balance between processing time and product quality in conventional processing (this is also true of microwave heating). Since microwaves can transfer energy throughout the volume of the material, the potential

exists to reduce processing time and enhance overall quality (Thostenson and Chou, 1999).

In addition to volumetric heating, energy transfer at a molecular level can have some additional advantages. Microwaves can be utilized for selective handling of materials. The molecular structure affects the ability of the microwaves to interact with materials and transfer energy. When materials in contact have different dielectric properties, microwaves will selectively couple with the higher lossy material. This phenomenon of selective heating can be used for a number of purposes. In conventional joining of ceramics or polymers, considerable time and energy is wasted in heating up the interface by conduction through the substrates. With microwaves, the joint interface can be heated in-situ by incorporating a higher lossy material at the interface. In multiple phase materials, some phases may couple more readily with microwaves. Thus, it may be possible to process materials with new or unique micro-structures by selectively heating distinct phases. Microwaves may also be able to initiate chemical reactions not possible in conventional processing through selective heating of reactants. Thus, new materials may be created.

Although direct heating by microwaves can offer advantages over conventional heat transfer, the different mechanism of energy transfer in microwave heating has led to new processing challenges. As materials are processed, they often undergo physical and structural transformations that affect the dielectric properties. Thus, the ability of microwaves to generate heat varies during the process. Sharp transformations in the ability of microwaves to generate heat can cause difficulties with the process modeling and control. Understanding the generation, propagation, and interaction of microwaves with materials is critical. Because the processing equipment determines the electromagnetic field, the design of microwave equipment is particularly important. The properties of the electromagnetic field, chemical composition of the material being processed, structural changes that occur during processing, size and shape of the object being heated, and the physics of the microwave/materials interaction all complicate microwave processing.

Recent interest in microwave processing of materials is highlighted in a number of recent symposia that have been dedicated to microwave processing of materials. To date, the Material Research Society (MRS), IMPI / IEEE and the American Ceramic Society have held a number of symposia that have focused on microwave processing of materials. The

recent research in microwave equipment design, microwave/materials interactions, dielectric properties measurement and materials processing continues to expand interest in microwave techniques.

2.2 Microwave-material interaction aspects

When microwaves are directed towards a material, part of the energy is reflected, part is transmitted through the surface, and of this latter quantity, part of it is absorbed. The proportions of energy which fall into these three categories have been defined in terms of the dielectric properties. The fundamental electrical property through which the interactions are described is the complex relative permittivity of the material, ε^*. It is mathematically expressed as:

$$\varepsilon^* = \varepsilon' - j\varepsilon'' \qquad (2.1)$$

where ε' is the dielectric constant and ε'' the dielectric loss factor. The absolute permittivity of a vacuum is ε_0 and it is determined by the speed of light (C_0) and the magnetic constant (μ_0), which are linked together by the equation :

$$C_o^2 \, \mu_o \, \varepsilon_o = 1 \qquad (2.2)$$

The numerical value for ε_0 is about 8.854×10^{-12} F/m and $\mu = 1.26 \times 10^{-6}$ H/m. In other media (solid, liquid and gaseous), the permittivity has higher values and it is usually expressed relative to the value in vacuum (Nyfors & Vainikainen, 1989) : ε_r, the relative permittivity of a material is equal to $\varepsilon_{abs}/\varepsilon_o$, where ε_{abs} is the absolute permittivity of material. Materials which do not contain magnetic components, respond only to the electric field.

The dielectric properties of materials dictate, to a large extent, the behavior of the materials when subjected to radio-frequency (RF) or microwave field for purposes of heating, drying or processing the materials. The characterization of dielectric properties is vital for understanding the response of a material to microwaves, since most useful quantities needed in the design of microwave thermal processes can be described in terms of them. The equations relating dielectric properties to thermal processing parameters are presented in the following section.

2.3 Quantities expressed in terms of dielectric properties

The proportion of reflected energy is a function of the dielectric constant, ε' and the angle of incidence. For an angle of incidence of 90°, it is simply:

$$P_r(90) = \left[\frac{\sqrt{\varepsilon'} - 1}{\sqrt{\varepsilon'} + 1}\right]^2 \qquad (2.3)$$

For example, the ε' of water is 78 at room temperature. The reflectivity is therefore greater than 0.64. However, the reflected energy will also reflect from the walls of the chamber and impinge on the water over and over again, resulting in a specific reflectivity of about 0.20. Thus, about 80% of the incident energy is absorbed by the target material and the rest is absorbed and dissipated by the walls of the microwave chamber. The ε' of moist foods is usually in the range of 50 - 70.

The power transmitted to the material is simply $P_{trans} = (1-P_r)$. The power absorbed and then generated as heat is directly related to ε" (lossiness) of the material, and can be calculated using the relation:

$$P_{Abs} = 5.56 * 10^{-4} f \varepsilon'' E^2 \qquad (2.4)$$

where :

P_{Abs} = absorbed power (W/cm^3)

E = rms electric field intensity (volts/cm) inside given volume

f = frequency (Hz)

ε" = dielectric loss factor of the food or other product.

The penetration depth, d_p is usually defined as the depth into a sample where the microwave power has dropped to 1/e or 36.8% of its transmitted value. Sometimes, d_p is defined as the distance at which the microwave power has been attenuated to 50% of P_{trans}. The penetration depth is a function of ε' and ε" :

$$d_p = \frac{\lambda_o \sqrt{\varepsilon'}}{2\pi\varepsilon''} \qquad (2.5)$$

where, λ_o = free space microwave wavelength; (for 2450 MHz, λ_o = 12.2 cm). The most common food products have ε"< 25, which implies a d_p of 0.6 -1.0 cm. Although dielectric

properties of some foods can be found in the literature (Thuery, 1992), data are mostly limited to pure foods and food components. As the wave travels through a material that has significant dielectric loss, its energy will be attenuated. If the attenuation is high in the material, the dielectric heating will taper off quickly as the wave penetrates the material. Attenuation is often expressed in decibels per meter (dB/m). In terms of power densities and electric field intensity values, this can be expressed as:

$$10\log_{10}\left(\frac{P_0}{P(z)}\right)=20\log_{10}\left(\frac{E_0}{E(z)}\right)=8.686\alpha(z) \qquad (2.6)$$

where P_0 is the power level at a point of reference, P(z) is the power level at distance z from the reference point, and α is the attenuation in nepers/m.

The power dissipated inside a material is proportional to ε". The ratio, ε"/ε', called the loss tangent or dissipation factor, a descriptive dielectric parameter, is also used as an index of the material's ability to generate heat (Mudgett, 1986).

The rate of heating can be expressed by the power equation :

$$P_v = 2\pi f \varepsilon_o \varepsilon'' |E|^2 \qquad (2.7)$$

where :

P_v = energy developed per unit volume (W/m^3)

f = frequency (Hz)

|E| = electric field strength inside the load (V/m).

The electric field inside the load is determined by the dielectric properties and the geometry of the load, and by the oven configuration. Therefore, this equation is generally impractical since the determination of the electric field distribution is very complex (Buffler, 1993). The heating effect in agri-food materials is the result of two mechanisms, dipolar rotation and ionic conduction. The dipole most responsible for heating is water, which is a major constituent of most agri-food materials. The response of dipoles to an oscillating field is an increase in rotational and vibrational energies, depending on the degree of symmetry of the molecule, with a resulting frictional generation of heat. Molecules that are non-polar but that are asymmetrically charged may behave as dipoles in an electrical field, however their responses to microwave energy are usually about an order of magnitude

less than that of water.

The other heating mechanism is ionic conduction. The electrical field causes dissolved ions of positive and negative charge to migrate towards oppositely charged regions. This results in multiple billiard-ball-like collisions and disruption of hydrogen bonds with water, both of which result in generation of heat. Ionic conduction has a larger influence on ε" than on ε', and therefore decreases the penetration depth. This behavior is predicted by the Hasted-Debye relations for aqueous electrolytic solutions (Hasted, 1948). There is a depression of ε' due to depletion of free water by dissolved ions and an increase in ε" due to an increase in the free charge density (Kudra et al., 1992). Magnetic field interactions are negligible, since foods contain only trace amounts of magnetic minerals such as nickel, cobalt, and iron.

The temperature profile and heating rate developed during exposure to electromagnetic radiation depends on the distribution and nature of susceptors, the relationships between the dielectric properties with moisture and temperature and frequency, as well as on the thermo-physical properties (thermal conductivity, thermal diffusivity, specific heat, etc.) of the other constituents. A detailed description of the temperature profile of a complex agri-food material is therefore extremely difficult to obtain.

It is important to recognize that the dielectric properties are not unique for a given material. They are specific only for a given frequency and state of the material. Thus, in a processing situation, they are transient since the state of the material is not constant in time. Therefore, it is necessary to establish the relationships between ε' and ε" with state variables at the processing frequency(ies). The principal state variables that influence the dielectric properties at a given frequency are temperature and moisture content. Relationships between these factors, composition and the dielectric properties of agri-food materials are reviewed in the following sections.

2.4 Factors Influencing Dielectric Properties at a Given Frequency

2.4.1 Temperature and moisture content dependencies

The dielectric properties of most materials vary with several different factors. In hygroscopic materials such as agri-foods, the amount of water in the material is generally a dominant factor. The dielectric properties depend on the frequency of the applied alternating electric field, the temperature of the material, and on the density, composition,

and structure of the material. In granular or particulate materials, the bulk density of the air-particle mixture is another factor that influences the dielectric properties. Of course, the dielectric properties of materials are dependent on their chemical composition and especially on the permanent dipole moments associated with water and any other molecules making up the material of interest. With the exception of some extremely low-loss materials, i.e., materials that absorb essentially no energy from RF and microwave fields, the dielectric properties of most materials vary considerably with the frequency of the applied electric fields. This frequency dependence has been well reported elsewhere in the literature. An important phenomenon contributing to the frequency dependence of the dielectric properties is the polarization arising from the orientation with the imposed electric field, of molecules which have permanent dipole moments.

There are several reviews of the dielectric properties at microwave frequencies of food and agricultural products (Bengtsson and Risman, 1971; Nelson, 1973; Ohlsson and Bengtsson, 1975; Kent, 1987). The temperature dependence of the dielectric constant is quite complex, and it may increase or decrease with temperature depending upon the material. Microwave heating is greatly affected by presence of water in foods (von Hippel, 1954; Mudgett, 1985; Nelson, 1990). Water is the major absorber of microwave energy in the foods and consequently, the higher the moisture content, the better the heating. In its pure form, water is a classic example of a polar dielectric. The dielectric properties of liquid water are listed in **Table 2.1** for several microwave frequencies at temperatures of 20 and 50°C as selected from data listed by Hasted (1973) and Kaatze (1989). Although earlier work indicated a better fit of experimental data with the Cole-Cole model rather than the Debye model, Kaatze (1989) has shown that the dielectric spectra for pure water can be sufficiently well represented by the Debye model when using the relaxation parameters given in **Table 2.2**. The relaxation frequency, $(2\pi T)^{-1}$, is provided in **Table 2.2** along with the static and high frequency values of the dielectric constant, ε_s and ε_∞, for water at temperatures from 0 to 60°C. These values can be used to provide close estimates for the dielectric properties of water over a wide range of frequencies and temperatures. Also, water is a strong, broad-band absorber of microwaves and is therefore used for selective heating during food processing (Craig et al. 1995). The amount of free moisture in a substance greatly affects its dielectric constant since that of free water is high (approx. 78

Table 2.1 Microwave dielectric properties of water at indicated temperatures (Hasted, 1973).

Frequency (GHz)	ϵ' (20°C)	ϵ' (50°C)	ε'' (20°C)	ϵ'' (50°C)
0.6	80.3	69.9	2.75	1.25
1.7	79.2	69.7	7.9	3.6
3.0	77.4	68.4	13.0	5.8
4.6	74.0	68.5	18.8	9.4
7.7	67.4	67.2	28.2	14.5
9.1	63.0	65.5	31.5	16.5
12.5	53.6	61.5	35.5	21.4
17.4	42.0	56.3	37.1	27.2
26.8	26.5	44.2	33.9	32.0
36.4	17.6	34.3	28.8	32.6

Table 2.2 Debye dielectric relaxation parameters for water (Hasted, 1973).

Temperature (°C)	ϵ_s	ϵ_∞	T (ps)	Relaxation Frequency (GHz)
0.0	87.9	5.7	17.67	9.007
10.0	83.9	5.5	12.68	12.552
20.0	80.2	5.6	9.36	17.004
30.0	76.6	5.2	7.28	21.862
40.0	73.2	3.9	5.82	27.346
50.0	69.9	4.0	4.75	33.506
60.0	66.7	4.2	4.01	39.690

at room temperature and 2450 MHz). That of container materials, such as Teflon, is of the order of 2. The moisture relationship is consistent in that higher moisture leads to higher values of both the dielectric constant and the loss factor. This relationship is temperature dependent. However, water in its pure liquid state appears in food products very rarely. Most often, it has dissolved constituents, is physically absorbed in material capillaries or cavities, or is chemically bound to other molecules of water. Dielectric relaxations of absorbed water take place at lower frequencies than the relaxation of free water (Hasted, 1973), which occurs at about 19.5 GHz for water at 25°C. Depending upon the material structure, there may be various forms of bound water, differing in energy of binding and in dielectric properties. Moist material, in practice, is usually an inhomogeneous mixture, often containing more than one substance with unknown dielectric properties. Thus, it becomes very difficult to understand and predict the dielectric behavior of such materials at different frequencies, temperatures, or hydration levels. At present, very little is known about the dielectric properties of moist materials of different structures containing water at various levels of binding. However, a complete understanding is not always necessary for the solution of practical problems.

As mentioned earlier, the dielectric properties of materials are quite dependent on temperature, and the nature of that dependence is a function of the dielectric relaxation processes operating under the particular conditions existing and the frequency being used. As temperature increases, the relaxation time decreases, and the loss-factor peak will shift to higher frequencies. Thus, in a region of dispersion, the dielectric constant will increase with increasing temperature, whereas the loss factor may either increase or decrease, depending on whether the operating frequency is higher or lower than the relaxation frequency. Below and above the region of dispersion, the dielectric constant decreases with increasing temperature. Distribution functions can be useful in expressing the temperature dependence of dielectric properties but the frequency and temperature dependent behavior of the dielectric properties of most materials is complicated and can perhaps best be determined by measurement at the frequencies and under the conditions of interest.

Dielectric constants and loss factors of fresh fruits and vegetables have been explored by several researchers (Tran et al., 1984, Nelson et al., 1994). Examples of the frequency dependence observed for twenty-four different fruits and vegetables are

illustrated. The dielectric constant exhibits the expected monotonic decrease in value with frequency in the 0.2 to 20 GHz range. In all the fruits and vegetables measured, the loss factor decreases as frequency increases from 0.2 GHz, reaches a broad minimum in the region between 1 and 3 GHz, and then increases as frequency approaches 20 GHz. This behavior is dominated by ionic conductivity at lower frequencies, by bound water relaxation, and by free water relaxation near the top of the frequency range. The dielectric properties of these fresh fruits and vegetables at 915 MHz and 2.45 GHz are shown in **Table 2.3** along with other descriptive information. **Table 2.4** shows the permittivities of milk and its constituents (Kudra et al, 1992).

The dielectric properties of powdered potato starch : Sx 920, Matheson, Coleman & Bell; locust bean gum : Germantown Manufacturing Co.; and carrageenan : product specification no. 160, type Seakem CM 514, lot no. 340807, marine colloids division, FMC corp;, were found to increase regularly with moisture content in the range of 0 to 20 % wet basis , at 2.45 GHz (Nelson, 1991). The dielectric properties of these hydro colloids were also found to increase with temperature over the range 20 to 100°C, and the temperature difference increased markedly as moisture content increased (Nelson et al. 1991).

It is important to note that food electrical and physical properties which affect microwave heating change dramatically at temperatures below freezing point. Ohlsson et al. (1974) found that ε' and ε'' increased significantly with falling frequency for most foods tested, and in most cases, dielectric properties increased sharply with temperature during the transition from -10 to 0°C (thawing). In preliminary studies of the dielectric properties of Tylose at different salt concentrations Venkatesh et al. (1996) observed a sharp increase in the loss factor from -20 to -15°C, with trends thereafter dependent on salt concentration.

Table 2.3 Permittivities of fresh fruits and vegetables at 23°C (Nelson et al. 1994).

Fruit / vegetable	M.C.(wb) (%)	Tissue Density(g/cm^3)	915 MHz		2450 MHz	
			ε'	ε''	ε'	ε''
Apple	88	0.76	57	8	54	10
Avocado	71	0.99	47	16	45	12
Banana	78	0.94	64	19	60	18
Cantaloupe	92	0.93	68	14	66	13
Carrot	87	0.99	59	18	56	15
Cucumber	97	0.85	71	11	69	12
Grape	82	1.10	69	15	65	17
Grapefruit	91	0.83	75	14	73	15
Honeydew	89	0.95	72	18	69	17
Kiwifruit	87	0.99	70	18	66	17
Lemon	91	0.88	73	15	71	14
Lime	90	0.97	72	18	70	15
Mango	86	0.96	64	13	61	14
Onion	92	0.97	61	12	64	14
Orange	87	0.92	73	14	69	16
Papaya	88	0.96	69	10	67	14
Peach	90	0.92	70	12	67	14
Pear	84	0.94	67	11	64	13
Potato	79	1.03	62	22	57	17
Radish	96	0.76	68	20	67	15
Squash	95	0.70	63	15	62	13
Strawberry	92	0.76	73	14	71	14
S. potato	80	0.95	55	16	52	14
Turnip	92	0.89	63	13	61	12

Table 2.4 Dielectric properties of milk and its constituents at 2.45 Ghz and 20°C (Kudra et al. 1992).

Description	Fat (%)	Protein (%)	Lactose (%)	Moisture (%)	ε'	ε''
1% Milk	0.94	3.31	4.93	90.11	70.6	17.6
3.25% Milk	3.17	3.25	4.79	88.13	68.0	17.6
Water + Lactose I	0	0	4.0	96.0	78.2	13.8
Water + Lactose II	0	0	7.0	93.00	77.3	14.4
Water + Lactose III	0	0	10.0	90.0	76.3	14.9
Water + Sodium Caseinate I	0	3.33	0	96.67	74.6	15.5
Water + Sodium Caseinate II	0	6.48	0	93.62	73.0	15.7
Water + Sodium Caseinate III	0	8.71	0	91.29	71.4	15.9
Lactose (Solid)	0	0	100	0	1.9	0.0
Sodium Caseinate (solid)	0	100	0	0	1.6	0.0
Milk Fat (solid)	100	0	0	0	2.6	0.2
Water (distilled)	0	0	0	100	78.0	13.4

2.4.2 Influence of composition

The dielectric properties of food products are also determined by their chemical composition. The influence of water and salt (or ash) content depends to a large extent on the manner in which they are bound or restricted in their movement by the other food components. This complicates the prediction, based on data for single ingredients, of the dielectric properties of a mixture. The organic constituents of food are dielectrically inert ($\varepsilon' < 3$ and $\varepsilon'' < 0.1$) and, compared to aqueous ionic fluids or water, may be considered transparent to energy (Mudgett, 1985). Only at very low moisture levels, when the

remaining traces of water are bound and unaffected by the rapidly changing MW field, do the components of low specific heat become the major factors in heating.

Ohlsson et al. (1974) found that for many foods, the influence of different water and salt content on dielectric properties was significantly large, especially at 450 and 900 MHz. It was also found that at temperatures above 60°C, ε' decreased gently with temperature, whereas ε" increased, particularly at lower frequencies for salty foods (Bengtsson et al. 1980). The dielectric properties of some foods as a function of temperature, at 2.8 GHz, are documented by Bengtsson & Risman (1971). A food map of some foods at 2450 MHz and 20-25°C has been illustrated by Buffler & Stanford (1991).

In the case of high carbohydrate foods and syrups, the dissolved sugars (in water) are the main MW susceptors (Mudgett, 1986). Dielectric properties of aqueous sugar solutions of different concentrations have been measured and compared with those of grapes of similar moisture concentrations (Tulasidas et al. 1995). In the case of high moisture in grapes, the dielectric constant and loss factor decreased with an increase of temperature and a reversed trend was observed in the lower moisture range (Tulasidas and Raghavan, 1995).

While alcohols and dissolved carbohydrates are active ingredients in some foods and beverages, their effects are negligible in most food products, except for high carbohydrate foods, such as bakery products or syrups, and alcoholic beverages. The effects are related to stabilization of hydrogen bonding patterns through hydroxyl-water interaction (Roebuck et al. 1972). The effects of pH are not believed to be significant per se, at the pH levels typical in foods (Ohlsson et al., 1974; Mudgett, 1986; Nyfors & Vainikainen, 1989).

The effect of fat on dielectric properties appears to be that of dilution (more fat, less water). The heating rate seems to have no effect per se on the dielectric properties, unless water and juices are lost. At lower frequencies (450 and 900 MHz), the relationships appear to be similar (Bengtsson and Risman, 1971; Ohlsson et al, 1974).

The microwave heating characteristics of extraction mixtures consisting of rosemary or peppermint leaves suspended in hexane, ethanol and hexane plus ethanol mixtures were reported and it was found that the temperature rises were dependent on the dielectric properties of the solvents and the leaves in question (Chen and Spiro, 1994).

There have been several attempts to develop relationships between the dielectric

properties and composition, based on weighted averages of the dielectric properties of individual components (eg. Sun et al., 1995; Kudra et al., 1992). However, these studies imply that the approach is incomplete due to possible synergistic and loss effects. Essentially, cross-binding of components in the parent material seems to play a role that cannot be easily accounted for when measurements are done on the components individually. The dielectric properties of four different processed cheeses are shown in **Table 2.5** for temperatures of 20 and 70°C (Datta, 1999). At higher moisture and lower fat contents, the loss factor increased somewhat with temperature.

2.4.3 Physical structure of target material

Dielectric properties vary with a number of physical attributes including bulk density, particle size, and homogeneity. Nelson (1992) developed relations for the dielectric properties of whole and powdered grains at different bulk densities, moisture content and frequency. Bulk density has been identified as a major source of variation for ε' and ε" (Kent, 1977; Nelson, 1983a, b; Nelson, 1984a, b; Kent and Kress-Rogers, 1986 and Nelson and You, 1989). The density dependence of the dielectric properties of materials must be accounted for in elaborating functions determining grain moisture content (Meyer and Schilz, 1980). This relation could also be used in the control of continuous on-line processing of the grain.

Table 2.5 Dielectric properties of processed cheese at 2.45 GHz as related to composition and temperatures 20°C & 70°C (Datta, A.K, 1999).

Composition		Dielectric Constant and Loss Factor			
% Fat	% Moisture	ε' (20)	ε" (20)	ε' (70)	ε"(70)
0	67	43	29	43	37
12	55	30	21	32	23
24	43	20	14	22	17
36	31	14	8	13	9

2.4.4 Density dependence

Since the influence of a dielectric depends on the amount of mass interacting with the electromagnetic fields, the mass per unit volume, or density, will have an effect on the dielectric properties (Nelson & Datta, 1999). This is especially notable with particulate dielectrics such as pulverized or granular materials. In understanding the nature of the density dependence of the dielectric properties of particulate materials, relationships between the dielectric properties of solid materials and those of air-particle mixtures, such as granular or pulverized samples of such solids, are useful. In some instances, the dielectric properties of a solid may be needed when particulate samples are the only available form of the material. This was true for cereal grains, where kernels were too small for the dielectric sample holders used for measurements (Nelson and You, 1990) and in the case of pure minerals that had to be pulverized for purification. For some materials, fabrication of samples to exact dimensions required for dielectric properties measurement is difficult, and measurements on pulverized materials are more easily performed. In such instances, proven relationships for converting dielectric properties of particulate samples to those for the solid material are important. Several well-known dielectric mixture equations have been considered for this purpose (Nelson and You, 1990).

The notation used below applies to two-component mixtures, where ε represents the effective permittivity of the mixture, ε_1 is the permittivity of the medium in which particles of permittivity ε_2 are dispersed, and ω_1 and ω_2 are the volume fractions of the respective components, where $\omega_1 + \omega_2 = 1$. Two of the mixture equations found particularly useful for cereal grains were the Complex Refractive Index mixture equation (Nelson, 1992):

$$(\varepsilon)^{1/2} = \omega_1(\varepsilon_1)^{1/2} + \omega_2(\varepsilon_2)^{1/2} \tag{2.8}$$

And the Landau and Lifshitz, Looyenga equation (Nelson, 1992):

$$(\varepsilon)^{1/3} = \omega_1(\varepsilon_1)^{1/3} + \omega_2(\varepsilon_2)^{1/3} \tag{2.9}$$

To use these standard equations to determine ε_2, one needs to know the dielectric properties (permittivity) of the pulverized sample at its bulk density (air-particle mixture density), and the specific gravity or density of the solid material. Nelson (1992) has reported that the Complex Refractive Index and Landau Lifshitz, Looyenga (Nelson, 1992)

relationships provided a relatively reliable method for adjusting the dielectric properties of granular and powdered materials with characteristics like grain products from known values at one bulk density to corresponding values for a different bulk density.

The differences due to bulk density follow expectations due to differences in the air-occupied volume. Nelson et al. (1991) reported that the dielectric properties of the solid material of various hydro-colloids were greater than of the powdered materials. Although many researchers are of the opinion that there is no theoretical basis for a pure particle size effect, studies on instant coffee and milk powder implied that such an effect is possible (Kress-Rogers and Kent, 1987). Measurements on various sieve fractions of powdered or crushed grains obtained by Venkatesh et al. (1998) also indicated the possibility of a particle size effect. Essentially, when a material is crushed or powdered, one may expect changes in the surface characteristics, and it is therefore possible that the proportion of energy transmitted changes. This would be reflected by a change in the dielectric constant, without necessarily a change in the loss factor. However, Kress-Rogers and Kent (1987) do not agree with this explanation. One of the problems in concluding particle size effects is to ensure that the moisture content and compositions of the various particle size fractions are the same and that there are no changes due to heating during grinding. If there is a "pure" particle size effect, it appears to be so small compared to the influence of bulk density and other factors, that only very thorough and precise experimentation will prove or disprove its existence.

2.5 Applications of Dielectric Properties Measurements

As mentioned in the introduction, the dielectric properties of materials depend on many factors, including some that are related to chemical composition. Once fundamental data on the relationships between the dielectric properties and other factors have been established, the rapidity with which the dielectric properties can be measured, and the non-destructiveness of the methods, can lead to better methods of quality analysis or monitoring of relevant properties or states, before, during or after processing. There are many examples of such applications in the literature of which only a few have been listed below.

2.5.1 Quality of agri-food material

These properties and the quality of a substance can be correlated. For example either high frequency or lower frequencies can be used to measure the quality of fish and meat (Kent & Kress-Rogers, 1987).

2.5.1.1 Edible oils / fats

Consumption of oil and fat based foods have rapidly increased in recent years. To guarantee an effective quality control for used frying fats, simple and rapid methods for the measurement of thermal abuse are needed. Some of the tests that are reported include heating several frying oils with and without food stuff and estimating the change of polar parts, acid number, colour, specific absorption and dielectric properties with prolonged heating time. However, it is reported that these routine analyses may not always be sufficient to characterize heated frying oils and fats. In recent reports, food-oil sensors (Hein et al. 1998, U.S and German patented) have been shown to be useful in determining heat abuse for frying fats and oils. So far, there is neither much information nor simple techniques that are available to understand the deteriorating nature of the heated edible oils which are ubiquitous in any food processing industry or operation. Consumers and food catering units continue to use and reuse the frying oils to meet everyday demands.

The dielectric constant was found to be the most significant indicator for quality control in commercial deep fat frying operations and it was also concluded that polymer content and changes in dielectric constant are useful for monitoring frying oil quality (Paul et al, 1996). It is reported that the measurements of the dielectric properties of edible oils were compared to conventional methods of analysis (viscosity, refractive index, iodine value, peroxide value & the fatty acids) for evaluating the frying quality of a blend of cotton seed and sunflower oils and also for predicting deterioration occurring during heating of the oil, as a large portion of edible oil is consumed in frying of foods (El-Shami et al. 1992). For fats and oils, both ε' and ε'' are very low. For some oils, tallow and lard, ε' is between 2.4 and 2.7 and physical state is not important for the values; however, it is more important for the ε'' values. These values vary from 0.035 (lard -10°C) to 0.172 (corn oil +60°C, impure) at 2.8 GHz. The ε'' increases with temperature and with transition from solid to liquid phase.

Venkatesh et al. (2000) reported the summaries of various recent studies related

to heated edible oils and their characteristics in an effort to establish comparative standards used in deep frying operations in routine food services and processing scenarios. Conventional analytical and chemical techniques have been identified and compared with recent methods including Microwave based sensors and instrumentation. The Food Oil Sensor (FOS) is reported to be the most useful and helpful tool to investigate the characteristics of oil / fats and to assess their quality for monitoring purposes. Since the technology is patented in Germany and the USA, the details of the design and development of FOS is difficult to access in reported literature. It is identified that edible oils and fats are thermally altered during frying operations and whether or not there will be residual effect on human consumption is yet to be fully justified. One has to understand the interaction mechanisms of oil / fat molecules subjected to MW radiation at broad range of approved frequencies and temperature ranges. The advent of MW-chemistry techniques will answer many issues related to quality sensing of used heated edible oils. On the other hand, dielectric properties measurement and its simplicity in analysis needs more research as well. This research study has attempted to integrate the need for chemical analyses and microwave techniques to address this challenging task of assessing quality of the most sensory and culinary element of human food and diet.

2.5.2 Water quality detection

Raveendranath et al.,(1995) have adapted the microwave technique to study and assess water quality aspects. They suggested that the dielectric behavior of artificially polluted water and polluted water collected from various industrial locations, could be related in the use of an effective technique for detecting the pollutants in water at microwave frequencies (2.685 GHz) based on the measurement of complex permittivity of polluted water at 27°C. This may be useful for the study of oil - water mixtures for both food and soil applications using microwaves.

2.5.3 Dielectric behavior of agri-food products

Dielectric properties vary considerably among different kinds of grain, crop and weed seed. In general, ε' and ε'' are greater for grain and seed samples of higher bulk densities and higher equilibrium moisture contents. The main interest in the dielectric properties of agricultural products is for the determination of moisture content.

Mathematical models for the ε' vs. ρ of cereal grains have been developed (Chugh et al., 1973; Nelson, 1973, 1987). Recent studies (Trabelsi et al. 1997) have indicated that measurement of dielectric properties has potential in estimating engineering properties such as mass, density, thickness, fat content, etc.

2.5.3.1 Baked food products and Flours

Microwave and radio-frequency heating have also been used for baking processes. Microwave permittivities of bread dough were measured over the frequency range from 600 MHz to 2.4 GHz by Zuercher et al., (1990) as a function of water-flour composition, proofing time, and baking time. The dielectric constant and the loss factor both decreased as the water content and baking time was reduced. Permittivity of cracker dough, starch and gluten were measured over the 0.2 to 20 GHz frequency range by Haynes and Locke (1995) who also studied the dielectric relaxation in this frequency range. They identified two relaxation regions, one for doughs below 35% moisture associated with bound water, and another for moisture contents above the level associated with free water. The dielectric constant and loss factor of baked dough, as a function of temperature and moisture, were reported at 27 MHz. The dielectric constant gradually increased with moisture content and temperature. Temperature also affected the dielectric loss factor beyond this point. The ionic conductivity and the bound water relaxation are considered the dominant loss mechanisms in the baked dough at this frequency. Seras et al. (1987) measured the permittivity during heating of different flours with varying water contents. The increase in ε' before reaching 100°C, for dry flour, or with 10% water content, shows that the water interacts with the polysaccharide matrix and thus is unfreezable. When water evaporates, ε' decreases to a stable level in a dry product. Dielectric properties for bread and flour have been measured mostly at high frequencies. Although ε" varies with moisture, as can be expected, temperature has only a minor effect.

2.5.3.2 Dairy Products

Dielectric properties of dairy products are relatively scarce. Properties of whey and skim milk powders have been measured by Rzepecka and Pereira (1974). Those of aqueous solutions of nonfat dried milk were modeled by Mudgett et al.,(1971, 1974). Representative dielectric properties of milk and its constituents at 2.45 GHz are shown in

Table 2.4 from the work of Kudra et al.,(1992). For butter, measurements between 30 Hz and 5 MHz were reported by Sone et al. (1970). The dielectric constant and loss factor of butter at 2.45 GHz as functions of temperature were measured by Rzepecka and Pereira (1974). Their results revealed that the permittivity of bound water at 2.45 GHz had a positive temperature coefficient, while the permittivity of free water decreased with temperature. At temperatures below the freezing point, free water crystallizes, and its permittivity decreased rapidly to very small values, so the behavior appears to result from the influence of bound water. The rapid increase in the permittivity of butter above 30°C may be caused by the disintegration of the emulsion. Over a small range of moisture content, Prakash and Armstrong (1970) showed a linear increase in dielectric constant with moisture content. Seasonal variation in dielectric properties of butter showed no significant changes (O'Connor et al., 1982).

To et al.(1974) used aqueous nonfat dry milk solutions as model systems & found that the milk salts do not dissociate completely. Bound salts (about 33% of milk fats) do not exhibit the same dielectric behavior as freely dissociated salts in solution. The dielectric properties of milk, based on the total ash content, are lower than expected from the chemical composition alone.

2.5.3.3 Fish, Seafood and Meat

Dielectric properties of codfish were measured at frequencies from 10 to 200 MHz at temperatures from -25 to 10°C (Bengtsson et al., 1963). An abrupt change in the dielectric constant was noted in the region of freezing temperatures which became slightly more gradual as frequency decreased. At temperatures below the freezing point, dielectric constants increased slightly from values below 10 to values between 10 and 20 before the abrupt increase, on thawing, to values between 60 to 90, depending on the frequency. At 10 GHz, dielectric properties of fish meal were studied by Kent (1970, 1972) as functions of the temperature and moisture content. Both the dielectric constant and loss factor increased non-linearly with moisture content. They also increased with temperature in a relatively linear fashion. Kent also examined the complex permittivity of fish meal in relation to changes in temperature, density, and moisture content (Kent, 1977), and concluded that at high moisture contents and temperatures, the dependence of the permittivity on density was similar in nature to its dependence on temperature and moisture content.

Kent and Anderson (1996) have reported the ability to distinguish differences in scallops from those soaked in water and also those soaked in polyphosphate solutions by a microwave spectra of the dielectric properties taken between 200 MHz and 12 GHz with data subsequently subjected to principal component analysis.

Zheng et al.,(1998) recently reported dielectric properties at 915 MHz and 2450 MHz on raw non-marinated and marinated catfish and shrimp at temperatures from about 10 to 90°C. Measurements showed that marination increased both the dielectric constant and loss factor. The dielectric constant generally decreased with increasing temperature whereas the loss factor increased with temperature. The dielectric properties of tuna fish at 2450 MHz and 915 MHz and from -30 to 60°C is reported elsewhere. The dielectric constant and loss factor varied with the composition of a substance and the temperature. The values of dielectric properties were small at temperatures below the freezing point. The sharp increase in dielectric properties was observed around the freezing point. The dielectric constant and loss factor increased with increased water content at constant temperature; the dielectric constant and loss factor of lean tuna were larger than those of fatty tuna.

Ohlsson et al.,(1974) studied meat emulsions at 900 and 2800 MHz and concluded that correlation with dielectric data for fat and protein content was complex. Bengtsson and Risman (1971) reported dielectric properties data for a large variety of foods, measured at 2.8 GHz with a resonant cavity as a function of temperature, including both raw and cooked meats. In general, both the dielectric constant and loss factor increased with increasing moisture content. When temperature increased through the freezing point, sharp increases were noted in both the dielectric constant and loss factor. They found no significant differences in dielectric properties between ground and whole meat. Although moisture content is dominant, both ash and protein content can affect meat dielectric properties. For beef products, both ε' and ε'' increase with falling frequency at constant temperature; however, ε' decreases and ε'' increases with increasing temperature at constant frequency. The ε'' of turkey is higher than that of beef, which is mainly due to moisture content. Both ε' and ε'' are small for frozen meat, under 10 and 2, respectively. Both the permittivity and loss factor for cooked beef are about 60% of those for raw beef (Kent, 1987).

When dielectric properties data for many different types of foods (meats, fruits, and

vegetables) are considered together, they showed very little correlation with composition (Sun et al. 1995). This was attributed to variability in sample composition and measurement errors, and the general unavailability of detailed composition data. To et al., (1974) reported statistical correlations of data at 2450 MHz from a restricted set containing about 25 data points for raw beef, beef juice, raw turkey, and turkey juice, with composition data taken from the USDA Handbook (USDA, 1976). Their study revealed significant relationships between both the dielectric constant and loss factor and the water and ash contents and temperature (Sun et al., 1995). The addition of terms for components such as protein, carbohydrates, and fat, did not improve the correlation significantly. Also, de Loor and Meijboom (1966) found that high water-content foods, such as potato, agar gels, and milk, had similar relaxation times, which they attributed to the availability of free water in the foods. Thus, since the moisture content of all the meats and meat products used in the correlation (Nelson and Datta, 1999) was greater than 60%, the free water should be the dominant component governing the overall dielectric behavior of these foods.

The ash content was taken to be a good indicator of the total salts in these foods. Increased ash content served to elevate the dielectric constant in the foods, which agreed with the experimental data reported by Bengtsson and Risman (1971), in contrast to the behavior of aqueous salt solutions which exhibit a reduction in the dielectric constant with increasing salt concentration. In the range of temperatures studied (Sun et al., 1995) the ash content elevated the dielectric loss, indicating that increased salt adds conductive charge carriers that increase the loss of the system as a result of charge migration.

The dielectric loss of aqueous solutions at microwave frequencies may be divided into effects of dipole rotation and ionic charge migration. As discussed by Mudgett (1980), the dipole loss component decreases as temperature increases, while the ionic component increases with temperature. Furthermore, if the ash content in the food is sufficiently high, the ionic component may dominate the dipole loss, resulting in an increase in the dielectric loss with temperature. Results of Sun et al.,(1995) showed that for salt contents greater than 2%, the predicted dielectric loss increased with temperature, which is in accord with the behavior of aqueous salt solutions. As discussed by Ohlsson et al.,(1974), the high temperature dependence of the ionic conductivity explains the rise in the dielectric loss with temperature.

2.5.3.4 Water mixtures of sugars and starches

Kent and Kress- Rogers (1987) measured the dielectric properties of sucrose, glucose and syrup. Complex permittivity varied with frequency and concentration. The values measured at 2.8 GHz differed greatly from those at 3.05 GHz. Roebuck et al. (1972) have studied the dielectric properties of potato starch, sucrose, glucose, ethanol and glycerol, at 1 and 3 GHz and 25°C. At intermediate water concentration, gelatinized potato starch has higher ε' and ε" values than starch in granular form. Carbohydrates do not show appreciable dipole polarization at microwave frequencies. Exclusion of water by the carbohydrate is, therefore, important for the dielectric properties of a carbohydrate-water mixture. Mladek and Komarek (1974) studied potato and wheat starches containing up to 40% water. At 10 GHz, the ε' of potato starch is higher than that of wheat starch, however the ε" of potato starch is lower. The losses are due to relaxation of hydroxyl groups in a dry starch molecule. The dielectric properties of chemically modified and some unmodified starches have been studied by Miller et al. (1991). The ε" for most starches generally decreased during heating, however it remained constant within the temperature range associated with the thermal transition of starch. The type of chemical modification seems to have a strong influence on the dielectric behaviour.

2.5.3.5 Salt Solutions

Many agricultural and food materials contain water as the major constituent. Also, salt ions can affect the dielectric properties significantly, especially the dielectric loss factor. Thus, it is useful to study the dielectric properties of salt solutions (Sun et al.,1995, Hasted et al.,1948) which are relatively simple systems and they may suggest trends in the dielectric behaviour of some food materials. The dielectric properties of saline solutions have been studied in detail and models for calculating these properties as functions of temperature and salinity have been developed and tested against experimental data from the reported literature. The dielectric constants of the salt solutions decreased as temperature rised. However, the sign of the temperature coefficient of the dielectric loss factor depends on the concentration for the range of temperatures selected.

2.5.3.6 Broth, gravy, soup and salad cream

The temperature dependence of broth, gravy and soup seems to be similar to that for water and other foods (Bengtsson & Risman, 1971). Laursen (1987) found that the permittivity curves for salad cream changed considerably during extended storage at room temperature; the droplet size had no effect.

2.5.3.7 Water Activity (a_w)

The "mobility" of water within the structure of a host material may vary widely - from highly mobile "free" water, to "bound" water that is somehow kinetically constrained. Mobility is linked to water activity, which describes water's contribution to the support of bacteria and fungi. Quantifying water activity is key to research on food preservation and safety. The dielectric measurements are a unique research tool in understanding aqueous solutions, water activity, food preservation, shelf life, hydration phenomena, and phase transitions. Properly designed and calibrated electrical instruments are used to quickly determine the moisture content.

2.5.3.8 Food dielectric properties at freezing and sterilizing temperatures

Accurate knowledge of dielectric properties in partially frozen material is critical to determining the rates and uniformity of heating in microwave thawing. As the ice in the material melts, absorption of microwave energy increases tremendously. Thus, the portions of material that thaw first, absorb significantly more energy and heat at increasing rates that can lead to localized boiling temperatures while other areas are still frozen. Dielectric properties of frozen food materials have been reported in the literature (Bengtsson et al.,1963, 1971; Kent et al., 1975) and these properties can be heavily influenced by composition, particularly total water and salt content. Salt affects the situation through freezing point depression, leaving more water unfrozen at a given temperature in this range. Salt also increases the ionic content and consequently the interaction with the microwave fields.

Chamchong (1997) reported dielectric properties at 2450 MHz of Tylose™, a food analog, covering the frozen range. For complex and formulated foods such as Tylose™, the dielectric properties must be measured or estimated (Ohlsson, 1989; Buffler & Stanford, 1991, Venkatesh, 1996). Both the dielectric constant and loss factor decreased

significantly as more water was frozen. Since the fraction of unfrozen water is a nonlinear function of temperature, the increase in dielectric properties of the partially frozen material is also nonlinear with temperature. Above the freezing range, the dielectric constant of tylose decreased linearly with temperature. With the addition of salt, the dielectric constant decreased while the dielectric loss factor increased.

Dielectric properties at high temperatures, useful for microwave pasteurization and sterilization, have been scarce (Nelson and Datta, 1999). In addition to temperature effects *per se*, physical and chemical changes such as gelatinization of starch (Miller et al., 1991) and denaturation of protein causing release of water and shrinkage (Li and Barringer, 1997) at higher temperatures can significantly change dielectric properties. Several authors have published equations to predict the dielectric properties as a function of water, salt and temperature. These equations work best for foods where a large number of related samples have been reported in the literature. For foods where little has been published, the accuracy of the models decreases, in some cases dramatically.

The dielectric properties of simple systems such as water or salt water are linear with respect to temperature. Liquid foods, such as broth and gravy, are also typically linear with temperature. More complex food systems, such as meats, are not linear. One reason may be due to phase changes, such as protein denaturation and starch gelatinization. Protein denaturation changes the water and salt binding capacity of the food, expels water, and changes the dielectric properties. For high-salt products such as ham, the change in dielectric properties at the denaturation temperature is strongly a function of salt content (Barringger, 2000).

The dielectric properties of several powdered hydrocolloids, including potato starch, locust bean gum, gum arabic, carrageenan and carboxymethylcellulose, were measured at 2.45 GHz as functions of temperature (20 to 100°C) and moisture content between 0% and 30%, wet basis (Prakash et al., 1992, Nelson et al., 1991). The dielectric constants and loss factors of all hydrocolloids increased with moisture content and with temperature indicating a relaxation frequency below 2.45 GHz at all moisture contents and temperatures. The degree of temperature dependence of all five hydrocolloids increased as moisture content increased but to a lesser extent in potato starch and a much lesser extent in locust bean gum.

2.5.3.9 Dielectric properties of nuts

Dielectric properties of nuts are quite limited. Dielectric constants and loss factors for peanuts have been reported for the 1 to 50 MHz frequency range (Nelson, 1973,1981) showing the expected variation with frequency and moisture content. More detailed information is available for chopped pecans (Nelson, 1981), and these data are reported for the frequency range from 50 KHz to 12 GHz over the moisture range from 3 to 9%, wet basis, at 22°C. The temperature dependence at 0 to 40°C of the dielectric constant and loss factor were determined for the frequency range from 100 KHz to 110 MHz over a similar range of moisture contents (Lawrence et al., 1992, 1998). Both the dielectric constant and loss factor increased regularly with moisture content at all frequencies and decreased as frequency increased. At low moisture contents, the temperature dependence was minimal, but both the dielectric constant and loss factor increased rapidly with temperature at high moisture levels.

2.6 Dielectric behavior of soils

Important factors in the behaviour of soils exposed to microwaves are the dielectric properties of the components of the soil. Only limited information is available on these properties in the literature, particularly at microwave frequencies. Hoekstra and Delaney (1974) studied the dielectric properties of a fine sand, a silt, a silty clay and a clay soil (f =100 to 26000 MHz). Their data revealed the following general relationships: ε' increase with an increase in temperature, but the ε'' remains fairly constant as temperature increases; the ε'' goes through a relaxation point; as temperature decreases the frequency (at which relaxation occurs) increases; both ε' & ε'' increase with an increase in water content; the frequency at which relaxation occurs increases with a decrease in water content; and ε' decreases with frequency and decreases more sharply after the relaxation frequency. In general, the ε' of soils varies from 2 to 3 for dry soils to up to 20 for soils with a moisture in the range of 0.3 g/cm^3. The ε'' vary from 0.5 for dry soils to approximately 4 for high moisture soils. Soils with concentrated organic compounds have higher electrical conductivities than soils permeated with the same amount of water. As well, if the compound has a low ε' and is present in a high concentration, clay particles will tend to flocculate.

2.6.1 Dielectric Behavior of Organic Solvent Mixtures

Very little information is available on the dielectric properties of solvents and solvent mixtures at microwave frequencies. Knowledge of these properties is required to understand how soil/solvent mixtures behave when exposed to microwaves. The testing of solvent mixtures is essential as it is found necessary to use a mixture of solvents in compounds. Certain selected solvents and their mixtures have been tested in this study.

2.6.2 MAP™

The Microwave-Assisted Process (MAP™) is an enhanced extraction technology patented by Environment Canada that uses microwaves to rapidly transfer target compounds from one phase to another by selectively heating the phase containing the target compounds (Pare et al., 1994). The MAP™ technologies use microwaves to assist in physical or chemical processes. The use of this technology for the solvent extraction of organic contaminants from soil has proven successful in the area of analytical sample preparation and has penetrated various areas of research including quality control; pharmaceutical, food products, food safety, nutritional verification, environmental sample separation; contaminated soil, contaminated water, waste streams, contaminated animal tissues, pesticide residues in plants and in forensic sciences - drugs in tissues.

2.7 Summary of Industrial, Scientific, Medical and Domestic (ISM & D) Applications

2.7.1 Agriculture

There have been relatively few recent attempts to apply microwave power to agriculture; more investigations were carried out in late 1960s and early 1970s (Person, 1972). The areas of application include drying of grains (Bhartia et al., 1968, Copson, 1962, Fanslow and Saul 1971, Rzepecka et al. 1972a, Wesley et al. 1974, insect control (Nelson, 1972; Nelson and Stetson, 1974), and seed germination (Jolly and Tate, 1971; Nelson, 1976a; Nelson et al., 1976). Such exotic uses of microwave energy as protection of plants form cold were also suggested (Bosisio et al., 1970; Bosisio and Barthakur, 1973). The main limitation of these applications is economics. Insect control and seed germination are more likely candidates for economic use of microwave power than grain drying.

Insect control is achieved by heating the insects for a sufficient period to a

sufficiently high temperature, preferably without heating the host material. The difference in the heating rate of the insects and grain depends on the dielectric properties of the two and their size and shape (Nelson, 1972; Nelson and Stetson, 1974). The dielectric properties of various grains were measured in a broad range of frequencies, temperatures, and typical moisture contents Chugh et al., 1972; Nelson, 1976b, 1977; Nelson and Stetson, 1975). Similarly, the permittivity of a few typical insects was investigated (Nelson, 1976b).

Seeds of many legumes present a germination problem owing to a large number of hard seeds. While these seeds are viable, an impermeable seed coat prevents the entry of moisture necessary to initiate gemination, and consequently the seeds germinate and grow late and may not have sufficient time to mature by the harvest time. Such seeds, when heated to an appropriate temperature, show greatly improved germination without any other undesirable side effects. Extensive studies were conducted of alfalfa seed germination after treatment at various frequencies (Nelson et al., 1976a). Also, germination of clover, acacia, Douglas fir, pine and spruce were investigated (Tran and Cavanagh, 1979).

2.7.2 Food

Application of microwave power to numerous food processes has been investigated on a laboratory scale, and a few successful industrial processes are presently in operation (Bengtsson and Ohlsson, 1974; Friedmann, 1972, 1973; Thourel,1979). The processes studied include drying (Anon, 1972; Maurer et al., 1971; Rzepecka-Stuchly, 1976; Rzepecka et al., 1976; Sobiech, 1980; Suzuki and Oshina, 1973), freeze-drying (Decareau, 1982; Ma and Peltre, 1975; Sunderland,1982), preheating, thawing (Bialod, 1978; Phan, 1977; Priou et al., 1978), sterilisation (Decareau, 1982; Jaynes, 1975; Kenyon et al., 1971; Lin and Li, 1971), enzyme inactivation (Aref, 1972; Goldblith et al., 1969), meat tempering (Meisel, 1973; Schiffmann, 1973, 1976), blanching (Avisse and Varoguaux, 1977; Chen et al.,1971a, 1971b), and cooking (Decareau, 1982; Goldblith, 1975; May, 1969; Nykvist and Decareau, 1976; Suzuki and Oshima, 1973).

In the baking industry one of the most successful applications developed on a commercial scale is the microwave doughnut proofing. The systems developed by DCA Food Industries, Inc. in the USA operates at 2.45 GHz, with an output power between 2.5

and 10 kW. Another microwave application in baking, also developed by the same company (DCA), was the doughnut fryer (Moyer, 1973; Schiffmann, 1973).

A highly successful system for meat tempering was developed by Raytheon Company (USA) (Schiffmann, 1973). One of the most important factors in tempering is to ensure that the product enters the microwave tunnel at a uniform temperature without any incipient thawing. Thawing is understandable in view of the difference in the dielectric properties of frozen foods and foods at temperatures close to 0°C. The high loss factor of the thawed part of the food is responsible for overheating and thermal runaway. The meat-tempering system operates at 915 MHz.

Drying of pasta products is another example of successful commercial-scale application of microwave power. The system developed by the Microdry Corporation (USA) operated at 915 MHz with a power of 30 or 60 k W (Schiffmann, 1976).

The growth of applications of microwave power in food industry in Europe has paralleled, if not outrun, that in the United States (Meisel, 1973). Tempering tunnels developed by LMI in France are used for beef, lamb and ham. They operate at 2.45 GHz with an output power of 2.5 kW or 5 kW.

Two interesting systems were developed by Thomson at CSF (France) and Nestle Company (Switzerland), and Thomson and Japanese companies (Nittan Foods Co., and Shimada Raka Kogyo Co.) (Meisel, 1973). The microwave power of 5 kW is supplied at 2.45 GHz. Various systems are in use operating at 915 MHz and 2.45 GHz with an output power ranging from 1.4 kW to 30 kW (Kase,1973; Ogura and Kase, 1978). Puffing and drying of snack foods is another popular application, and some products have been developed specifically for microwave processing, e.g. puffed rice cake and seaweeds. In China, microwave power is used to dry chocolate powder and milk cake, and to age wine and spirits (Chen et al.,1982).

2.7.3 Paper and Textiles - industrial application

Microwave power has been used in processing of paper and textiles in several ways including moisture leveling or finish drying (Chen et al., 1982; Galeano, 1971; Jones et al., 1974; Jones and Lawton, 1974;Williams, 1966), rapid drying of print (Anon, 1968; Moore,1968; VanKoughnett and Wyslouzil, 1972), drying of glue on paper (Cumming and Bleackley, 1967), polymer sturation (Minami and Branion, 1972; Takahashi et al., 1969),

and drying of various coatings.

2.7.4 **Aspects on Scientific Applications** - biological

2.7.4.1 **Power**

Microwave power is used to generate and heat plasma. A relatively large number of studies has been carried out in this field (Bosisio et al., 1972, 1973; Johnston, 1970; Moisanet al., 1979). Rapid microwave heating aids or even enables chemical reactions.

In neuro-chemical studies microwave power is used to rapidly fix the brain of living animals (Butcher et al., 1976; Kant et al., 1979; Lenox et al., 1976; Merritt and Frazer, 1977; Schmidt et al., 1972; Sharp-less and Brown, 1978). Special applicators have been developed to apply the energy effectively to the desired brain volume. The microwave-fixation technique permits the measurement of neurochemical parameters unobtainable by other methods (Merritt and Frazer, 1977). Microwave energy was also used for the in-situ fixation of cells grown in tissue culture (Patterson and Bulard, 1980), in rapid processing of the vitreous carbon-polymethacrylate implant (Hodosh et al., 1978).

2.7.4.2 **Measurements** - emerging fields

Microwaves have been extensively used in investigating molecular structure of materials in dielectroscopy (measurements of the permittivity) and in spectroscopy. Both of these fields are very sophisticated and highly developed, and detailed description is outside the scope of the review. From the measurements of the permittivity of liquid and solid materials, including biological materials, as a function of frequency in the microwave range, and temperature, it is possible to determine some features of the molecular and cellular structure. Microwave spectroscopy is also used in investigating molecular structure of gaseous media. This technique can also be utilised in industry; for instance, for air-pollution control (Schiek et al., 1977). Plasma can not only be generated with the aid of microwave energy, but low-intensity microwaves are used in diagnosis of the state of plasma (Johnston, 1970). Another interesting application is in a study of laser-production blast waves (Hall, 1969).

2.7.4.3 **Monitoring properties of materials; nondestructive testing**

Since dielectric properties depend on properties other than water content; these

other properties can also be monitored by measuring the dielectric properties at microwave frequencies. The organic content (e.g. macromolecule kerogen) of oil shale was measured by Judzis et al. (1977). The void fraction in organic coolants used in nuclear reactors was continuously monitored in an open-ended cavity similar to that used for measuring bound water (Stuchly et al., 1974). A linear dependence of the resonant frequency as a function of void fraction was obtained for a sensor operating at about 1.5 GHz. The sensor was designed to be incorporated into the cooling system. Changes in the dielectric properties were also used to measure the density of cryogenic fluids (Ellerbruch, 1970), to monitor concrete curing (Rzepecka et al., 1972) and to measure the fibre orientation in paper (Tiuri and Liimatainen, 1975). A pulsed-radar technique was utilised to detect oil on water (Klemas, 1972), and the microwave ferromagnetic resonance was used to find flaws in metals (Anon, 1978).

2.7.4.4 Safety aspects of I S M & D applications

From the numerous devices developed for industrial, scientific, medial and domestic use, emissions from microwave ovens, both industrial and domestic, are regulated in most of the Western countries. It has been established that exposure to the operator from a leaking oven is extremely low. Surveys of the ovens in the USA and Canada (Stuchly et al., 1979) indicated compliance with the regulations and leakage well below the limit for many models. A large number of simple and inexpensive devices have been developed for detecting leakage from microwave ovens (Bojsza, 1980; Voss and Turner, 1982).

III DIELECTRIC PROPERTIES MEASURING TECHNIQUES
- an Overview of Usefulness, Applications, Instrumentation and Development

3.1 Background

Dielectric parameters of various agri-foods and biological materials are finding increasing application as fast and new technology is adapted for use in their respective industries and research laboratories. Dielectric properties and measurement studies dates back to more than 80 years. Earlier concept of permittivity measurements based on dc electrical resistance to determine grain moisture content. Non-linear increase in resistance of the grain as temperature increased, gave useful observation. However, no quantitative data were reported. Later on, ac measurements were commonly employed to measure the change in capacitance and suitable sample holding capacitors were developed. Grain moisture measurement based on dielectric properties data became the most prominent agricultural application. Newer instruments and their calibration led to the development of a Standard -oven technique which further contributed to several applications of radio-frequency dielectric heating and supplemented the quest for more quantitative values. First moisture meter was designed and developed in U.S.S.R. for barley and wheat moisture measurement. In recent times, the concept of permittivity measurement has extended to various agricultural, food and biosystems problems. Research and development in this area need to be intensified.

With a need for development of improved sensing devices for the control and automation of several agricultural, environmental and food processes, there is an absolute need for better understanding of the dielectric properties of materials and techniques for measuring these parameters. Measurement of the bulk dielectric properties (ε', ε'') is not an end unto itself. Rather, these parameters are an intermediary vehicle for understanding, explaining and empirically relating certain physico-chemical properties of the test material. Therefore, in this study, an attempt is made to further the knowledge of dielectric properties (complex permittivity), their role and importance in agri-food sectors, and concept of various measurement techniques and development are briefly summarized. An extensive review of literature on measuring techniques, comparison and potential application of dielectric properties is reported and the readers are advised to follow the appropriate literature cited for detailed and complete reference.

3.2 Literature on Measurements - *an overview*

Early measurements of food dielectric properties were made by Dunlap and Makower (1945) for carrots at frequencies in the range of 18 KHz to 5 MHz. The dielectric constant and conductivity were reported to depend largely on moisture content as influenced by frequency, temperature, density, and particle size. The dielectric constant was essentially constant at moisture contents up to 6-8% and increased rapidly at higher moistures; similar behavior was seen for measured conductivities. Their results suggested that higher frequencies were most suitable for moisture determinations in food products. Dielectric properties of potato, carrot, apple, and peach tissue were measured by Shaw and Galvin (1949) at frequencies form 1kHz to 40 MHz. Their measurements showed a general region of dispersion between 100 kHz and 20 MHz and provided some useful data on the temperature dependence of conductivity in fruits and vegetables. Dielectric properties of raw potato at frequencies from 300 to 3000 MHz dropped appreciably with increasing frequency (Pace et al., 1968b). The dielectric properties of apples (Thompson and Zachariah, 1971) at frequencies of 300 to 900 MHz were found to vary with maturity, dropping appreciably in the process of aging.

Morse and Revercomb (1947) measured the dielectric constant and loss of meats and vegetables at temperatures above and below the freezing point and found large differences in the properties of frozen and unfrozen samples. Thawed portions of processed samples also showed "runaway" heating effects, resulting from selective energy absorption by unfrozen fluids; unevenness in thawing was also reported by Brown et al., (1947). Harper et al., (1962) measured dielectric properties for a variety of products at 500 MHz and 2 GHz in connection with freeze -drying studies. Values obtained for peaches, pears, beef steak, and beef fat showed that loss factors decreased as frequency increased or as temperature decreased. Measurements of various meats and fish, including raw beef, pork, beef and pork fat, codfish, and herring, were made by Bengtsson et al. (1963) at radio frequencies from 10 to 100 MHz. They found large differences in the dielectric properties of frozen and unfrozen samples and significant differences between samples with fibers oriented perpendicular of parallel to the field, i.e., anisotropic behavior. The dielectric properties of raw potato, potato starch, and milk were measured by de Loor and Meijboom (1966) at microwave frequencies form 1.2 to 18 GHz.

The effect of moisture content on the dielectric properties of granular solids was

studied at 9.4 GHz by Stuchly (1970) over a wide range of temperature and moisture contents. Temperature dependence was not seen for dried solids but increased dramatically at higher moisture contents. Similar behavior as a function of moisture has been reported by many other researchers.

Goldblith and Pace (1967) considered the potential for microwave finish drying of potato chips and found that energy absorption at 1000 and 3000 MHz increased at higher moisture contents and temperatures. In connection with this study, Pace et al., (1968a) measured the dielectric properties of 11 commercial fats and oils at frequencies from 300 MHz at 3 GHz and at varying frying temperatures. Little variation in dielectric behavior was seen for these measurements. One of the oils was measured over an extended frequency range form 100 Hz to 80 GHz and was found to have a region of dispersion (rapid variation in dielectric behavior with frequency) from about 19 MHz to 1 GHz. The dielectric properties of reconstituted ground beef were measured at 915 MHz by van Dyke et al.,(1969) to study the influence of moisture, ash, and fat contents. Moisture contents below 20% showed little variation in dielectric loss. Losses increased sharply with an increase in moisture form 20% to 45% and then more slowly at higher moistures. Losses were also found to increase with ash content and to decrease with fat content. More recently, Bengtsson and Risam (1971) measured food properties at 2.8 GHz and temperatures from -20 to 60^{0}C. Foods included raw beef and pork, cooked beef and ham, fish, carrots, peas, mashed potatoes, gravy, and fats. Their results showed a wide variation in dielectric behavior due to differences in chemical composition, physical state, and temperature. It is not possible to estimate the effects of conductivity in these measurements, because ash contents of the samples were not reported. The effects of dissolved salts on dielectric loss in milk were also investigated in chemical simulation studies, which showed that predictions of milk loss based on conductivities implied by ash contents needed to be corrected for binding and nonbinding interactions of milk salts (Mudgett et al., 1971).

Abstracts on much of the work described above and on areas of related interest in microwave food processing are available in a bibliography on microwaves by Goldblith and Decareau (1973). During the next decade, food measurements providing a broad overview of dielectric behavior for liquid and semisolid products at frequencies and temperatures of interest in food processing were reported based on research performed at the Massachusetts Institute of Technology (Roebuck et al., 1972; Mudgett, 1974; Mudgett et

al.,1974a,b; To et al., 1974; Roebuck and Goldblith, 1975; Mudgett et al., 1977, 1979, 1980), the Swedish institute for Food Research (Ohlsson et al., 1974; Ohlsson and Bengtsson, 1975), and the U. S. Department of Agriculture (Nelson, 1973, 1980).

Although dielectric properties and their effects in food processing can now be predicted over a wide range of frequencies for many foods and processing conditions based on models, there still remain some interesting questions on the dielectric behavior of a number of polar and non-polar food constituents and their mechanisms of interaction with an electromagnetic field. A quantitative model for the coupling of electrical energy in foods by radiative transfer has not yet been found. The relationships between the dielectric properties of foods and the electrical characteristics of microwave applicator/generator with respect to mutual interactions between the loaded cavity and the generator during the course of dielectric heating or processing is not clearly understood or interpreted.

The mechanisms of interaction between complex polysaccharide such as starch, pectin, and cellulose with water and their effects on dielectric behavior and organoleptic quality and the basis for energy coupling and attenuation by high molecular weigh lipids (fats or oils) and food solids (colloidal proteins) at both microwave and sub-microwave frequencies are of particular interest. Dielectric measurements of foods at elevated temperatures and pressures would also be useful for developing predictive sterilization models, particularly for the design of high temperature short time (HTST) processes to optimize retention of nutrients. At microwave frequencies, dipole losses in high and intermediate moisture foods are dominant at low temperatures while ionic losses become increasingly dominant at higher temperatures, where penetration depths become increasingly shallow, or lower moisture, where penetration depths become increasingly large. While ionic losses and penetration depths are much greater at sub-microwave frequencies, dipole losses for high, intermediate, and low moisture foods are negligible at frequencies below 100 MHz.

3.3 Methods of Measurement of Dielectric Properties

The measurement of dielectric properties has gained importance because it can be used for non-destructive monitoring of specific properties of materials undergoing physical or chemical changes. There are several techniques to measure the dielectric properties of agri-food materials (Sucher and Fox 1963; de Loor and Meijboom 1966;

Bengtsson and Risman, 1971; Metaxas and Meredith, 1983). The dielectric properties of food materials in the microwave region can be determined by several methods using different microwave measuring sensors (Kraszewski, 1980). The particular method used depends on the frequency range of interest and the type of target material. The choices of measurement equipment and sample holder design depend upon the dielectric materials to be measured, the extent of the research, available equipment and resources for the studies. Vector Network Analyzers (VNA) are expensive but very versatile and useful if studies are extensive. Scalar network analyzers and impedance analyzers are relatively less expensive but still too expensive for many programs. For limited studies, more commonly available RF and microwave laboratory measurement equipment can suffice if suitable sample holders are constructed. Nyfors and Vainikainen (1989) gave four groups of measurement methods : lumped circuit, resonator, transmission line and free-space methods. The lumped circuit techniques are no longer used to any great extent since they were only suitable for low frequencies and high loss materials. The latter three and the recent open-ended coaxial probe (Hewlett-Packard, 1992) employ impedance, spectrum or network analyzers. Current developments are aimed at eliminating the need for these expensive yet versatile accessories (Nelson, 1991).

3.4 Measurement Principles and Techniques

The measurement methods relevant for any desired application depend on the nature of the dielectric material to be measured, both physically and electrically, the frequency of interest and the degree of accuracy required. Despite the fact that different kinds of instruments can be used, measuring instrument that provide reliable determinations of the required electrical parameters involving the unknown material in the frequency range of interest can be considered (Nelson, 1998). The challenge in making accurate permittivity or dielectric property measurements is in designing of the material sample holder for those measurements (RF and MW frequency ranges) and adequately modeling the circuit for reliable calculation of the permittivity from the electrical measurements. If one can estimate the radio-frequency (RF) circuit parameters appropriately, the impedance or admittance for example, the dielectric properties of that material at that particular frequency can be determined from equations that properly relate the way in which the permittivity of the material affects those circuit parameters.

Field (1954) has reviewed the techniques for permittivity measurements in the low, medium and high frequency ranges, including the use of several bridges and resonant circuits. Dielectric properties of grain samples were reported from measurements with a precision bridge for audio frequencies from 250 Hz to 20 kHz with sample holders confined in a coaxial sample holder (Corcoran et al., 1970). Attention must be paid to electrode polarization phenomena at low frequencies, which can invalidate measurement data.

Results of grain and seed samples tested using Q-meter based on resonant circuit have been documented in the range of 1- to 50-MHz range (Nelson, 1979a). Other techniques were designed and developed for higher frequency ranges with coaxial sample holders modeled as transmission-line sections with lumped parameters and measured with an RX- meter for the 50- to 250- MHz range (Jorgensen et al., 1970) and for the 200- to 500- MHz range, measured with an Admittance meter (Stetson and Nelson, 1970). Lawrence et al. (1998) have designed and modeled a coaxial sample holder to accommodate flowing grain and characterized by full two-port parameter measurements, with use of several organic solvents such as alcohols of known permittivities, and signal flow analysis, to offer dielectric properties of grain over a range of 25 to 350 MHz.

At microwave frequencies, generally about 1 GHz and higher, transmission-line, resonant cavity, and free-space techniques have been commonly used. Principles and techniques of permittivity measurements have been illustrated in several reviews (Westphal, 1954; Altschuler, 1963; Bussey, 1967). Dielectric property measurement techniques can be categorized as reflection or transmission types using resonant or non-resonant systems, with open or closed structures for sensing of the properties of material samples (Kraszewski, 1980). Waveguide and coaxial-line transmission measurements represent closed structures while the free-space transmission measurements and open-ended coaxial-line systems represent open-structure techniques respectively. Resonant structures can include either closed resonant cavities or open resonant structures operated as two-port devices for transmission measurements or as one-port devices for reflection measurements (Nelson, 1998).

In the earlier measurements by Roberts and von Hippel (1946), the standing wave ratios (SWR's) were required to measure in line with and without the sample inserted. Based on the shift of the standing-wave node and changes in the widths of nodes, related to SWR's, sample length, and waveguide dimensions, etc., ε' and ε'' can be computed with

suitable computer programs (Nelson, et al., 1974). Similarly, the complex reflection coefficient of the empty and loaded sample holder can be measured using a network analyzer or other instrumentation, where similar determinations can be made as discussed above.

Microwave dielectric properties of wheat and corn have been reported at several frequencies by free-space measurements with a network analyzer and dielectric sample holders with rectangular cross-sections between horn antennas and similar radiating elements (Trabelsi et al., 1997).The attenuation and phase shift are the two main components of the complex transmission coefficient, which permits the calculation of ε' and ε'' of the material under test. It is important that an attenuation of 10 dB through the sample layer be maintained to avoid disturbances resulting from multiple reflections between the sample and the antennas, and the sample size, laterally, must be sufficiently large to avoid problems caused by diffraction at the edges of the sample, for free-space measurements (Trabelsi et al., 1998).

For liquid and semi-solid materials including biological and food materials, open-ended coaxial-line probes have been used for broad-band permittivity measurements (Grant et al., 1989; Blackham and Pollard, 1997). Similar technique is used for permittivity measurements on fresh fruits and vegetables (Tran et al., Nelson et al., 1994a,b). Due to density variations in material, such techniques are not free of errors. If there are air gaps or air bubbles between the end of the coaxial probe and the sample, the technique is not suitable for determining permittivities of very low-loss granular and pulverized samples when bulk densities of such samples were established by auxiliary permittivity measurements.

3.5 Perturbation Technique

The cavity (TM or TE mode) perturbation technique is frequently used for measuring dielectric properties of homogeneous food materials because of its simplicity, easy data reduction, accuracy, and high temperature capability (Sucher and Fox, 1963; de Loor and Meijboom, 1966; Bengtsson and Risman, 1971; Metaxas and Meredith, 1983). The technique is also well-suited to low-loss materials (Kent & Kress Rogers, 1987; Hewlett Packard, 1992). It is based on the shift in resonant frequency and the change in absorption characteristics of a tuned resonant cavity, due to insertion of a sample of target

material. The measurement is made by placing a sample completely through the center of a waveguide (rectangular or circular) that has been made into a cavity. Changes in the center frequency and width due to insertion of the sample, provide information to calculate the dielectric constant. Changes in the Q-factor (ratio of energy stored to energy dissipated) are used to estimate the dielectric loss. EM field orientation for two standard cavity modes (TE and TM) is shown in **Figure 3.1.**

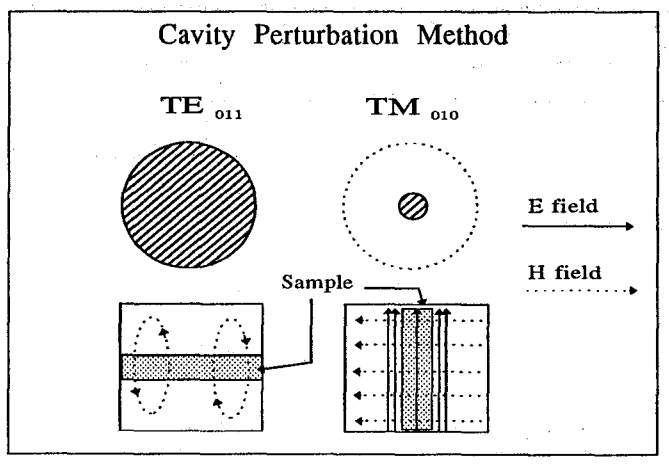


Figure 3.1 Schematic of a circular perturbation cavity in simple TE and TM modes.

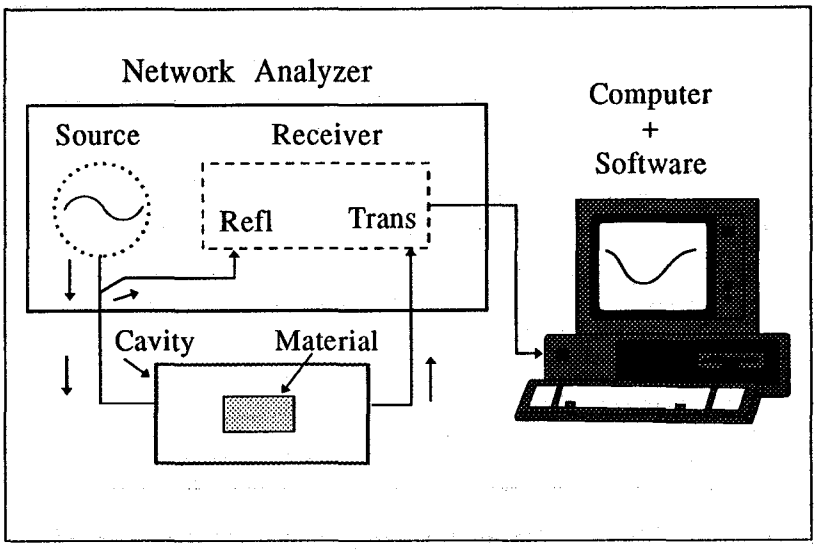


Figure 3.2 Schematic of a resonant cavity method.

The size of the cavity must be designed for the frequency of interest, the relationship being inverse (higher frequency, smaller cavity). Each cavity needs calibration, but once the calibration curves have been obtained, calculations are rapid. Sample preparation is relatively easy, and the permittivities of a large number of samples can be determined in a short time. This method is also easily adaptable to high (up to +140°C) or low (-35°C) temperatures (Risman & Bengtsson, 1971; Ohlsson & Bengtsson, 1975; Venkatesh, 1996), and has been used to determine the dielectric properties of many agri-food products over a wide range of frequencies, temperatures and compositions.

For ease of measurement, VNA can be used to automatically display changes in frequency and width (Engelder and Buffler, 1991). A recommended waveguide cavity design with skeletal theory and design details are available as a standard procedure published by the American Society for Testing and Materials (ASTM, 1986). The proposed research will target the development of such a measuring system to operate at certain ISM approved frequencies (915, 2450 MHz) and wide temperature ranges, etc., however the use of a very expensive network analyzer could be eliminated in the future. **Figure 3.2** represents a typical cavity measurement system using network analyzer.

3.5.1 Solid sample preparation

For solid materials, samples in the form of rods can be formed, molded, or machined directly from their material into microwave transparent test tubes or tubing. While quartz is the best available material for this purpose, borosilicate glass is considered acceptable, and ordinary glass should not be used. Wall thickness should be as thin as possible while having the required mechanical rigidity. Paper or plastic straws may also be used if glass is not available. For a semi-solid material such as Tylose™, the sample preparation is quite difficult; however a special micropipeting equipment for such gel type materials, has been successfully designed and built (Venkatesh, 1996) .

3.5.2 Liquid sample preparation

Liquids are filled into test-tube sample holders with a pipet. Small diameter pipettes themselves also make excellent sample holders. For los-loss materials, 200 μL pipettes are suitable and 10 μL pipettes for high-loss materials. Materials that can be melted can be poured into sample holders and allowed to solidify. This technique is appropriate if the material does not change its properties following melting and resolidification.

3.5.3 Semi-solid samples

Sample preparation involves either filling the sample in its molten state and then solidify or applying vacuum at one end while forcing the sample into a thin cylindrical shaped holders. Since temperature measurements may be difficult due to the nature of the materials such as cheese, butter, etc., it is important to develop suitable fixtures to contain samples at different threshold conditions.

3.6 Waveguide and Coaxial Transmission Line Method

Early efforts to characterize the dielectric properties of materials were made at the Massachusetts Institute of Technology (Roberts and von Hippel, 1946; von Hippel, 1954b). The values of ε' and ε'' were derived from transmission line theory, which indicated that these parameters could be determined by measuring the phase and amplitude of a reflected microwave signal from a sample of material placed against the end of a short-circuited transmission line, such as a waveguide or a coaxial line. **Figure 3.3** shows schematic of a reflected wave method, often adopted by a coaxial line. For a waveguide

structure, rectangular samples that fit into the dimensions of the waveguide at the frequency being measured are required. For coaxial lines, an annular sample needs to be fabricated. The thickness of the sample should be approximately one-quarter of the wavelength of the energy that has penetrated the sample. Since the shift in wavelength is related to the dielectric constant, a guess must first be made as to the magnitude of the constant. Typical thickness at 2450 MHz range from 0.2 " (0.5 cm) for woods to 0.75" (1.9 cm) for fats and oils. Dielectric sample holder design for a particular material of interest is an important aspect of the measurement technique.

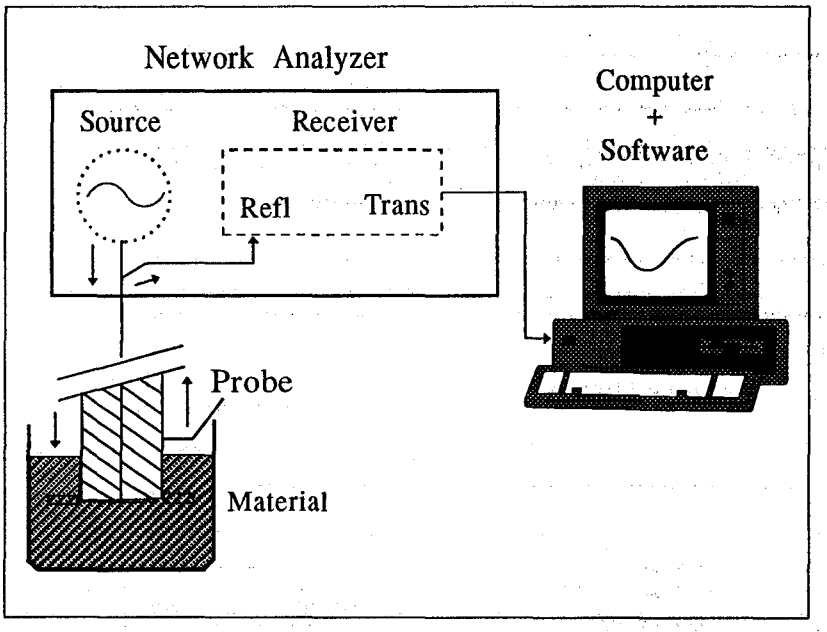


Figure 3.3 Schematic of a reflected wave method.

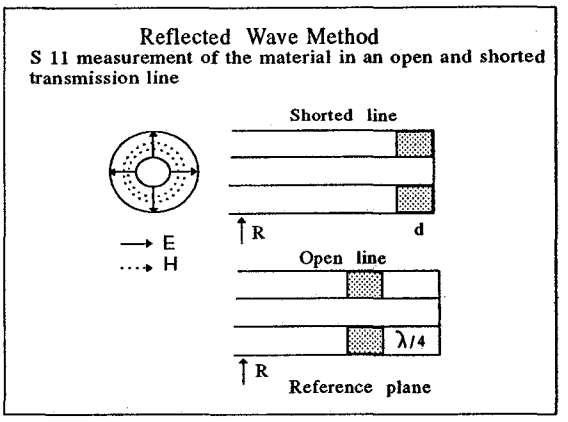


Figure 3.4 Schematic of a coaxial transmission method.

Coaxial-line and rectangular wave-guide sample holders were used with various microwave measurement systems assembled for dielectric properties determination on grain, seed, and fruit and vegetable tissue samples at frequencies from 1 to 22 GHz (Nelson, 1972, 1973b, 1980, 1983a). The same sample holders were also found to be useful for measurements on pulverized coal and mineral samples (Nelson et al. 1980,1989). The details of each of the above techniques is described in the following section. **Figure 3.4** represents coaxial measurement system.

3.7 Transmission line technique

This technique is cumbersome because the sample must be made into a slab or annular geometry. At 2450 MHz,the sample size is somewhat large, particularly for fats and oils. Commonly available waveguide test equipment for 2450 MHz is designated WR-284. For measurements at 915 MHz, only the coaxial line technique is practical due to the large size of waveguide required. Liquids and viscous-fluid type foods can be measured with this method by using a sample holder at the end of a vertical transmission line. The dielectric parameters can be easily and inexpensively obtained by the transmission line technique, particularly if one utilizes a slotted line and standing-wave indicator (Nelson et al. 1974). A more sophisticated implementation of the technique utilizes a swept-frequency network analyzer, where the impedance is measured automatically as a function of frequency. **Figure 3.5** represents a typical transmission measuring system using VNA.

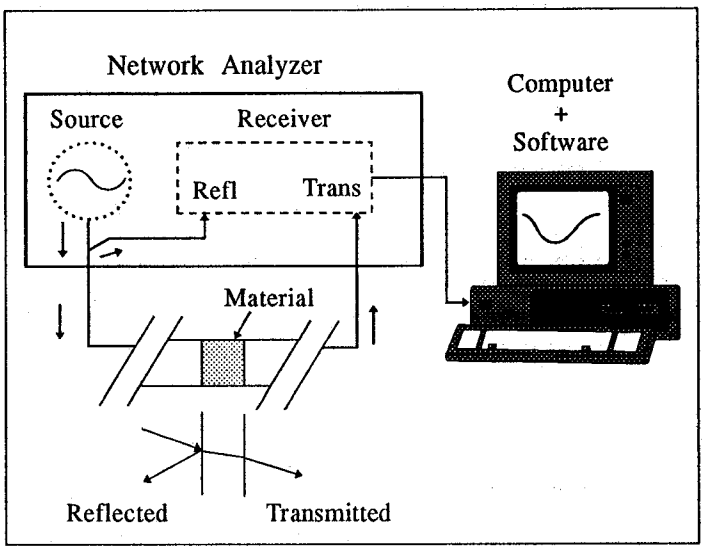


Figure 3.5 Schematic of a transmission line - waveguide method.

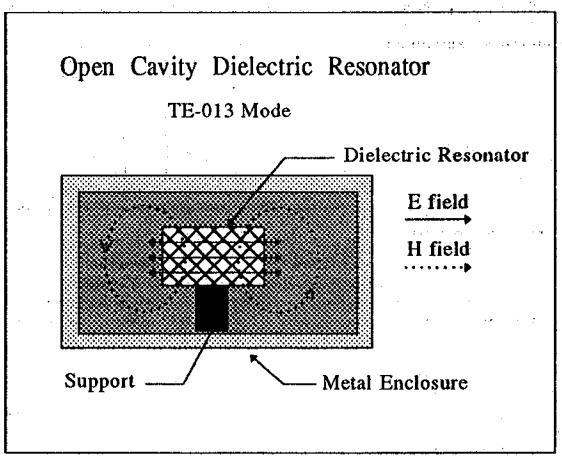


Figure 3.6 Schematic of an open cavity (TE_{013}) dielectric resonator.

3.8 Resonators and transmission line

A microwave resonator (as shown in **Figure 3.6**), partly or completely filled with a material can also be used to determine permittivity. The resonator (perturbation technique) is usually calibrated with materials whose dielectric properties are known, usually with organic solvents such as methanol, ethanol, etc. The measurement frequency range is from 50 MHz to above 100 GHz. If the transmission line is enclosed (ie. it is a waveguide), the permittivity of a material can also be measured without the resonator, by putting it directly inside the waveguide. The method applies to all liquid and solid materials, but not to gases since their permittivities are too low . There are, however, problems with the sample preparation of solid materials. The accuracy is not as good as that of the transmission line with resonator.

In transmission line methods, a sample of the substance is put inside an enclosed transmission line. Both reflection and transmission are measured. Although this method is more accurate and sensitive than the more recent coaxial probe method (described later), it has a narrower range of frequencies. As the substance must fill the cross-section of the transmission line (coaxial or rectangular), sample preparation is also more difficult and time consuming (Engelder & Buffler, 1991; Hewlett-Packard, 1992). When such methods are used to determine moisture content, the frequency used should be above 5 GHz to avoid the influence of ionic conductivity and bound water relaxation (Kraszewski, 1995). For this reason, some studies on dielectric properties vs density relationships have been concentrated to high frequencies. However, the size of microwave components is usually proportional to the wavelength and therefore, inversely proportional to frequency.

3.9 Open ended probe technique

A method that circumvents many disadvantages of the transmission line measurement technique was pioneered by Stuchly and Stuchly (1980). The technique calculates the dielectric parameters from the phase and amplitude of the reflected signal at the end of an open-ended coaxial line inserted into a sample to be measured. Care must be exercised with this technique because errors are introduced at very low frequencies and at very high frequencies, as well as for low values of dielectric constant and loss factor. This technique is valid for 915 and 2450 MHz, for materials with loss factors > 1. Interpretation for lower-loss materials such as fats and oils must be treated

with caution. Typical open-ended probes utilize 3.5 mm diameter coaxial line. For measurement of solid samples, probes with flat flanges may be utilized (Hewlett Packard, 1991). The open-ended probe technique has been successfully commercialized and software and hardware are available (HP dielectric probe kit).

The coaxial probe method is basically, a modification of the transmission line methods. It uses a coaxial line which has a tip that senses the signal reflected from the material. The tip is brought into contact with the substance by touching the probe to a flat face of a solid or by immersing it in a liquid. While the method is quite easy to use and it is possible to measure the dielectric properties over a wide range of frequencies (500 MHz - 110 GHz), it is of limited accuracy particularly with materials with low values of ε' and ε'' (Engelder and Buffler, 1991; Hewlett-Packard, 1992).

3.10 TDR (reflectometry) method

Time domain spectroscopy (or reflectometry) methods were developed in the 1980s and used for studies of the dielectric properties of food. Essentially, this method also utilizes the reflection characteristic of the material under test to compute the dielectric properties. They cover a frequency range from 10 MHz to 10 GHz. Measurement is very rapid and accuracy is high, within a few percent error. The sample size is very small and the substance measured must be homogeneous. Although these methods are expensive, they are excellent tools for advanced research on the interaction of the electromagnetic energy and materials over a wide frequency range (Mashimo et al, 1987, Ohlsson, T., 1987).

3.11 Free-space transmission technique

Of the measurement techniques available, free-space techniques is also grouped under non-destructive and contactless measuring methods. They do not require special sample preparation. Therefore, they are particularly suitable for materials at high temperature and for inhomogeneous dielectrics. In addition, they may be easily implemented in industrial applications for continuous monitoring and control. e.g, moisture content determination and density measurement (Kraszewski, et al. 1995).

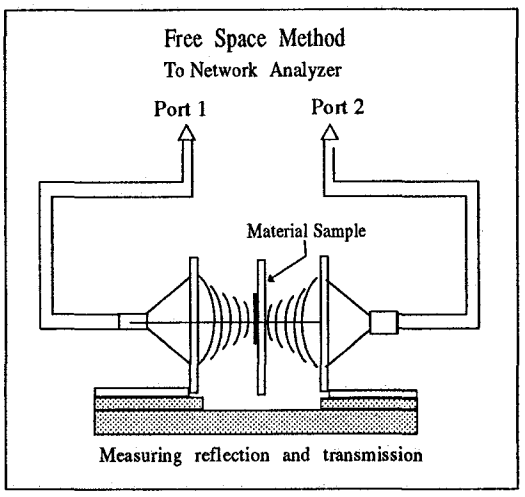


Figure 3.7 Schematic of a free-space transmission technique.

In a free-space transmission technique, a sample is placed between a transmitting antenna and a receiving antenna, and the attenuation and phase shift of the signal are measured. The results of which can be used to translate the material dielectric properties. Accurate measurement of the permittivity over a wide range of frequencies can be achieved by free space techniques. In most systems, the accuracy of ε' and ε'' determined depends mainly on the performance of the measuring system and the validity of the equations used for the calculation. The usual assumption made during this technique is that a uniform plane wave is normally incident on the flat surface of a homogenous material, and that the planar sample has infinite extent laterally, so that diffraction effects at the edges of the sample can be neglected. **Figure 3.7** represents a free-space measuring technique with the transmitting and receiving antenna elements.

Trabelsi et al. (1997) accounted for multiple reflections, mismatches, and diffraction effects at the edges of the sample as they are generally considered the main sources of errors. To enhance the measurement accuracy, special attention must be paid to the choice of the radiating elements, the design of the sample holder, and the sample geometry and location between the two radiating elements.

3.12 Microstrip transmission line

Microstrips have long been used as microwave components, and shows many properties which overcome some of the limitations, and thus making it suitable for use in dielectric permittivity measurement. It is well known that the effective permittivity (a combination of the substrate permittivity and the permittivity of the material above the line) of a microstrip transmission line (at least for thin width to height ratios) is strongly dependent on the permittivity of the region above the line. This effect has been utilized in implementing microwave circuits and to a lesser extent investigation of dielectric permittivity. Furthermore the measurement of effective permittivity is relatively straight forward, and well suited to implementation in industrial equipment. Such a system could be based on determining the effective permittivity of a microstrip line covered by an unknown dielectric substance (Keam et al., 1995). Use of printed circuit boards and adding substrate materials to characterize materials and measuring permittivity using algorithmic models, have been reported. However, its applicability to food and agricultural material processing would still be an anticipatory issue at this stage.

3.13 Six-port Reflectometer using an Open-ended Coaxial Probe

Ghannouchi et al., (1989) have been working on nondestructive broad-band permittivity measurements using open-ended coaxial lines as impedance sensors, which are of great interest in a wide variety of biomedical applications. An attempt is made to replace expensive automatic network analyzer (ANA) such as the HP8510B by combining the capabilities of personal computers with customized software to derive all the necessary information from less expensive components. The reported measuring system consists of a microwave junction designed to operate from 2 to 8 GHz and a number of standard microwave laboratory instruments (power meters, counters, sweepers, etc.) controlled by IEEE 488 bus interface by a microcomputer (HP9816) to provide a precision low-cost automatic reflectometer suitable for permittivity measurements. The device under test (DUT) is an open-ended coaxial test probe immersed in the test liquid kept at a constant temperature. Data acquisition and reduction are fully automatic. The complex reflection coefficient is calculated from the four power readings and the calibration parameters of the six-port reflectometer.

It is concluded that the SPR (six-port reflectometer) can provide nondestructive

broad-band permittivity measurements with an accuracy comparable to commercial ANA accuracy but at a considerable reduction in equipment costs. This effective transmission line method, used to represent the fringing fields in the test medium, provided a good model to interpret microwave permittivity measurements in dielectric liquids. Using such a model, the precision on relatively high-loss dielectric liquid measurements is expected to be good. However this method involves more complex mathematical procedure in order to translate the signal characteristics into useful permittivity data.

On a similar line, current research elsewhere has been initiated (Venkatesh et al., 1998) to measure the dielectric properties of agri-food materials which basically aims at reduction in huge costs (in relation to VNA) and accessories. The functional aspects of the network analyzer can be utilized to design and build the basic system which analyzes the transmission and reflection characteristics, resulting in dielectric measurements.

3.14 Colloid Dielectric Probe : (*Hewlett Packard*)

Engineers at HP have developed what they say is the first radio-frequency dielectric probe for evaluating colloidal liquids such as milk, etc. The unit can quickly and accurately measure dielectric properties of these types of materials, offering the promise of improving a variety of food, chemical, pharmaceutical and bio-chemical products.

The HP E5050A Colloid Dielectric Probe is designed for permittivity evaluation of colloidal liquid materials in the food, chemical, pharmaceutical, and biochemical industries. It operates from 200 kHz - 20 MHz with the HP4285A precision LCR meter and HP vectra personal computer. The advanced sensing technique provides permittivity vs. frequency characteristics. Its electromagnetic technique eliminates the electrode polarization effect which causes measurement error when ionic materials are measured with metal electrodes.

3.15 Instrumentation

3.15.1 Network Analyzers

This section is aimed at recognising the usefulness, development and role of simplified instrumentation steps in a MW measuring context. Network analysis is the process of creating a data model of the transfer and / or impedance characteristics of a linear network (active or passive). This is done through stimulus response testing over the

frequency range of interest. Some analyzers do this with point-to-point frequency testing, while others do this by sweeping the frequency band at one time.

Network analysis is generally limited to the definition of linear networks. Sine wave testing is an ideal method to characterize magnitude and phase response as a function of frequency. Network analyzers are instruments that can measure the transfer and/or impedance functions through sine wave testing. Since transfer and impedance function are ratios of various voltages and currents, a means of separating the appropriate signals from measurement ports of the device under test is required. The analyser must detect the separated signals, form the desired signal ratios, and display the results. At microwave frequencies, where standing waves might occur on the transmission line, the analyzer must be capable of separating the signal from the traveling waves.

Automatic Vector Network Analyzers (AVNA) are commonplace for doing these precise forms of measurements. Scalar network analyzers (magnitude only) and vector network analyzers (both magnitude and phase) are available.

Two types of detection methods are usually employed by network analyzers. Broadband detection accepts the full frequency spectrum of the input signal, while narrow band detection involves tuned receivers that convert continuous wave (CW) or swept RF signals to a constant intermediate frequency (IF) signal. Each detection scheme has its advantages.

Scalar analyzers usually employ broadband detection techniques. Broadband detection reduces instrument cost by eliminating the IF section required by narrowband analyzers. This sacrifices noise and harmonic rejection. Broadband systems can make measurements when the input and output frequencies are not the same, as in measurements of the insertion loss of mixers and frequency doublers. Narrowband systems cannot make these measurements.

Vector network analyzers normally employ narrowband detection techniques. This makes for a more sensitive low-noise detection of the constant IF. This also increases the accuracy and dynamic range for frequency selective measurements (as compared to broadband systems).

There is a good range of adaptability and flexibility in analyzer systems. Impedances can be shown on a Smith-chart overlay for a polar display. An S-parameter test set can be attached to perform S-parameter measurements. Computer controlled

network analyzers can be programmed to set up and make many measurements automatically. The measurement process is further accelerated by the computer's ability to store, transform, summarize, and output data in a variety of formats to a number of peripherals. Functions that are normally displayed in the frequency domain can be converted to the time domain for additional analysis.

3.15.2 Microwave measurements with network analyzer

When a network analyzer system is used for performing microwave measurements, there exist certain inherent measurement errors which can be separated into two categories : instrument errors and test set / connection errors. Instrument errors are measurement variations due to noise, imperfect conversions in such equipment as the frequency converter, cross-talk, inaccurate logarithmic conversion, non-linearity in displays, and overall drift of the system. Test set / connection errors are due to the directional couplers in the reflectometer, imperfect cables, and the use of connectors and adapters. The instrument errors exhibited by modern network analyzers are very small.

In a probe network analyzer measuring system, the primary source of measurement uncertainty is due to test set / connector errors at ultra high frequency and microwave frequencies. These uncertainties are quantified as directivity, source match, and frequency tracking errors. Hewlett Packard has developed a suitable analytical model to account for test set / connection errors for correcting reflectivity measurements on their semi-automatic network analyzer system. This model has been implemented for use with *in-vivo* measurement probe and equations which correct for the open-circuit fringing capacitance of the probe have been added to the algorithm (Burdette et al. 1982). Data for standard reference liquids such as water, methanol and ethanol might be helpful for correcting errors.

Since voltage and current values vary along the length of a transmission line, they are not suitable for accurate measurements at microwave frequencies; therefore, it is much more convenient to measure power. The measurement of microwave power requires that one should know how to operate power detectors and indicating instruments and how to apply techniques that minimize errors and increase the accuracy of the measurement. For power measurements (or any other parameter) to have any significance, the instruments used must be calibrated to specifications. Concept of uncertainty analysis will be useful

for accounting possible causes of error. Usually, there are three different power levels in a microwave measuring system : the power generated by the source, the amount on the transmission line, and that absorbed by the load. Evaluating these power differences involves a concept that is quite mathematical in nature.

3.15.2.1 Noise

It is helpful to divide noise into two types : internal noise, which originates within the microwave component or equipment, and external noise, which is a property of the channel. The channel is the link through which the signal travels. At any temperature above absolute zero (0°K or -273°C), electrons in any material are in constant random motion. Although this random motion does not produce a current flow in any direction, it does produce current pulses that are the source of noise. Most electronic systems are evaluated on the basis of a signal-to-noise ratio (S/N or SNR). It is not really the amount of noise that concerns, but rather the amount of noise compared to the level of the desired signal; that is the ratio of signal to noise power. This signal to noise ratio can be expressed in decibels. It is noted that the ratio is always given in power and not voltage. The formula for S/N in dB is given by ($S/N_{(dB)} = 10 \log P_s/P_n$), where P_s is the signal power and P_n is the noise power. The ratio is difficult to measure; since it is not possible to turn off the noise in order to measure the signal power alone.

3.15.2.2 Frequency measurements

Many microwave procedures require a measurement of frequency. There are basically two methods to measure microwave frequencies. The first approach, and the most accurate, is to measure the frequency directly with a frequency counter. Direct frequency measurements are made by comparing an unknown signal to a reference frequency, the crystal oscillator. The input signal is first conditioned into a series of pulses, then passed to the main gate. The frequency is measured by generating a gate time, consisting of a number of cycles of the reference clock, during which the input signal is counted. The frequency is calculated by dividing the number of cycles by the gate time.

To make frequency measurements at microwave frequencies various down-conversion techniques are used to convert the microwave input to an IF so that the resultant signal can be directly counted. The three basic techniques for down-conversion

are prescaling, transfer oscillator, and harmonic heterodyne.

3.15.2.3 Prescaling

Prescaling uses a divider circuit to reduce the frequency of the input signal to a lower frequency that can then be counted by the direct counter circuit. However, this technique has frequency limitations.

If the microwave signal's amplitude is sufficiently low, the output of the detector is proportional to the square of the microwave signal voltage and, therefore, proportional to the microwave signal power (since $V^2 \propto P$). When the voltage is low, the detector is said to be operating in its quadratic, or square-law region. When the microwave signal power is greater than -15 dBm, the voltage of the detector's output signal tends to be directly proportional to the microwave signal voltage. The detector is said to be operating in its linear region; that is, it rectifies the applied signal.

3.16 Development Aspects

3.16.1 Optimum I S M & D frequency based equipment

As we know, domestic microwave ovens operate at 2450 MHz. However, a large commercial oven might be destined to operate at any of the other approved ISM frequencies, which could give gains in cost efficiency, processing time, or product quality. A key factor in such a choice is ε of the product being processed. Dielectric measurements and computer modeling will help to choose the optimum frequency.

Researchers need to understand microwave performance and optimize product design, instead of using expensive "trial and error" guesswork. Dielectric measurements also have uses in package design, process control, and physical/chemical analysis.

Any measuring system needs improvement and accurate considerations. The need, suitability and the operating conditions, all play a dominant role in evaluating the performance of such systems. Since biological materials are transient in nature, it is difficult to standardize the tools for dielectric measurements; however, applying proper calibration and mathematical routines one can minimize errors and generate useful information on the MUT. The following section deals with some of the recent developments in the permittivity measurement domain. The details can be obtained by referring to cited literature.

3.17 New Microwave sensor - On-line moisture and density measurement

King (1997) has reported new developments and applications for a continuous, on-line determination of moisture content and dry density of food products with microwave sensors. Two unique types of sensors and their ancillary electronics for process control and/or product quality measurement are introduced. For both, measurement principles are based on the interaction of electromagnetic fields with the dielectric and power dissipative properties of matter, particularly of water. These properties are diagnostics of the partial water and dry densities.

The material being measured is interfaced through various ways with an open reflection type resonator which is in contact with the material. Sensors of this type can be flat or curved for flush mounting in a shaker, hopper, chute or a conveyor pipe. The other sensor developed is a non-contacting type, wherein a microwave beam is transmitted through the test material. Signal attenuation (absorption) and phase delay measurements are measured and then related to the moisture and dry basis weights by suitable empirical algorithms. Either type of sensors indicated above can be used for continuous, on-line measurements or as stand-alone benchtop instruments (King, 1997). Both sensor types have some advantages as well as limitations with respect to flexibility, applicability to different forms of materials, resolution and accuracy, cost, speed of data acquisition, etc. However, the relative advantages and limitations are matters of degree, depending on the particular application.

3.18 Other useful applications of permittivity measurements

There are other uses for dielectric properties measurements (not related to microwave heating of food) that can be of interest to the food researcher. Some of them are listed below (Nelson, 1973a):

✓ An important use of the dielectric properties of grain and other agricultural products is their exploitation for rapid, nondestructive sensing of moisture in materials.

✓ Moisture content is often the most important characteristic of agricultural products, because it determines their suitability for harvest and for subsequent storage or processing. It often determines the selling price of the products for intended purposes.

✓ Dielectric properties have been utilized with properly designed electronic sensors with reasonable accuracy. Such moisture testing instruments, operating in the range of 1-50

MHz range, have been developed and used for rapid determination of moisture in grains for many years.

✓ More recently, techniques have been studied for sensing the moisture content of single grain kernels, seeds, nuts, and fruits so that instruments for measuring the moisture content of individual objects can be developed.

✓ There is a need for more precise on-line moisture monitoring equipment that can provide continuous records for commodities moving into and out of storage, or being processed or loaded for transport.

✓ Recent studies have shown that with proper measurements, such as simultaneous measurement of microwave signal attenuation and phase changes, reliable moisture measurements can be obtained independent of density fluctuations (while products are conveyed).

Continued research and development of such techniques are aimed at providing tools for better management of factors important in sensing, preserving, processing and maintaining the quality of agricultural and food materials for ever growing consumers.

IV MATERIALS AND METHODS

4.1 Perturbation theory

This developmental research study is based on the existing concept of cavity perturbation and its principles. It is well known that the insertion of a dielectric material different from air inside the cavity affects its resonant frequency, f_o and quality factor, Q. If the sample is small enough compared to the cavity dimensions, perturbation theory may be used to link frequency shift Δf to the dielectric constant, ε' and the quality factor shift ΔQ to the loss factor ε''.

The survey and the experimental procedures as well as the results of the well known cavity perturbation concept, commonly employed for measuring the dielectric properties of agricultural and food materials have been reported (Altschuler, 1963, Gallone et al. 1996, Kraszewski, 1996; Venkatesh, 1996). This method allows for simple and precise cavity measurements and also improves the rapidity, which makes it useful for process monitoring applications. Calibration and measurement procedures are necessary to carry out in any new method and design.

4.1.1 Principles and Concept

The concept of cavity perturbation has been developed and widely applied in the study of dielectric properties of several materials (plastics, ceramics, foods) over wide frequency ranges in recent years. Presently, many passive and active techniques for measuring dielectric quantities are available and much work has been carried out by researchers using this method, thus proving it to be a very powerful tool of investigation.

The experimental requirement consists in the determination of the characteristic parameters of a resonant cavity, namely the resonant frequency and the merit factor Q (Q-factor) of the cavity. The parameters have to be measured with the empty cavity and with a small dielectric sample, properly inserted. Then the real and imaginary parts of the complex permittivity can be calculated. All known techniques feature the resonant frequency measurement by taking the minimum (maximum) of the power reflected (transmitted) by the cavity to the external network during an appropriate frequency sweep (Ney and Gardiol, 1977), or in phase-lock conditions (Akyel et al., 1978; Martinelli et al., 1986). The Q-factor is usually found by measuring the positions of the -3 dB points in the

resonance curve with appropriate coupling conditions, although it is possible to measure the Q-factor directly from the decay time of the output signal of a cavity excited by microwave pulses (Montgomery, 1947) or through the phase shift with a frequency tracking system (Akyel et al., 1978, 1983).

When the aim of an experiment is the study of dielectric properties of a material for monitoring some chemical or physical processes, the response time (reaction time) of the experimental apparatus must be sufficiently small in comparison with the time scale involved in the process. As a consequence, when dealing with such purposes, researchers have so far neglected techniques involving time consuming frequency sweeps, turning rather to phase-lock methods (Akyel et al., 1978) whose performances are well established in giving both rapid and accurate measurements. Indeed, a phase lock loop ensures an almost immediate recognition of the resonance frequency so it allows a continuous monitoring of its value and, consequently, the real part of the complex permittivity.

In such development work reported, the microwave power is usually supplied by the frequency synthesizer (signal generator) : it provides a narrow frequency sweep which crosses the resonance point. The frequency extremes of the sweep can be rapidly and smartly adjusted in order to follow the migration of the cavity resonance. Time resolution and frequency range must be chosen according to the process to be monitored, therefore determining the type of microwave source, power detector and cavity shape and size. Synthesizers are able to change frequencies very rapidly, within 50 ms (milliseconds) with phase-lock, but some can also be used in a fast swept mode ; such instruments are called 'Synthesized Sweepers'.

The following sections briefly describe the working components (electronics) and essentials of the permittivity analyzer (measuring equipment) being designed and developed. It is designed to operate on the same lines as the network analyzer however the cavity perturbation technique is the main focus. This limits the choice of wide frequency sweeps and ranges. The present measuring system is aimed at measuring the dielectric properties of agri-food materials for further microwave / RF processing applications at two major ISM frequencies.

4.2 Permittivity Analyzer at 915, 2450 MHz - using cavity perturbation concept

The development of a new permittivity analyzer with the earlier concept of cavity

perturbation, operating at two major ISM frequencies (915, 2450 MHz) is presented and discussed in the following sections. The main process variables considered for practical interest are frequency and temperature even though moisture content and bulk density are equally important. Comparative studies involving network analyzer measurements and permittivity analyzer developed, may be necessary to evaluate the performance and validation of the results (Venkatesh et al., 1998).

The permittivity measurement is based on the perturbation concept and uses a circular cavity operating in standard TM_{010} mode (ASTM, 1986). The material used for cavities are copper (915 MHz) and brass (2450 MHz), which have high thermal conductivity and are recommended for such measuring systems (Gauthier, 1997). These circular cavities were coupled to a constant external recirculating temperature control bath permitting the dielectric properties measurement at temperature range of practical interest (freezing, cooking and boiling). TM_{010} mode is widely used for its simplicity in mathematics and symmetry. The design and fabrication of the resonant cavities for various modes have been reported elsewhere. The excitation of the microwave signal and the coupling is usually achieved with standard antenna loop and iris. Proper tuning of the loop was necessary to achieve the best peak conditions for the desired resonant frequency of the cavity. The main perturbation equations followed by the perturbation theory are:

$$\varepsilon' = 1 + \left(Bessel \times v_c \times \left(\frac{f_o - f_s}{f_o} \right) \right) \div v_s \qquad (4.1)$$

$$\varepsilon'' = \frac{Bessel}{2} \times \left(\frac{1}{Q_s} - \frac{1}{Q_o} \right) \times \frac{v_c}{v_s} \qquad (4.2)$$

where;

Bessel = Bessel function of first kind for circular (TM_{010}) cavity = 0.539

Q_s = Q of cavity with sample

Q_o = Q of empty cavity

v_s = volume of sample $(mm)^3$

v_c = volume of cavity $(mm)^3$

f_o = resonant frequency of empty cavity (GHz)

f_s = resonant frequency when sample is in place (GHz).

To perform measurements with this technique, the user has to input the following main parameters regarding the cavity:

(i) resonant frequency of empty cavity and when loaded with sample,

(ii) Q of cavity with and without sample,

(iii) volume of the cavity,

(iv) volume of the sample (all SI units).

4.3 Sample Selection and Preparation

The samples selected in this study represent some of the commonly used standard agri-food materials and chemicals (Table 4.1). In using the cavity perturbation technique, it is very convenient to prepare liquid samples *vs.* solid or semi-solids as it not only eliminates the density effect on the dielectric parameters but also offers homogeneous sample representation.

4.3.1 Organic Solvents

Ethanol and ethanol-hexane mixtures (90% ethanol+10% Hexane, 70% ethanol+30% hexane, 50% ethanol + 50% hexane, 30% ethanol +70% hexane, 10% ethanol + 90% hexane) were prepared and properly sealed in the standard glass laboratory bottles. The sample bottles were kept in ambient conditions until the dielectric measurements were performed. The solvents were purchased from Fisher Scientific, Canada and all the tested samples were of laboratory grade purity. Measurements were performed at both 915 and 2450 MHz and at varying temperatures.

4.3.2 **Edible oils** (vegetable)

Commercially available cooking / edible oils such as canola (*Brassica napus L.*), sunflower (*Helianthusannuss L.*) and soya (*Glycine Max.*) were stored in the refrigerator (4°C) for 2 days and small quantities were drawn and kept at ambient conditions for 1-2 hours before the start of dielectric measurements.

4.3.3 Neem Oil /pulp

The Neem oil was procured from a pharmaceutical laboratory (Medinova pharmacy, India) and was stored in the refrigerator until hours before dielectric properties testing. The neem kernel was manually separated from the seed to obtain the pulp that was very soft and delicate. The seeds were crushed using a standard grinder (Multi speed Osterizer™) for 5 seconds and the pulp was separated. The history of the seed production and processing was not known.

4.3.4 Milk

Commercially available homogenised milk (Quebon™ : 1%, 2% and 3.25% fat) was procured from the local super market and was stored in the refrigerator for 2 days. Small quantities were drawn and kept in ambient conditions for 1-2 h before the start of dielectric measurements.

4.3.5 Liquid sample preparation

Liquid samples such as ethanol, hexane (and their mixtures), and edible oils and milk were pipetted into the standard corning / micro-pipettes (10, 50,100µl) made of borosilicate glass. The selection size of the micro-sampling pipettes depend on the lossiness of the sample to be tested. High loss materials such as milk prefer lower range (volume) micropipets since the cavity demands the small sample size for the signal to interact with such samples. One end of the pipet was sealed using a standard tube sealant (S/P™ Sure-seal, Baxter Model B4425, Canada) which is a very low loss material itself. The volume of the sample (mm^3) was calculated by knowing the height of the sample or the cavity and the rated capacity of the micropipet:

* Height of the sample = 68mm
* Height of the cavity = 40mm
* Capacity of the micropipet (sample holder) = 100 µl
* Volume of the sample (v_s) = (40/68) x 100 = 58.82 mm^3

4..4 Temperature control

The cavity was conditioned to various temperature settings by an external refrigerated circulator (Fisher Scientific ISOTEMP 1013S, Canada). Ethylene glycol was

used as the cooling / heating fluid. The temperature readings were read on an LCD screen. The sample attained the set temperature value within 2-3 minutes after inserting the sample into the cavity. Care was taken to avoid heat loss by proper insulation. The operating range of the unit was -30 to +200^{0}C. The set values were confirmed by measuring the inside (geometric center of the cavity) temperature using the standard 'K' type thermocouple sensor whose tip was mounted with the similar sample holder.

4.5 Design and Fabrication of Resonant Cavities (TM_{010} Mode)

All the steps followed in the design and development of the resonant cavities (both 915 and 2450 MHz) were based on the concept reported in the **ASTM Designation D2520-81** (ASTM Standards Standard Test Methods for Complex Permittivity (Dielectric constant) of Solid Electrical Insulating Materials at Microwave Frequencies and Temperatures to 1650^{0}C). Plane wave propagation in the cavity can follow the transverse electric mode (TE) or the transverse magnetic (TM) mode. The second alphabet designates which field has its direction always and everywhere transverse to the direction of wave propagation. The mode notation also includes three subscripts in the form of TM_{mnk} or TE_{mnk}. The alphabet 'm' represents the number of full cycles of transverse field variation in one revolution through 2 radians of diameter; 'n' represents the number of zeros of the transverse field along the radial of a guide; 'k' represents the number of repeatition of the TM_{mnk} or TE_{mnk} mode with the opposite phase of the field configurations in the axial direction. In this research, the mode of configuration of electromagnetic waves was $\mathbf{TM_{010}}$ type (transverse magnetic) for its simplicity in construction (symmetry) and mathematical steps. The shape of the cavity was circular.

4.5.1 Cylinder Walls

The perturbation method requires that the specimen be relatively small compared to the volume of the cavity and that the specimen be positioned symmetrically in a region of maximum electric field. Although resonant cavities are sensitive to low-loss materials, the small specimens size limits the precision attainable. Nevertheless, the method has several additional advantages besides reasonably good precision.

The permittivity analyzer (detailed below) was connected to the resonant cavity operating in standard TM_{010} mode (ASTM, 1986). The materials used for cavities are high

quality copper (915 MHz) and copper-brass combination (2450 MHz). As their thermal conductivities are high, they demonstrate low electrical losses and are recommended for such measuring systems (Gauthier, 1997). These circular cavities were connected to an external constant temperature recirculating bath, permitting the dielectric properties to be measured at a range of temperatures of practical interest. TM_{010} mode was chosen for its mathematical simplicity and symmetry. The design height of the cavity was 45 mm and the sample-microwave exposure height was 40 mm. The cavities were made at the Post-harvest Engineering Laboratory of McGill University, Macdonald Campus. The design and fabrication of the resonant cavities for various EM modes (TM, TE) and measuring techniques is reported (ASTM Standards,1986). Proper tuning of the antenna was necessary to minimize the loss in the coupling and to obtain the maximum Q_0 (the ratio of energy supplied to the energy absorbed inside the resonant cavity).

4.5.2 915 MHz resonant cavity

The cylinder was constructed out of a 6 mm thick flat copper bar made of copper of high thermal and electrical conductivities. The material was rolled into a cylindrical shape using a roller press. After the bar was rolled into shape, the joint was soldered using a 50/50 lead solder. The piece was then loaded into a lathe to machine the side walls into a uniform thickness and to finish the inside surfaces to the required smoothness. Both ends of the cylinder were grooved to accept a fitting end cap. The piece was cleaned and made ready for assembly. The thickness of the bottom and top plates was 15.24 mm. The circumference of the cavity with the clearance (wall thickness) was 822.2 mm. The inner diameter of the cylinder was 249.68 mm. The height of the cavity was maintained at 40 mm (inside) for both frequency schemes. The microwave transmission was confined to the enclosed volume of the cylinder with the above dimensions. The total enclosed volume of the cavity (V_c) is 196,7862 mm^3. **Figures 4.1** and **4.2** illustrate the pictorial view of the end cap and the end cap- cavity before assembly. The cavity was connected to the test ports of the Permittivity Analyzer through appropriate coaxial cables (M17184-RG223).

4.5.3 2450 MHz resonant cavity

A similar procedure was followed for the 2450MHz cavity. In this case the metal used was a combination of copper and brass for better conductivity. Since the diameter

and the overall size of the cavity is smaller than the 915MHz frequency cavity, a combination of copper and brass metals offered least resistance from the machining and polishing point of view . The top and bottom plates were made of brass (125 mm thick) and the wall was made by a sheet of copper (60 mm thick). The coupling of the cavity and the analyzer was accomplished exactly as the 915 MHz system. The diameter of the cavity was 96 mm and the height was maintained at 40 mm. The empty cavity volume (V_c) was calculated to be 280,308 mm^3.

Inner surface area was always kept smooth and polished to enable the cavity to yield higher and optimum Q values. The cavity was connected to the test ports of the Permittivity Analyzer through appropriate coaxial cables (MIL-C-17 68999 AA-3413).

4.5.4 2 End Caps/parts assembly

The assembly procedure of the end caps and parts were similar for both the frequency schemes. Two end caps of the same grade copper and thickness were prepared to close the ends of the cylinder. The bottom end cap was first cut into a circular shape in a band saw. To mount the piece into the lathe, a hole was drilled at its ends to accept a bolt to hold the piece into the lathe's chuck. A matching groove was cut on the circumference of the disc to fit into a groove at the end of the cylinder. The disc was sweat welded into the cylinder using a 50/50 lead solder. After the joined piece cooled, two holes (95.3mm dia.) were drilled into the cylinder to accommodate two microwave couplers that will introduce the microwave 'antennas' into the cavity. One brass fitting was soldered into each hole. Each brass fitting was threaded to the pitch required by the couplers. The centering hole for the disc was also plugged with a small copper disc soldered into the hole. The piece was then re-machined to remove excess solder and smoothen the joints between the two pieces as well as between fittings and plug.

A second disc was prepared using the same method as the bottom cap. In this disc, the centering hole was plugged with a brass fitting used to connect a threaded specimen holder. The inner surface of the disc was thoroughly smoothed with the lathe and the grooved edge was dampened to avoid sweating out of solder when the piece was welded.

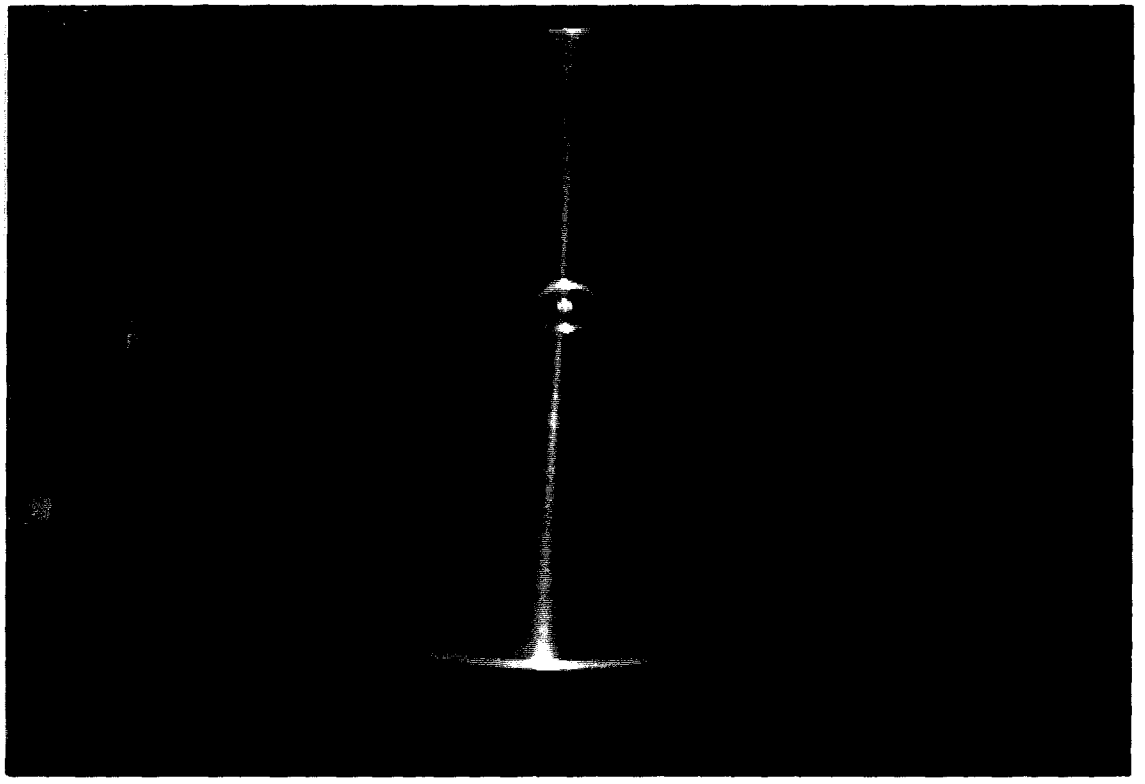


Figure 4.1 A photograph of the End-Cap of a 915 MHz cavity set-up

Figure 4.2 A photograph showing interior of 915 MHz cavity and an end-cap

The disc was then press fitted into the groove of the cylinder and welded using a 50/50 lead-tin solder. All the openings of the cavity were closed except for the top end cap. Using a suitable brass fitting for connector rings, compressed air was introduced into the cavity to check for leaks. When the cavity was found to be air tight, the external surfaces were cleaned and insulated with an aluminum / Styrofoam duct insulating material. The joints of the insulation were sealed with aluminum tape. The cavity was then mounted over four leveling supports made up of (11.1 X 50.8) mm bolts (Fig. 4.14). The coupling antenna (standard) and specimen holders were installed and the unit was coupled to the microwave generator / Vector Network Analyzer (HP 8753D) for testing and evaluation purposes. The **Figures 4.3 and 4.4** exhibit pictorial representation of cavity with coupling element and a standard MW coupler antenna element for the 915 MHz cavity.

4.6 Critical Cavity Parameter

The dimensions of the cavities are to be determined with very specific rules. Cylindrical cavities are more commonly used than rectangular ones (Akyel, 1991). For simplicity, only the equations for the size of the cylindrical cavities is shown here;

Cylindrical cavity in the TM_{010} mode : $f = 22.966 / D$ (4.3)

where : f = resonant frequency in GHz

D = inside diameter in cm

The only restriction to the height of the cavity is that it must be less than 0.5 D. Also, it is recommended that the cavity be made of brass, copper or aluminium. High moisture content samples and high frequency cavities (5 GHz and above) are not recommended for this type of design. The rule of thumb is that the sample size should be equal or smaller than $1/1000^{th}$ of the volume of the empty cavity to minimize perturbations (ASTM, 1986). Literature suggested the use of simple and easy mathematical steps (Akyel, 1991) for basic modes of electromagnetic energy configuration. Accordingly, the was decided to use and simplify the reported steps of calculations that formed the basis of the cavity design and development. Suitable constants were introduced to take into account the size, shape and the nature of various materials under test. The following section represents the tracking of resonant frequency limits and computational steps during a standard sweep of the measurement .

Figure 4.3 A photograph of the 915 resonant cavity with the MW coupler

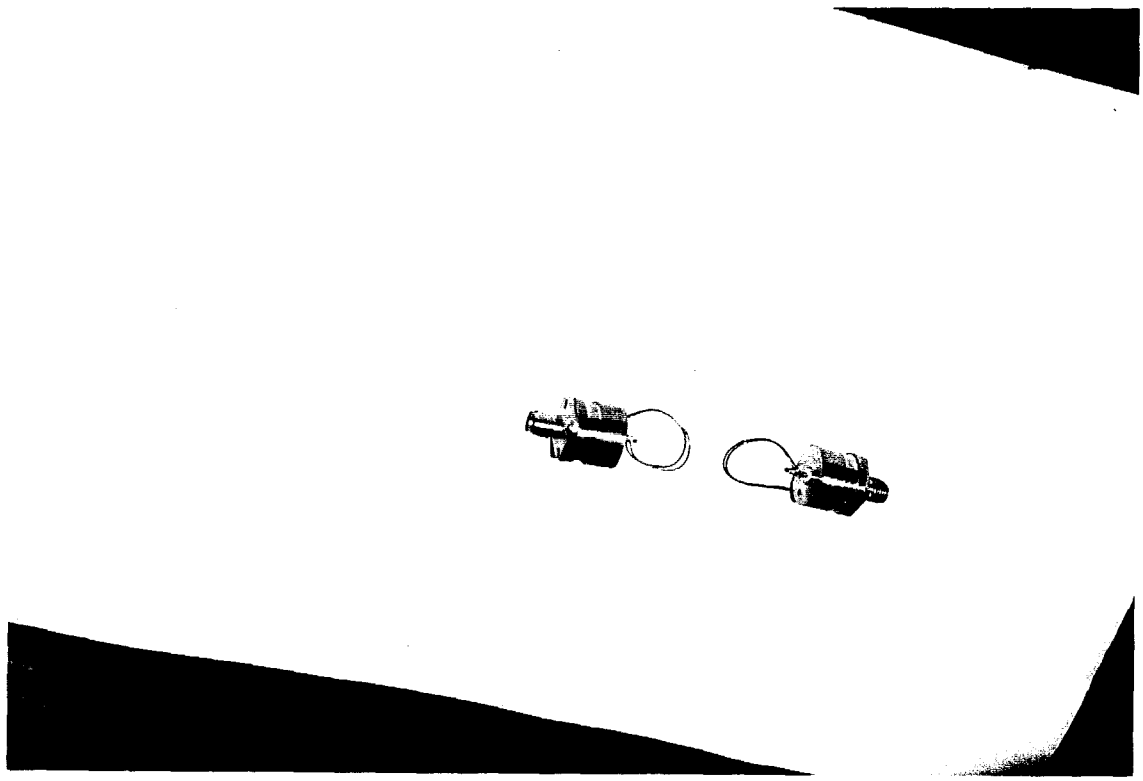


Figure 4.4 A photograph of standard MW couplers with an antenna loop

4.7 Mathematical representation

The Q-factor (Q) represents the ratio of energy supplied and energy stored in a given enclosure of electromagnetic field. It is the basic governing parameter necessary to estimate the dielectric loss factor and heat dissipation mechanisms.

$$Q = \frac{f}{f_2(3dB) - f_1(3dB)} \tag{4.4}$$

where :

f = resonant frequency

f_2 (3dB) = frequency 3 dB past f (frequency where signal is 3db below resonant frequency, (f_2>f)

f_1 (3dB) = frequency 3 dB before f (frequency where signal is 3db below resonant frequency (f_1<f).

Accomplishing this procedure with the empty cavity results in the determination of Q_o, the Q factor of the empty cavity. With this reference Q factor, it is then possible to obtain the Q factor with samples with a simple relation of frequencies:

$$Q_s = Q_o * \frac{\Delta\omega_O}{\Delta\omega_S} * \frac{\omega_S}{\omega_O} \tag{4.5}$$

where :

Q_s = Q of cavity loaded with a sample

Q_o = Q of empty cavity (without sample)

ω_o = resonant angular frequency of empty cavity

ω_s = resonant angular frequency of loaded cavity

$\Delta\omega_o$ = resonant angular frequency band width of empty cavity

$\Delta\omega_s$ = resonant angular frequency bandwidth of loaded cavity

4.7.1 Dielectric Properties Calculation

Some of the common assumptions used in the calculation of ε' and ε" are:

(i) the maximum electric field intensity is maximum at the geometric center of a tuned resonant cavity

(ii) the shape and orientation factor (Bessel) of the cavity did not change with the geometry

of the resonant cavity for 915 and 2450 MHz frequencies.

(iii) there was no perturbation inside the cavity for empty cavity measurements

(iv) there was no influence of magnetic field on agri-food materials at both frequencies

(v) coupling coefficient was negligible and was not calculated

The following equations apply to all methods using the cavity perturbation technique and therefore are not restricted to Active Cavity Perturbation. The perturbation theory has been discussed by many authors (Waldron, 1967). It establishes a relationship between the properties of the cavity (shift in resonant frequency, Δf and Q-factor, Q) and the dielectric properties of the material (ε' and ε"). The transmission factor (ΔT) must first be described as :

$$\frac{\Delta T}{Q_o} = \left(\frac{1}{Q_s} - \frac{1}{Q_o}\right) \tag{4.6}$$

Then, the perturbation equations are :

$$\Delta F = 2(\varepsilon' - 1)Kf_o\left(\frac{v_s}{v_o}\right) \tag{4.7}$$

$$\Delta T = 4\varepsilon'' k^2 Q_o\left(\frac{v_s}{v_o}\right) \tag{4.8}$$

where :

ε' = dielectric constant

ε" = dielectric loss factor

v_s = volume of the sample

v_o = volume of the cavity

K = factor dependent upon object shape, orientation and permittivity.

Combining equations (4.6) and (4.8), will result in:

$$\frac{\Delta T}{Q_o} = \left(\frac{1}{Q_s} - \frac{1}{Q_o}\right) = 4\varepsilon'' K^2\left(\frac{v_s}{v_o}\right) \tag{4.9}$$

or

Other perturbation equations found useful for TM_{010} circular cavity are:

$$\varepsilon' = 1 + 0.539 \left(\frac{v_o}{v_s}\right) \left(\frac{\Delta f}{f_o}\right) \tag{4.10}$$

$$\varepsilon'' = 0.269\left(\frac{v_o}{v_s}\right) \left(\frac{1}{Q_s} - \frac{1}{Q_o}\right) \tag{4.11}$$

where, $\Delta f = (f_o - f_s)$, Bessel =0.539 for circular cavity.

4.8 Instrumentation and Software

The operation of the apparatus was made user friendly with the development of an appropriate modification of a Quick Basic Program to a Visual Basic version 6.0 for Windows 95/98/NT. When using the software, it first prompts the user to enter the parameters which remain constant regardless of the samples. It first asks for the resonance frequency of the cavity, in order to know what frequency to sweep during the sensing. The next thing the operator must enter is the volume of the empty cavity, for further use as v_o in Eqns. 4.10 and 4.11. The last input is the shape of the cavity (rectangular or circular), to use the proper value of Bessel constants in the equations.

When proceeding with sample testing, the next step is to put the empty sample holder in the cavity and press enter on the PC Keyboard. The program then activates the permittivity analyzer (PA) to read ΔF and Q_o for an empty cavity but with the sample holder present so that the tested material is the only addition in the real test. The program then prompts the user to put the sample in the sample holder and press enter. It then takes only a few seconds for the program to calculate ΔF and Q. The next information the operator is asked to enter is the volume of the sample v_s. The program then has all the variables necessary to calculate ε' and ε'' and display them on the screen.

4.9 Essential components and functions of new Permittivity Analyzer (PA)

4.9.1 Description of the functional block

The entire measuring system consists of a PA, computer (PC) and a cavity, as

shown in **Figure 4.5**. The PA is connected to the computer through a standard serial port. The connection is the same for both frequency schemes, only the resonant cavity is changed. The functional block of the permittivity analyzer consists of a micro-controller, microwave signal synthesizer, low pass filter, detector, ADC (16 bit) and a resonant cavity as represented in **Figure 4.6**.

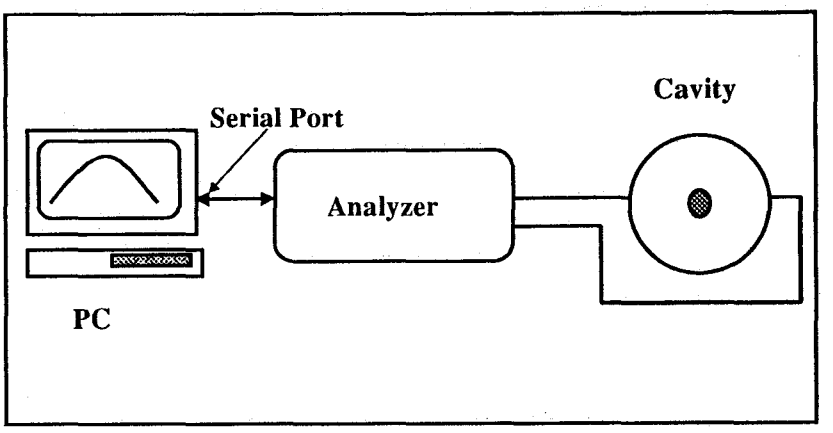


Figure 4.5 Schematic of the measuring set up

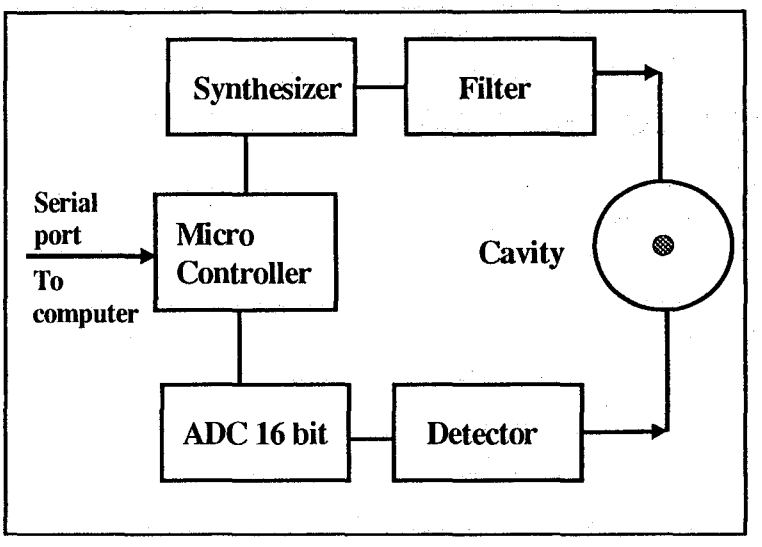


Figure 4.6 Functional block of the Permittivity Analyzer & resonant cavity

Figures 4.7 and 4.8 illustrate the photographic view of experimental set-up with both resonant cavities (915, 2450 MHz) along with the permittivity analyzer and the refrigerated re-circulator. Computer interface is also shown.

4.9.2 Microwave Synthesizers

Two synthesizers were installed in the equipment. The synthesizer for the 915 MHz band swept from 600 to 1200 GHz, whereas the synthesizer for the 2450 MHz band,

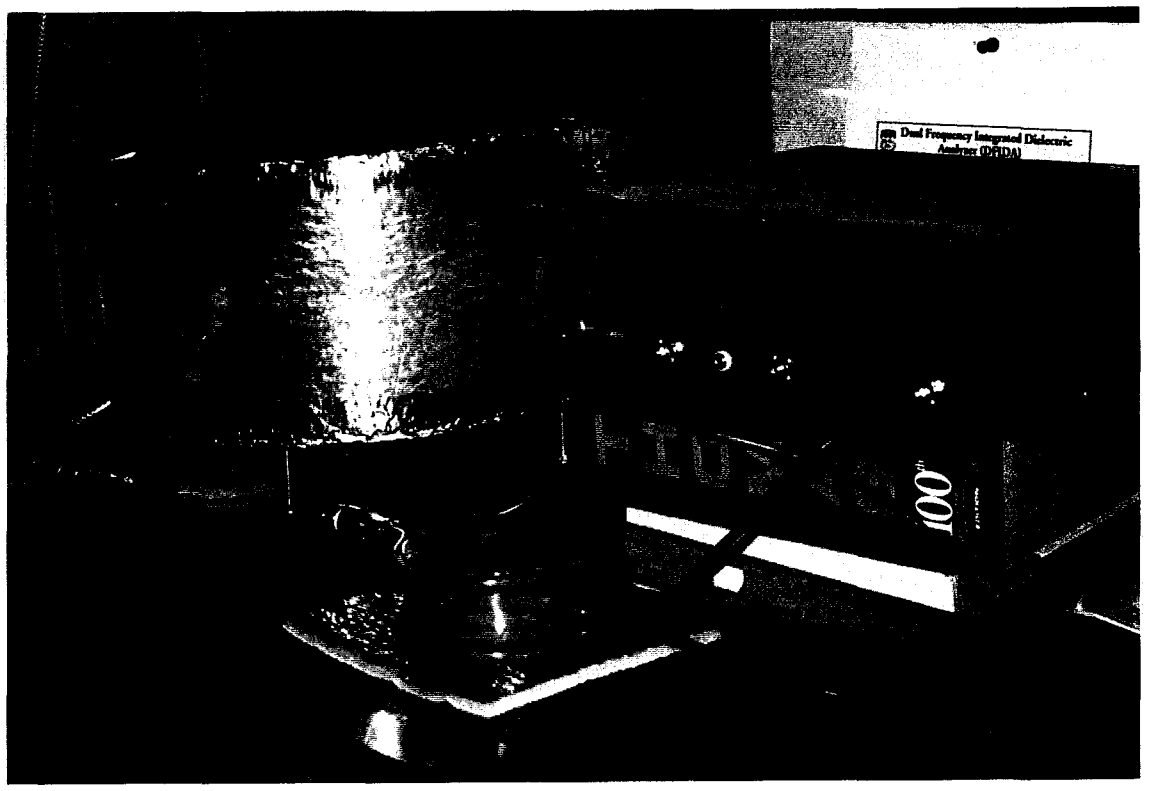


Figure 4.7 A photographic view of experimental set-up with both resonant cavities

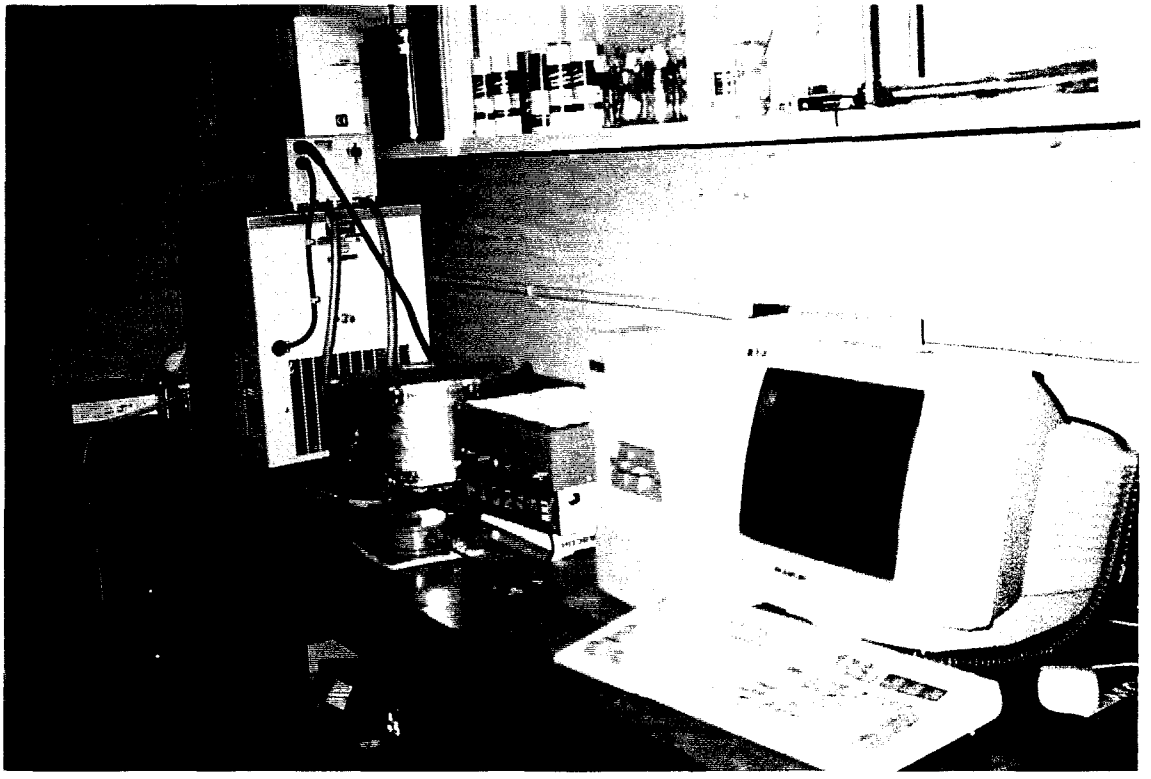


Figure 4.8 A photographic view of experimental set-up with both resonant cavities (915, 2450 MHz), permittivity analyzer, refrigerated circulator and a PC

swept from 2200 and 2500 MHz.

4.9.3 Synthesizer specifications

The LMX 2325 (National Semiconductor Corporation, 1996) is a high performance frequency synthesizer with integrated prescaler designed for operation up to 2.5 GHz. Using a proprietary digital-phase locked-loop technique, the synthesizer generates a very stable, low phase noise signals to perform permittivity analysis.

The synthesizer **Figure 4.9** consists of a crystal oscillator (highly stable), reference counter, normal (main) counter, phase comparator, prescaler, voltage control oscillator. The R counter divides the frequency of the crystal oscillator (10 MHz).

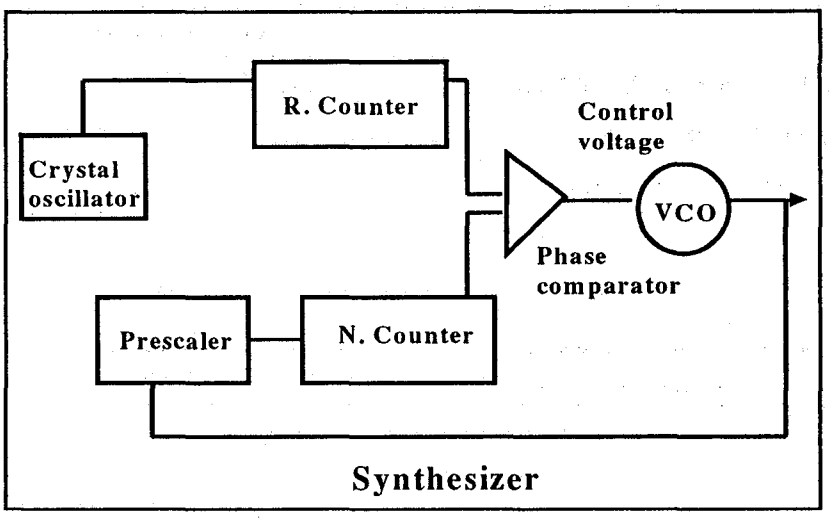


Figure 4.9 Schematic of microwave synthesizer

The output of the Voltage Control Oscillator (VCO) was in the range of 2.2 to 2.5 GHz (**Figure 4.10**). For a 2450 MHz frequency scheme, 0V refers to a frequency of 2.2 GHz and the 5V refers to a maximum scale of 2.5 GHz. The frequency span limit ranged from 350 MHz to 0.3 GHz, respectively, for the 915 and 2450 MHz. Inside the synthesizer there are two counter chains; one going in a phase comparator and the other one to the reference (R) counter. This divides the frequency of the reference signal. In our case the reference signal was a 10 MHz signal originating from a crystal oscillator. On the other counter chain the signal comes from the VCO. A pre-scaler makes the first division while

the N counter makes the second one. These two chains of counters are individually programmable. When the signals are coming from the R chain and the N chain, they are compared in a phase comparator. The output of the phase comparator changes the voltage of the VCO input to tune it to the right frequency. As a result of this, the control voltage will change and the system tracks and locks when both the signals have attained the same frequency.

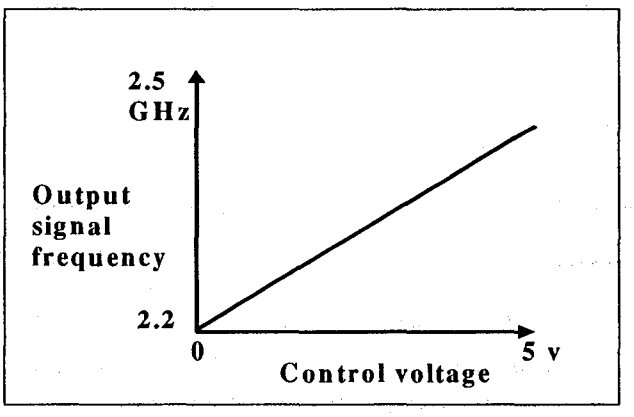


Figure 4.10 Standard VCO output curve

The following equation was used to program this system:

$$f_{out} = \frac{prescaler \times Ncounter}{Rcounter / f_{crystal}} \tag{4.12}$$

where $f_{crystal}$ was the frequency of the crystal oscillator. The number of bits in the R and N counters were 18 and 19 respectively and te prescaler was fixed at 64 bits. Two signals are present at the output of the VCO; the fundamental of 2.45 GHz and the second harmonics (**Figure 4.11**). A low pass filter is connected at the output of the VCO to eliminate the harmonic.

A similar configuration was accomplished at the lower band ; 600 to 1200 MHz for the 915 MHz system.

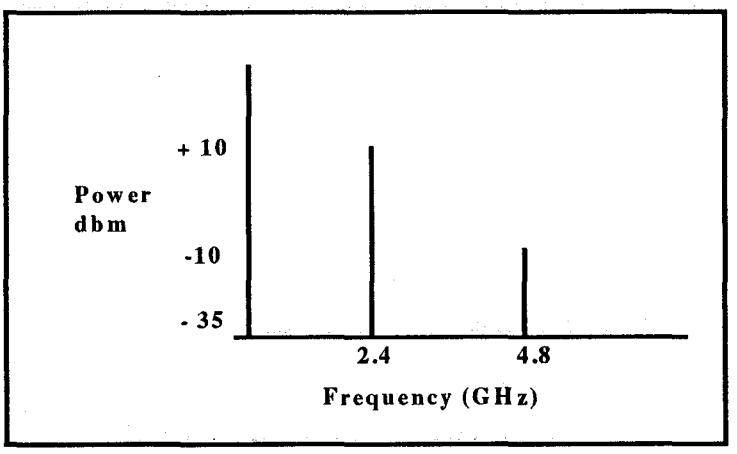


Figure 4.11 Secondary harmonics of VCO

4.9.4 Detector

Power was detected using a schotky diode. This zero bias diode rectifies the RF signal. A 50 Ω resistor was placed in parallel to the input to match it. The output voltage is related to the input power by the following relationship :

$$V = \sqrt{PR} \tag{4.13}$$

In our case, R = 50 Ω. This equation represents the voltage as a square law function.

4.9.5 ADC : *Analog to Digital Converter*

An ADC was used to translate the voltage to bits. The resolution of this system was 16 bit, or 65536 different states. The input signal to the ADC was ∓2 volts = 4 volts whereas the maximum output of the detector was only 200 mV. Therefore, an amplifier was added with a logarithmic gain. The fraction gain of the amplifier allowed a higher gain at low power and a lower gain at high power. The minimum microwave signal that could be detected with this system was -40 dBm. The functional elements of the ADC are shown in **Figure 4.12.**

4.9.6 Microcontroller

The micro-controller is a mini computer. It has a 32 byte RAM, EEPROM (hard disk) of 2 kb and 16 serially programmable input/output ports. The purpose of this micro-controller (**Figure 4.13**) is to interface data between the computer and different circuits of

the analyzer. The micro-controller receives data coming from the serial port of the computer and addresses the synthesizer and ADC. Then it sends back the binary values of the ADC reading to the computer.

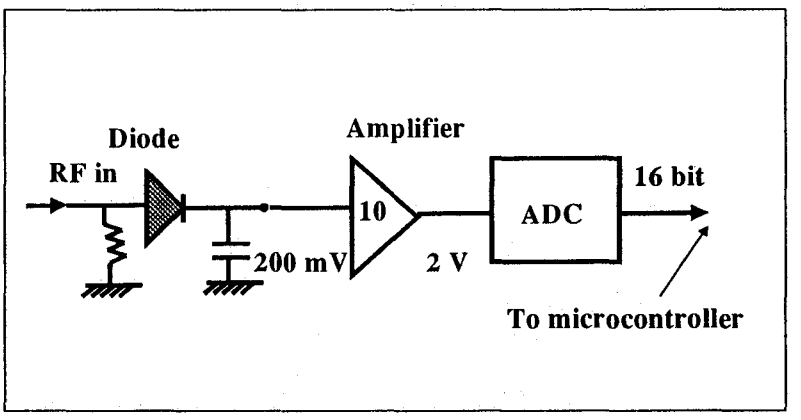


Figure 4.12 ADC circuit diagram

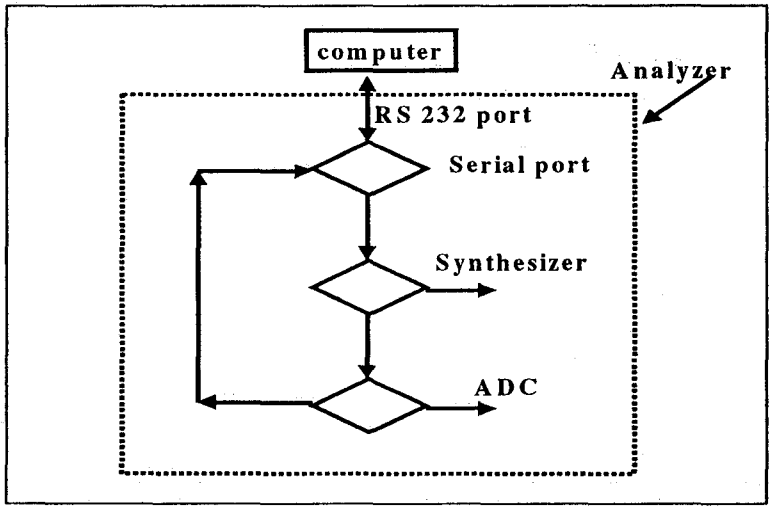


Figure 4.13 Flow chart of the Micro-controller

4.9.7 Amplifier

A 20dBm amplifier could be plugged into the output port of the permittivity analyzer. The purpose of this amplifier was to increase the power into the cavity for sample heating. The sample is small in size and just a fraction of a watt is sufficient to heat it. At the same time, by adding an amplifier, the gain of the total chain is higher. More gain is necessary when one attempts to measure high loss materials. By this means the dynamic

range of the PA is improved. However, if a low loss material is to be tested (with amplifier), one can expect saturation of the detector because too little microwave energy is absorbed by the sample. To prevent this an attenuator was added to the input of the detector. Typically the maximum power at the input of the detector is 0 dBm and the minimum power is -40 dBm. Without the amplifier the dynamic range of the analyzer is 40 dB. By using the amplifier properly we can increase the dynamic range of PA by the factor of the gain of the amplifier. Without a sample (empty cavity) the attenuation of the cavity is in the range of 12 to 15 dB, and with a high loss material the attenuation in the cavity reaches 40 to 60 dB. Thus, with a proper amplifier the factor of the gain reduces the attenuation in the cavity for high loss material such as water. Since most of the agri-food materials have high dielectric loss factor values, it will be necessary to optimize the working dynamic range of the system before permittivity measurements are planned. Addition of suitable amplifiers may be an easy solution to handle high loss materials, but it increases the associated cost of other components in the assembly. For measurement purposes to handle various materials, the control software was modified to allow the user to input appropriate numbers. **Figure 4.14** shows a photograph of an amplifier and attenuator elements for both the frequency schemes. **Figure 4.15** shows a snap shot of the actual control software window display screen.

4.9.8 Component specification

The amplifiers comprised of the following specification:

Attenuation : 10 dB and 6 dB

Amplifier : from Mini Circuits parts NO ERA 5

Gain is 13 dB at 2.45 GHz and power output is 100 mW

Gain is 17 dB at 915 MHz and power output is 100 mW

The original RF output of our system was < 5 mW however it was upgraded with suitable amplifiers and detectors to handle power output of about 150 mW in all the measurements reported in this study.

4.9.9 Control software

The algorithms were translated from Q-basic to Visual Basic programming to control the PC that first makes a sweep in frequency to find roughly the value of resonant

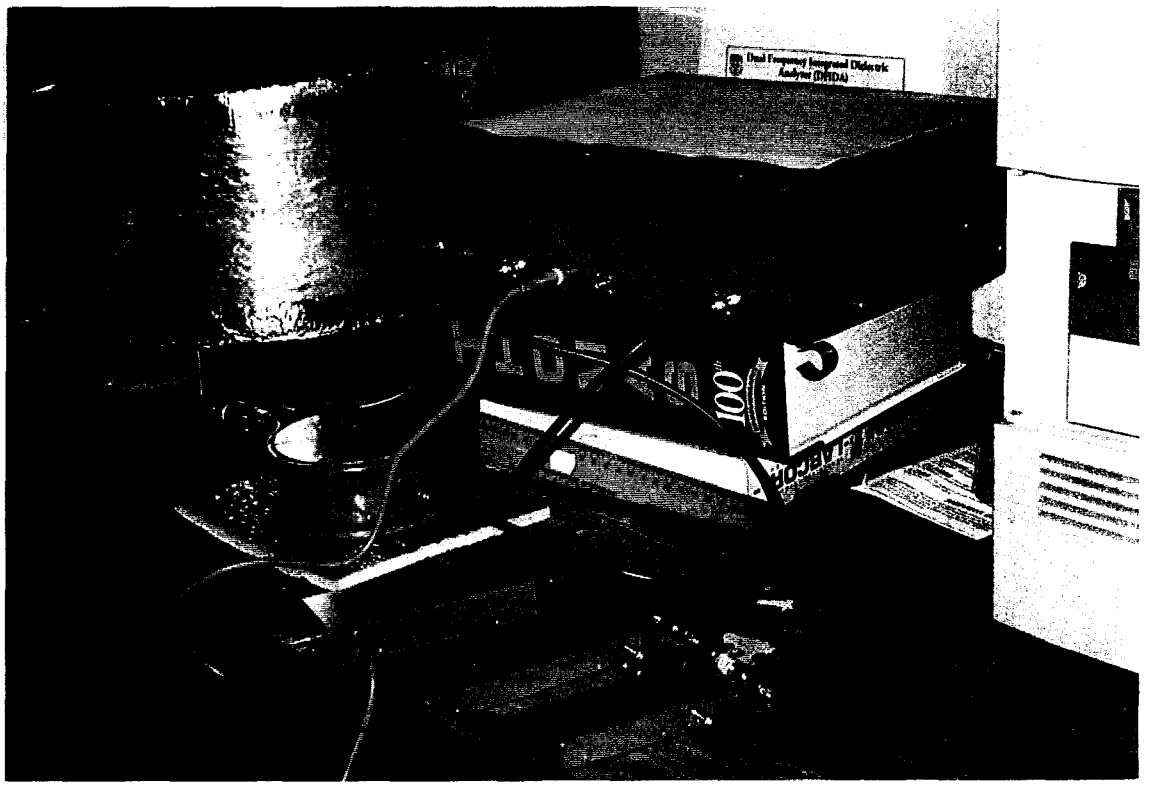


Figure 4.14 A photographic view of experimental set-up (915, 2450 MHz), permittivity analyzer and amplifier / attenuators and filters

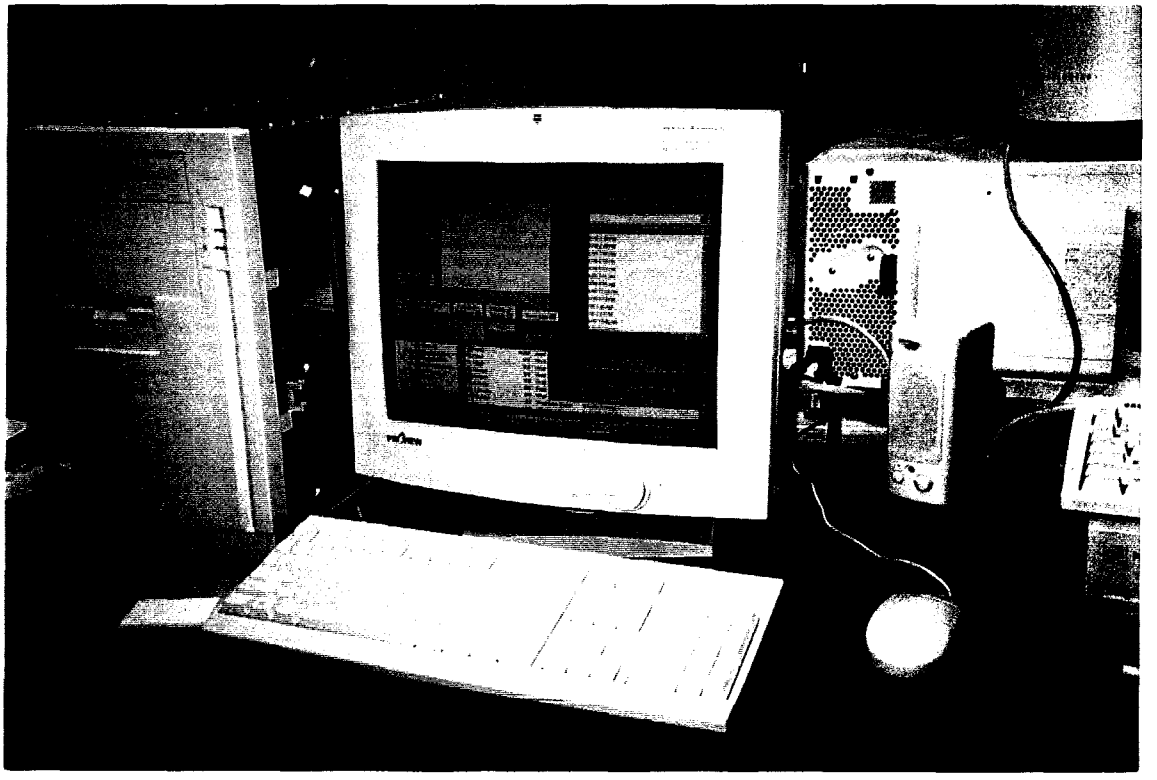


Figure 4.15 A snapshot of development of the control software in Visual Basic for windows environment for 915 & 2450 MHz schemes

frequency. After the initial sweep, it is zoomed further at this point with a maximum resolution of 20 kHz and a curve of resonant frequency vs. power is plotted. Subsequently, it searches for the maximum and minimum (± 3 dBm points) power. The calibration curves (**Figure 4.16, 4.17**) for the detector are included in the main program. A frequency sweep is made at the input of the cavity and the power is read at the output and subsequently, a curve of transmitted power vs. frequency can be generated to track the resonant frequency. **Figure 4.17** illustrates the typical 3dBm range points and frequency shift.

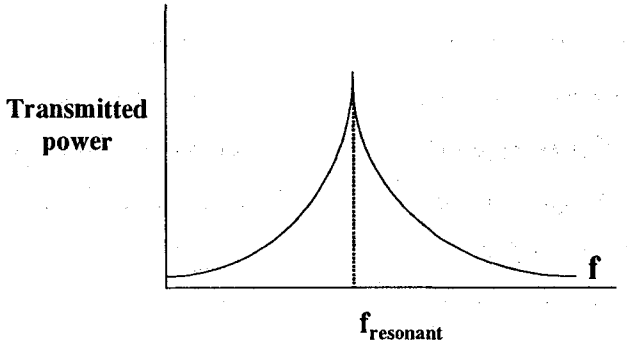


Figure 4.16 Typical resonant frequency curve

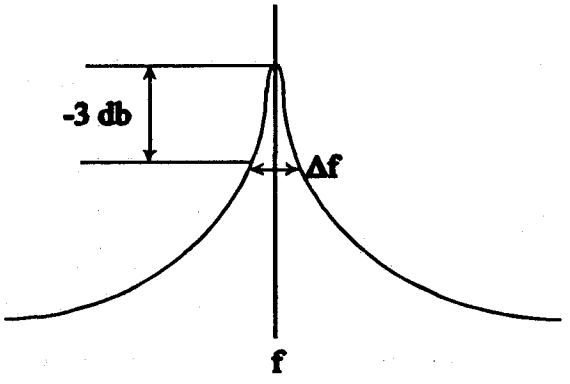


Figure 4.17 Typical resonant frequency shift curve

With the help of a computer program, the value of the maximum resonant frequency was found and from this information, dielectric constant ϵ', was computed. At the same time, with a calibrated output detector, we can compute the Q. Once Q is known, the dielectric loss factor $\epsilon''$is computed. With this information, we calculate f_o and Q_o. The sample is loaded and the steps are repeated to calculate Q_s and f_s. Further, with this data, one could easily obtain the dielectric properties (ϵ', ϵ'') of material under test.

This equipment (**Figure 4.18**) can be connected to any IBM compatible PC (486 or higher) computer through a RS232 cable to a serial port. The 915 MHz cavity assembly along with a temperature sensing unit is illustrated in **Figure 4.19**.

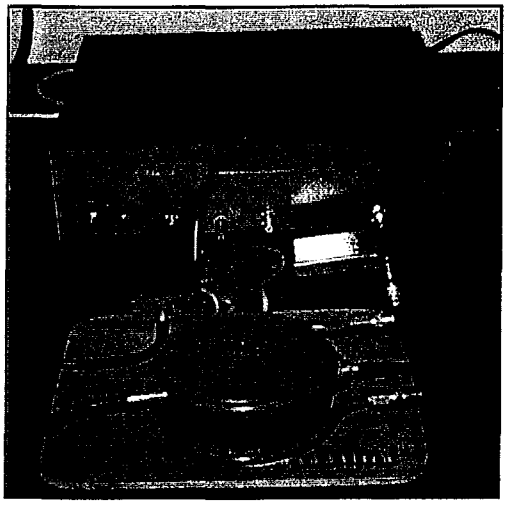


Figure 4.18 Pictorial view of the permittivity analyzer assembly for a 2450 MHz resonant cavity connected with an amplifier and an attenuator.

A control software was developed to control the analyzer functions and compute the complex permittivity of the material under test. The schematics and the circuit diagrams of each component and the data acquisition layout for both frequencies of the analyzer is shown in the **Appendix.**

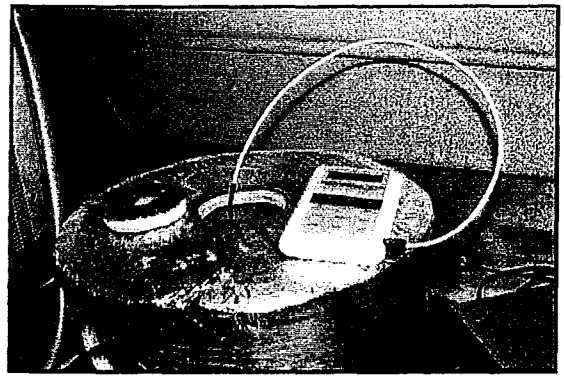


Figure 4.19 A 915 MHz resonant cavity (insulated) with a temperature sensor (K type thermocouple) at the maximum electric field position.

4.10 Experimental Plan

After a series of trials and testing of the developed permittivity analyzer with standard reference materials such as air, water and alcohol, it was decided to carry out the permittivity analysis of certain edible oils of plant origin (soya, canola and sunflower), homeogenized milk (1, 2 and 3% fat), neem oil and pulp, ethanol and hexane mixtures and selected standard laboratory reagents. These materials were tested for their repeatability and to ensure the proper working of the equipment. Cavity perturbation technique was used to measure parameters such as: dielectric constant, loss factor, Q-factor (empty, loaded), resonant frequency (empty, loaded). 2 replicates were used for all the samples and an average of 4 readings each were recorded for both 915 and 2450 MHz frequency schemes at varying temperatures.

Table 4.1 Experimental samples and treatment levels.

The table presents the sample types and test parameters configured in this study.

Material	Frequency (MHz)	Temperature (°C)
1. Edible Oils	915, 2450	-25 to 100
Soya, Canola, sunflower		-25 to 100
2. Milk (homogenised)	915, 2450	-25 to 100
1% fat, 2% fat, 3% fat		-25 to100
3. Neem products (seed oil, pulp)	915, 2450	0 to 100
4. Organic solvents Ethanol+Hexane mixtures (6 levels)	915, 2450	20 to 60
5. Standard chemicals Alcohols, reagents, etc	915, 2450	22

V RESULTS AND DISCUSSION

5.1 Development of a Permittivity Analyzer

The main objective of the research presented in this thesis was to develop an equipment package permitting the determination of the dielectric properties (permittivity) of materials over a wide range of temperatures, as well as at two frequencies. The equipment specifics and measurement procedure are described in Chapter IV. The present chapter attempts to evaluate the performance of the developed technology, and consists of three sections. The first section focuses on the working of the equipment and control software features. The second section focuses on repeatability and validity of measurements on the test substances listed in Tables 5.1 and 5.2. The last section represents the results of dielectric properties measurement of all the samples, followed by a section on economic and other advantages of permittivity analyzer.

A permittivity analyzer was designed and developed based on the existing concept of cavity perturbation at two ISM approved frequencies; 915 and 2450 MHz ranges and suitable for obtaining the values at variable temperature levels, according to the design principles reported in ASTM Designation:D2520-81 (Method B, C -Resonant cavity perturbation method). Suitable control software was developed to carry out the measurement procedure and analysis. Selected standard reference materials (solvents, water, air and alcohols) were tested for their ε' and ε'' values at single frequency and temperature combinations. Both the resonant cavities (915, 2450 MHz) exhibited very high Q values (> 4000) and are therefore inherently sensitive for low loss measurements. Once the performance (shift in Δf and Q-factor) was tested and found satisfactory for standard reference materials, detailed experiments were carried out on various agri-food materials such as edible oils (soya, sunflower and canola), plant based oils (neem oil and pulp), solvent mixtures (ethanol and hexane)and homogenised milk at varying fat percentages at both 915 and 2450 MHz frequencies and varying temperatures.

5.2 Working of the permittivity analyzer, software development and testing

As discussed in Chapter IV, the permittivity analyzer consists of a resonant cavity at 915 MHz and 2450 MHz each, and the component assembly that consists of two MW synthesizers, a micro-controller, a detector, and an analog-to-digital converter (ADC).

These synthesizers are in fact programmable microwave generators operating in a frequency ranges: 600 to 1200 MHz and 2200 to 2500 MHz, respectively for 915 and 2450 MHz frequency schemes. They are programmable by incremental steps of 20 kHz frequency, thus giving the possibility of 50,000 and 12,000 points in the range of interest, for 915 MHz and 2450MHz, respectively. A micro-controller is needed in this analyzer to manage all signals coming from the PC to control the synthesizer and ADC. In order to reduce the reaction time, the software starts with a wide sweep in the selected band, and once the resonant frequency is tracked, the analyzer will focus to this point and perform an accurate measurement at that point. The number of points was as high as 800 in this small portion of spectrum. At the output of the cavity the power is measured using a zero-bias diode (Schottky). The voltage of the diode is a function of the power of the received signal. A calibration curve is stored by the software to translate this voltage to power. The voltage measurement is made by a 16-bit ADC. For each voltage measurement, signal is converted and returned to the computer through the serial port. Hence, a complete permittivity analysis can be carried within 10 seconds. This equipment can be connected to a standard PC through a RS232 cable to a serial port. Software has been developed to control the analyzer functions and compute the dielectric parameters.

5.2.1 Tuning of the resonant cavities (915 and 2450 MHz)

The resonant cavity was coupled to a network analyzer (VNA) (HP 8753D) through standard co-axial cables and the coupler-antenna element was tuned at both ISM frequencies (915, 2450 MHz). Standard procedure (Log-Mag Marker™) was followed to excite the MW signal and check the performance of the cavities. Initial tuning is necessary to optimize the EM energy configuration mode (TM_{010}) of the system and to test for it's peak resonant conditions that govern both frequency and Q-factor shifts during subsequent empty and loaded cavity measurements. **Figure 5.1** shows the standard resonance curves (power vs. frequency) of the PA system tuned by VNA at both frequencies. The 'start' and the 'stop' points of the frequency sweep are narrowed to a smaller range during each sweep and in the case of 915 MHz system, the peak value of resonant frequency is in the order of 916.10008 MHz and for the 2450 MHz system, it was 2489.1367 MHz (ISM tolerance is 100MHz). Similar resonant curves with and without the perturbing object are reported by Kraszewski & Nelson (1994) and Akyel et al(1978).

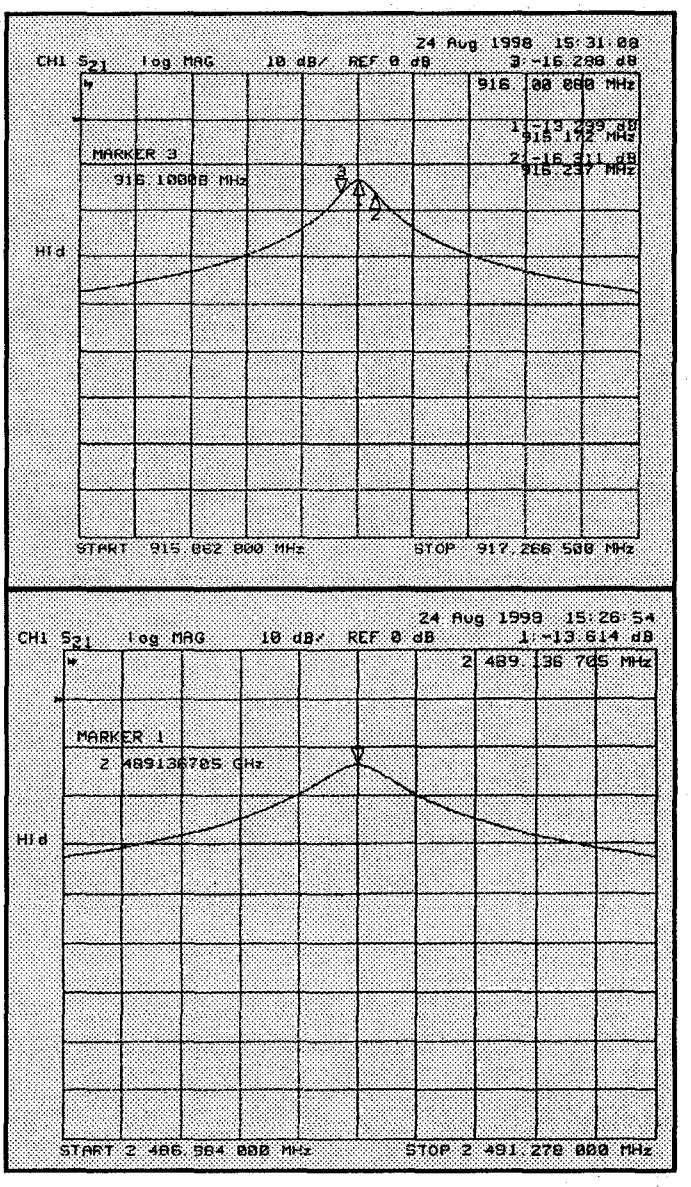


Figure 5.1 Standard calibration curves of permittivity analyzer obtained by network analyser tuned to both 915 MHz (a) and 2450 MHz (b) frequency schemes

5.2.2 Control software development

Figure 5.2 shows the block diagram and flow sequence of the dielectric properties measurement steps. The main program consists of defining all the variables and constants with calling statements to various sub-programs dedicated to specific tasks, such as configuring input parameters, calculating empty resonant frequency and Q-factor, tracking peak resonant frequency conditions, etc. The entire user-friendly program (control software) was developed in Visual Basic 5.0 (MS-Windows 95/98/NT) from the original program written in Q-Basic (MS-DOS). The interface of the analyser and the computer is controlled by this software. Microwave signal generation and sweeping across the selected bandwidth takes place according to the desired test material and experimental conditions. Initialization refers to the normalizing of diode functions to zero before the input parameters are configured. The program allows an empty cavity measurement and stores the values of the peak conditions of resonant frequency and Q-factor. The measurement of the loaded cavity follows the final sweep and the change in both the peak conditions will be inputted in another sub-program and is called in the main module every time there is a sweep. **Figure 5.3** represents the main control page of the software that governs the overall dielectric measurement. The computation of dielectric parameters and the generation of graphics (frequency vs. power) along with the calculated values of the above parameters. Pull-down menus are made available to the user for inputting the cavity specifications (volume, shape) for configuration. **Figure 5.4** shows the frame designed to 'save' all the data points and computed values along with other useful information. This option was not possible in the Q-basic version. The snapshot frame of the 'configuration' window appears as shown in **Figure 5.5**. Both the empty and loaded cavity measurements can be performed in less than one min for both frequencies and the program has the 'time delay' function to accelerate the translation of binary data. Every aspect of the measurement was incorporated in the main program supported by various sub-routines for different actions.

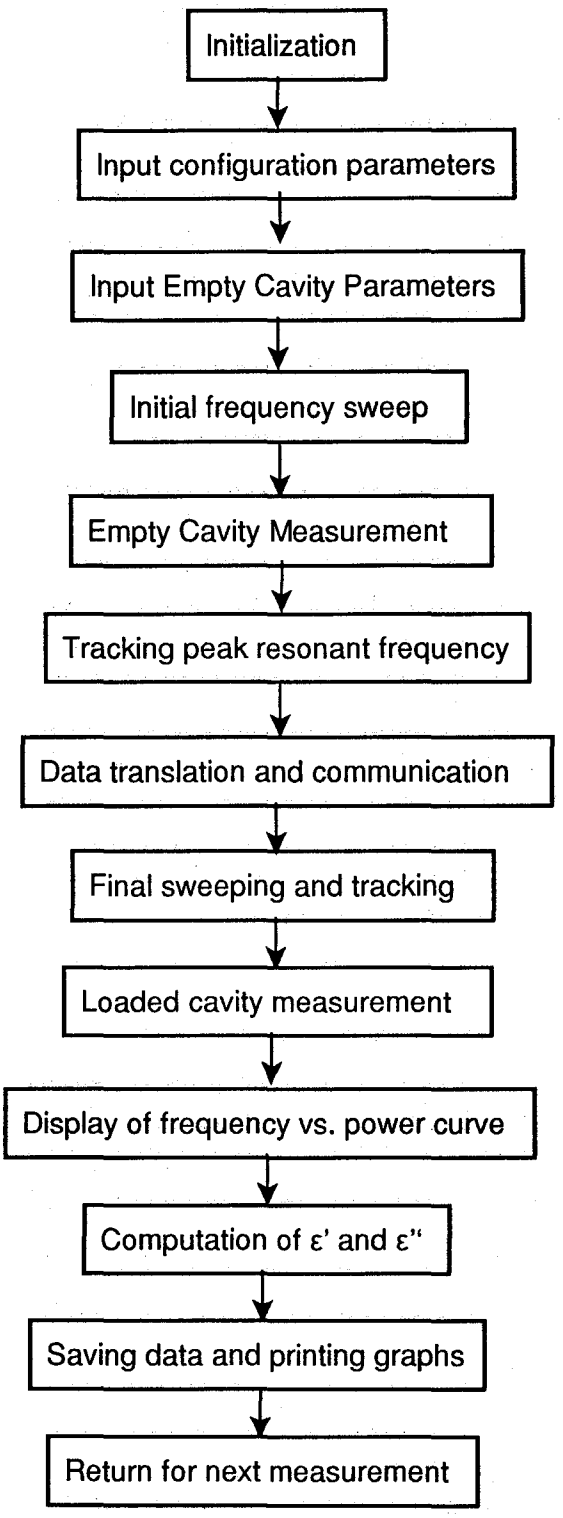


Figure 5.2 Flow diagram of the dielectric properties measurement sequence

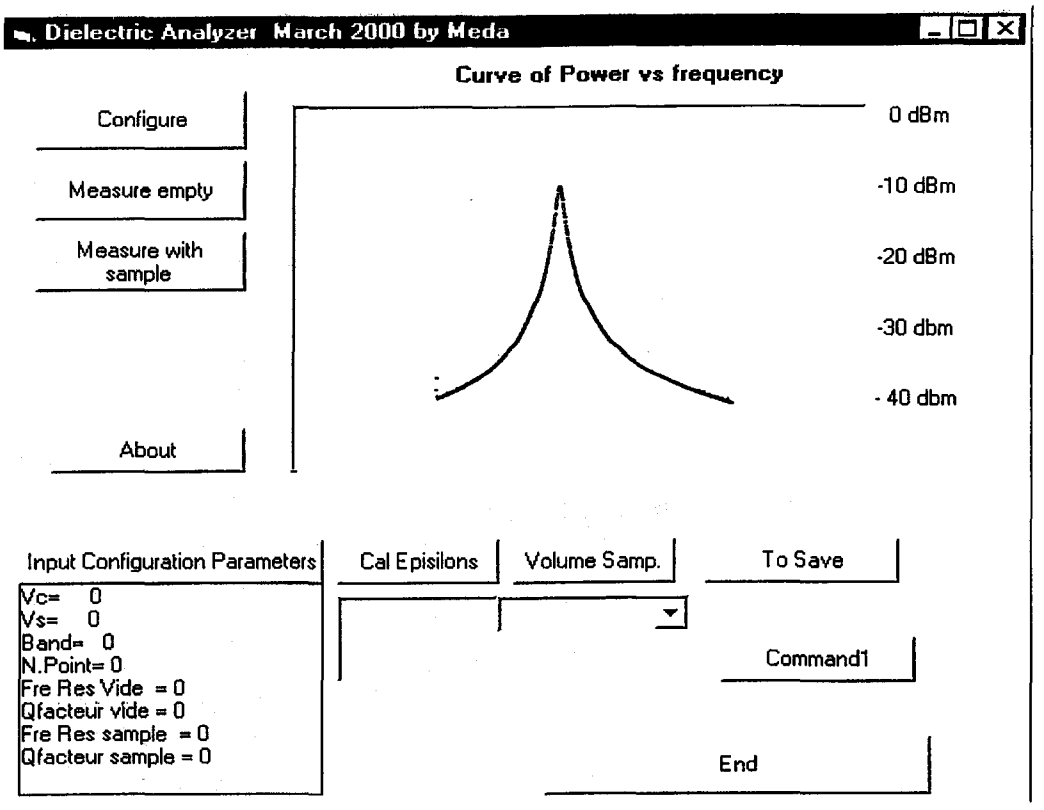


Figure 5.3 Snapshot of the main interface control software to analyze permittivity

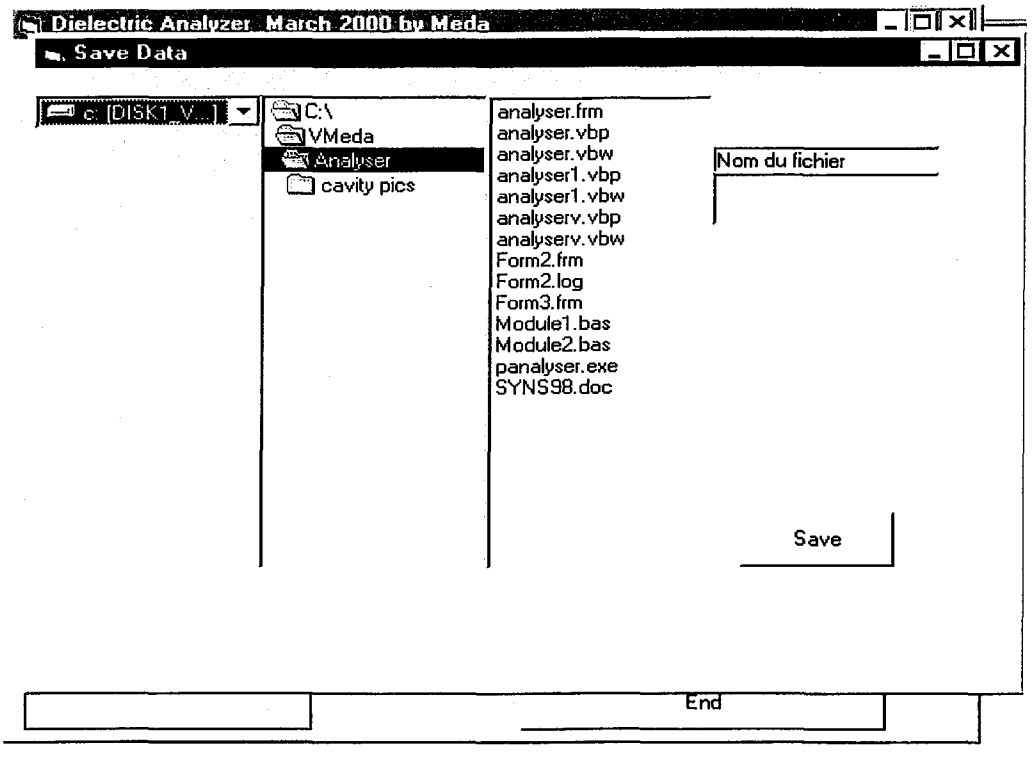


Figure 5.4 Display of an integrated data saving feature of the control software

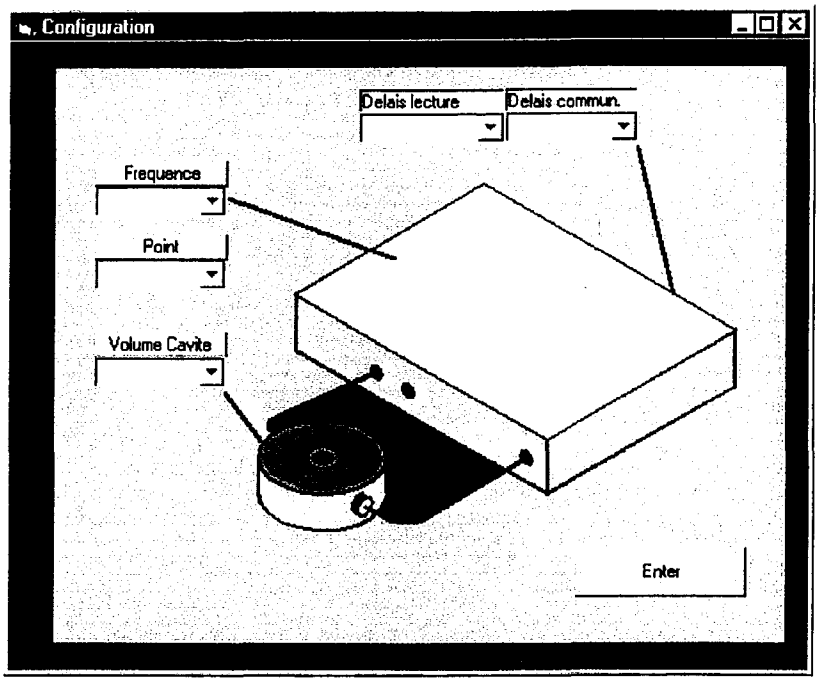


Figure 5.5 Display of initial configuration window of the control software

5.3 Calibration of Permittivity Analyzer

The standard curve (as shown in **Figure 5.6**) of power vs. frequency for distilled water revealed that the measuring system and components performed well as the observed noise level was not significantly high. The above sample was repeatedly tested to determine its peak resonance condition at 2.45 GHz frequency and room temperature. The results obtained with the permittivity analyser matched with the standard network analyser based values for similar test conditions. The resolution of each measurement was in the range of 10 - 20 kHz step size and the number of data points for each sweep was 801. In general, the lower the Q, higher the heating rate. High Q-factor defines the bandwidth (or sharpness) of the resonance curve of field intensity plotted against frequency. There is a slight scatter in the data points for water media due to noise produced by the system's electronic components however it is very minimal or absent for air (**Figure 5.7**). These checks will be made at the start of the dielectric measurements of an unknown sample and in-between measurements to verify the functioning of the unit.

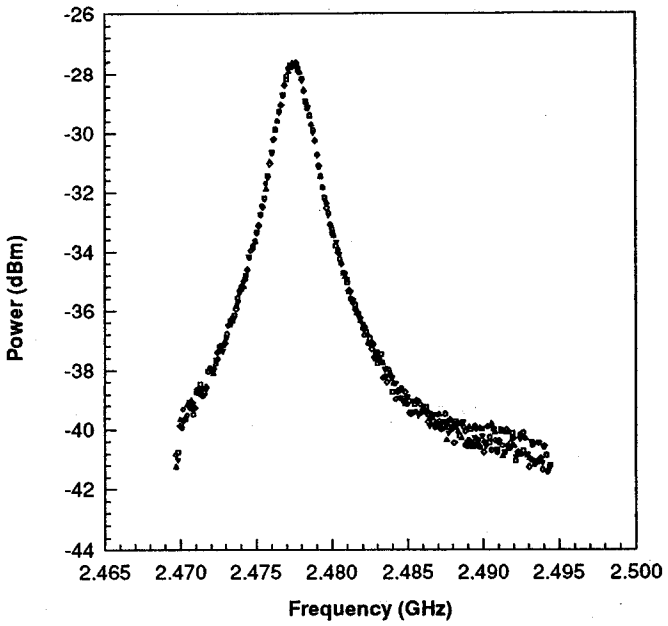


Figure 5.6 Peak resonant frequency curve for distilled water at 2.45 GHz (22°C)

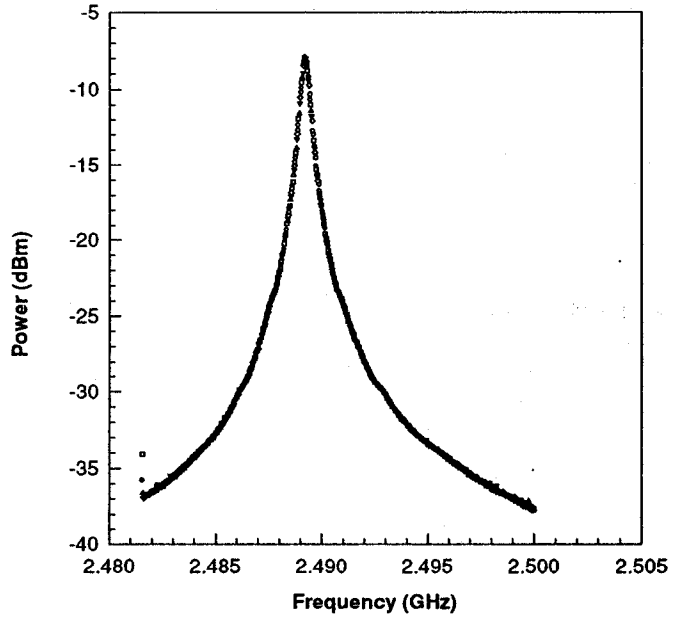


Figure 5.7 Peak resonant frequency curve for air at 2.45 GHz (22°C)

5.3.1 Factors Affecting the Measurement Conditions and Performance

The following factors and experimental difficulties were observed during the measurement and are worth to consider in carrying out the accurate permittivity analysis:

- it takes 3-6 hours to set and stabilise the system (temperature re-circulating bath) at freezing temperatures (-15 to -35°C).
- sample reaches the set temperatures in 2 to 3 min and K type thermocouple sensor can be used to check the temperature at the geometric center of the resonant cavity. The difference between the set point value and the actual temperature inside the cavity was found to be 1 to 2°C.
- heat loss can be minimized by perfect insulation, however at the sample insertion point, coupling holes, and cable connection points, they are not perfectly air tight. Condensation effect will be evident when working at low temperatures (-15°C and below). This physical change is speculated to affect the measurement values as the cylindrical core of the sample holder is enclosed by a thin ice layer (exterior) and perhaps the outer walls of the cavity could be covered with ice (wet). Detailed observation was not possible because of the enclosure and limited access space. In our set-up, there was no deposit of ice inside the cavity.
- values of f_o and Q-factor remain constant for empty cavity measurements and are saved in memory for use in computing dielectric properties of the sample. The shift in the above parameters forms the basis of calculation of dielectric constant and loss factor.
- sources of errors may be due to instability of the temperature bath (re-circulator) at sub-zero temperatures.
- electrical noise and vibration due to temperature bath placed on the same level as the electronic components.
- difficult to store and measure volatile samples at high or low temperatures (example, organic solvents)
- system errors due to voltage fluctuations (electronics)

  Data saving capability feature of the control software eliminates any transcription errors and can be recalled for comparison purposes.

5.4 Repeatability, measurement accuracy, comparison with other reported data.

This section evaluates system performance in terms of the data obtained. The first issue is the stability of measurements taken on the same sample under the same conditions in sequence. The second is that of variability of measurements taken on different samples of material of the same batch with respect to the different temperatures and frequencies. The third issue with respect to the gathered data is that of validity in terms of values reported in the literature where available. Finally, the dielectric properties of the different test materials are presented and discussed in terms of their frequency and temperature dependencies, and in light of available data from the literature.

5.4.1 Repeatability

The following procedure was used in most of the measurement sequences. The recirculating bath was set to bring the cavity to the desired temperature. A borosilicate glass micropipette (10 µl) containing the sample was then introduced into the cavity, and the sample was allowed to equilibrate to set cavity temperature for less than two minutes (in the case of temperatures above 22°C) and about five min for sub-zero temperatures. The measurement sequence was then initiated. Four scans (quadruplets) were made on the sample within 50 to 60 seconds. The micropipette was then extracted and replaced with another holding either: (i) a different sample of the same material, or (ii) a sample of another material to be tested at the existing temperature. Since the limiting factors for speed of operation are changing the desired temperature, and changing to a different frequency, it would be desirable, in practice, to work at a given frequency for as many types of materials as are to be tested in a session, and to test them all at a given temperature before resetting the desired temperature. The measurements were repeated both from coldest to warmest and warmest to the coldest range and there was no difference in the values of ε' and ε''. However, it was not possible to execute tests according to this strategy because all of the materials were not available at the same time over the course of this research.

The stability of quadruplets of dielectric constant, considered over all of the materials, temperatures and frequencies tested was excellent. In 95% of the tests, all members of a quadruplet were identical. The stability was very good for the dielectric loss factors; 84% of quadruplets having identical members. When members were not identical,

variations were nevertheless quite small (<1% of the average reading). Thus, a nominal error of less than 1% can be attributed to the system's circuitry and cavity conditions.

5.4.2 Testing of the Permittivity Analyzer

Table 5.1 shows the values of dielectric properties and cavity Q-factor of standard solvents and chemical reagents commonly used in the microwave enhanced chemistry applications and calibration materials. The values represent the average of four readings over two replicates measured using cavity perturbation technique at 2.45 GHz and ambient temperature (22-25°C). They are in agreement with the literature values obtained by (Kingston and Haswell, 1997; Giroux, 1987; Chen and Spiro, 1994; Gabriel et al. 1998). The high value of Q_o (5096) for an empty cavity measurement and the corresponding numbers for these samples indicates that the measurements tested under certain conditions are highly reliable. The variation in Q-factor for some standard solvent and chemical reagents indicates the heat dissipation qualities, i.e., methanol (ε'' =13.92) heats faster than acetone (ε'' = 0.93) or toluene (ε'' =0.01).

The standard errors are calculated for all the temperature levels and the percentage mean values of the standard errors along with the measured data are presented in the **Tables 5.2** and **5.3.**

Table 5.4 represents the numerical values of ε' and ε'' of selected agri-food materials (solids, semi-solids, particulates and liquids) determined under conditions similar to the standard materials, as above. The values obtained by using the new permittivity analyzer is found to agree with the reported values (Giroux, 1987). The standard errors are calculated and the percentage mean values along with the measured data are presented in the **Table 5.5** and **Table 5.6.**

Table 5.1 Dielectric properties and cavity Q factor of some standard solvent and chemical reagents at 22-25°C and 2.45 Ghz.

Sample	ε' (meas.)	ε' (litr.)	ε'' (meas.)	ε'' (litr.)	Q_{sample}
Distilled water	78.5	78.6[a]	12.57	9.48[a]	457
Starch -H_2O solution	81.3	-	10.36	-	485
DMF	38.5	-	5.35	-	714
Methanol	23.1	21.9[b] 24.7[c] 22.1[d]	13.92	14.6[b] 14.9[c] 16.0[d]	348
Acetone	21.9	21.4[e]	0.93	0.83[e]	2659
Methyl ethyl ketone	19.2	-	1.19	-	2327
Ethanol	7.19	7.49[f] 7.22[g]	7.24	7.1[f] 7.95[g]	629
Ethyl acetate	6.75	-	0.25	-	3854
n-amyl alcohol	3.05	3.51[h]	1.51	1.2[h]	2756
Toluene	2.83	2.67[i]	0.01	0.06[i]	5021
Fumaric acid	2.37	-	0.006	-	5021
Hexane	2.01	1.9[j]	0.009	0.009[j]	5021
Dimethyl fumarate	1.93	-	0.12	-	4966
Petroleum ether	1.84	-	0.002	-	5092

Empty cavity Q_o = 5096, ε' (meas.) = experimentally determined values, ε' (litr.) = literature value

Reported literature measurements by other techniques:

[a]Gabriel et al.(1998), [b]Gabriel et al.(1998), [c]Giroux (1987), [d]Liao et al. (2001)

[e]Gabriel et al.(1998), [f]Gabriel et al.(1998), Chen & Spiro (1994), [g]Liao et al (2001)

[h]Giroux (1987), [i]Gabriel et al.(1998), [j]Chen and Spiro (1994)

Table 5.2 Quadruplet data of ε' and standard errors as percentage of mean values for the standard solvent and chemical reagents measured at 22-25°C, f= 2450 MHz using Permittivity Analyzer. The standard error and the standard error as % of mean were calculated across all four values (eg.,for material #1: ε'= 81.26, 81.14, 81.26, 81.22) for both replicates (1,2).

Material*	Replicate	Temp. (°C)	Freq. (MHz)	ε'	ε'	Std.error	Std error as % of mean
1	1,2	22	2450	{81.26	81.14	0.028284	0.034824
1	1,2	22	2450	81.26	81.22}		
2	1,2	22	2450	{78.46	78.38	0.022174	0.028281
2	1,2	22	2450	78.42	78.36}		
3	1,2	22	2450	{39.29	38.45	0.210000	0.543197
3	1,2	22	2450	38.45	38.45}		
4	1,2	22	2450	{23.13	22.95	0.044230	0.191615
4	1,2	22	2450	23.13	23.12}		
5	1,2	22	2450	{21.79	21.92	0.068602	0.312360
5	1,2	22	2450	22.04	22.10}		
6	1,2	22	2450	{18.68	18.68	0.125000	0.664717
6	1,2	22	2450	19.18	18.68}		
7	1,2	22	2450	{07.19	07.19	0.007500	0.104203
7	1,2	22	2450	07.19	07.22}		
8	1,2	22	2450	{07.02	06.52	0.125000	1.881114
8	1,2	22	2450	06.52	06.52}		
9	1,2	22	2450	{03.05	02.95	0.048045	1.642568
9	1,2	22	2450	02.86	02.84}		
10	1,2	22	2450	{02.84	02.82	0.005000	0.176367
10	1,2	22	2450	02.84	02.84}		
11	1,2	22	2450	{02.37	02.37	0.000000	0.000000
11	1,2	22	2450	02.37	02.37}		
12	1,2	22	2450	{02.05	02.01	0.010000	0.495050
12	1,2	22	2450	02.01	02.01}		
13	1,2	22	2450	{01.94	01.93	0.004082	0.211528
13	1,2	22	2450	01.92	01.93}		

Material*: 1-Starch-Water solution, 2-Distilled water, 3-Dimethyl Formamide (DMF), 4-Methanol, 5-Acetone, 6-Methyl Ethyl Ketone, 7-Ethanol, 8-Ethyl acetate, 9-n-Amyl alcohol, 10-Toluene, 11-Fumaric acid, 12-Hexane, 13-Diemethyl Fumarate

Table 5.3 Quadruplet data of ε" and standard errors as percentage of mean values for the standard solvent and chemical reagents measured at 22-25°C, f= 2450 MHz using Permittivity Analyzer.

Material*	Replicate	Temp. (°C)	Freq. (MHz)	ε"	ε"	Std. error	Std error as % of mean
1	1,2	22	2450	{10.3617	10.1200	0.058983677	0.574562039
1	1,2	22	2450	10.3617	10.2200}		
2	1,2	22	2450	{12.5700	12.3800	0.059494397	0.477195889
2	1,2	22	2450	12.5700	012.350}		
3	1,2	22	2450	{05.3074	05.5973	0.091354461	1.706618981
3	1,2	22	2450	05.1571	05.3500}		
4	1,2	22	2450	{13.9200	13.9200	0.001700000	0.012211152
4	1,2	22	2450	13.9268	13.9200}		
5	1,2	22	2450	{00.9355	00.9368	0.008522177	0.90234284
5	1,2	22	2450	00.9355	00.9700}		
6	1,2	22	2450	{01.1900	01.1871	0.006746975	0.564836721
6	1,2	22	2450	01.2146	01.1863}		
7	1,2	22	2450	{07.2492	07.2400	0.002374167	0.032760229
7	1,2	22	2450	07.2492	07.2500}		
8	1,2	22	2450	{00.2490	00.2501	0.000275000	0.110077054
8	1,2	22	2450	00.2501	00.2501}		
9	1,2	22	2450	{01.5100	01.4065	0.047151326	3.182996965
9	1,2	22	2450	01.6024	01.4065}		
10	1,2	22	2450	{00.0158	00.0160	0.000173205	1.103217075
10	1,2	22	2450	00.0152	00.0158}		
11	1,2	22	2450	{00.0060	00.0061	6.29153E-05	1.052975514
11	1,2	22	2450	00.0058	00.0060}		
12	1,2	22	2450	{00.0090	00.0095	0.000144338	1.560406133
12	1,2	22	2450	00.0095	00.0090}		
13	1,2	22	2450	{00.1239	00.1231	0.000230940	0.186996039
13	1,2	22	2450	00.1239	00.1231}		

Material*: 1-Starch-Water solution, 2-Distilled water, 3-Dimethyl Formamide (DMF), 4-Methanol, 5-Acetone, 6-Methyl Ethyl Ketone, 7-Ethanol, 8-Ethyl acetate, 9-n-Amyl alcohol, 10-Toluene, 11-Fumaric acid, 12-Hexane, 13-Diemethyl Fumarate

Table 5.4 Dielectric properties of selected agri-food and MW transparent materials.

Sample	ε'(meas.)	ε'(litr.)	ε"	ε"(litr.)
Solid				
Teflon™ (88%)	2.74	2.62[a]	0.0003	0.00032[a]
Borosilicate glass (micropipette /corning)	4.65	4.28[b]	0.008	0.006[b]
Semi-solid				
Wax (plastic)	2.01	-	0.007	-
Particulates				
Clay soil (12% m.c.)	2.44	2.51[c]	0.14	0.15[c]
Loamy soil (7% m.c.)	7.01	7.15[d]	0.05	0.08[d]
Grape seed powder - (freeze dried)	3.57	-	0.26	-
Edible Oils				
Corn oil	2.59	2.61[e]	0.13	0.14[e]
Sunflower oil	2.51	2.49[f]	0.18	0.18[f]

Empty cavity $Q_o = 5096$, ε' (meas.) = experimentally determined values, ε'' (litr.) = literature value.

Reported literature measurements (other techniques):

[a]Giroux (1987), HP Product Information, [b]Gabriel et al.(1998), [c]Breccia et al.(1987), [d]Breccia et al.(1987), [e]Bengtsson and Risman (1971), [f]Akyel et al.(1983).

Table 5.5 Quadruplet data of ε' and standard errors as % of mean values for the standard agri-food and MW transparent materials measured at 22-25°C, f= 2450 MHz using Permittivity Analyzer. (Material*: 1-Teflon™, 2-Borosilicate glass, 3-wax, 4-Clay soil (12% m.c.), 5-Loamy soil (7% m.c.), 6-Grape seed powder, 7-Corn oil, 8-Sunflower oil)

Material*	Rep	Temp. (°C)	Freq. (MHz)	ε'	ε'	Std.Err	Std error as % of mean
1	1,2	22	2450	{2.74	2.62	0.028284	1.047566
1	1,2	22	2450	2.74	2.70}		
2	1,2	22	2450	{4.65	4.28	0.101366	2.265155
2	1,2	22	2450	4.65	4.32}		
3	1,2	22	2450	{2.01	2.01	0.000000	0.000000
3	1,2	22	2450	2.01	2.01}		
4	1,2	22	2450	{2.44	2.51	0.021747	0.868997
4	1,2	22	2450	2.54	2.52}		
5	1,2	22	2450	{7.01	7.15	0.040415	0.570827
5	1,2	22	2450	7.01	7.15}		
6	1,2	22	2450	{3.57	3.57	0.002500	0.069979
6	1,2	22	2450	3.57	3.58}		
7	1,2	22	2450	{2.59	2.61	0.005774	0.222058
7	1,2	22	2450	2.59	2.61}		
8	1,2	22	2450	{2.51	2.49	0.005774	0.230940
8	1,2	22	2450	2.51	2.49}		

Table 5.6 Quadruplet data of ε'' and standard errors as % of mean values for the standard agri-food and MW transparent materials measured (22-25°C,f=2450 MHz using Permittivity Analyzer.

Material*	Rep	Temp (°C)	Freq (MHz)	ε''	ε''	Std. Err	Std Err as % of mean
1	1,2	22	2450	{0.0003	0.00032	4.78714E-06	1.556792045
1	1,2	22	2450	0.0003	0.00031}		
2	1,2	22	2450	{0.0080	0.00810	6.29153E-05	0.7889064200
2	1,2	22	2450	0.0080	0.00780}		
3	1,2	22	2450	{0.0070	0.00700	0.000375000	5.8823529410
3	1,2	22	2450	0.0055	0.00600}		
4	1,2	22	2450	{0.1400	0.15000	0.002500000	1.6949152540
4	1,2	22	2450	0.1500	0.15000}		
5	1,2	22	2450	{0.0500	0.05200	0.001181454	2.2830027180
5	1,2	22	2450	0.0500	0.05500}		
6	1,2	22	2450	{0.2600	0.26000	0.002500000	0.9708737860
6	1,2	22	2450	0.2500	0.26000}		
7	1,2	22	2450	{0.1300	0.13000	0.002500000	1.8867924530
7	1,2	22	2450	0.1400	0.13000}		
8	1,2	22	2450	{0.1800	0.18000	0.001493039	0.8329369070
8	1,2	22	2450	0.1750	0.18200}		

5.5 Variability across Samples at Different Frequencies and Temperatures

In order to gain some insight into the variability one might expect in an experimental study of dielectric properties or in a quality control or monitoring situation, the variability of the dielectric constant and of the loss factors of milks and edible oils were evaluated in the following manner. The average of each dielectric property was assigned to the readings (quadruplets) obtained from the sample in a given micropipette (section 5.4.1). For each combination of material, frequency and temperature, the standard error ($SE = s\, n^{-1/2}$, where s is the standard deviation and n is the number of observations) of the quadruplet averages was determined and then normalized by dividing by the mean of the samples at that combination (usually 4 or 6 samples). The normalized standard error is hereafter denoted as NSE. These calculations were done directly in the Microsoft Excel (2000) spreadsheets containing the raw data. (Note: The standard errors were normalized to permit comparison across the different combinations of material, frequency and temperature since the magnitudes of dielectric constant and loss factor ranged from the order of 10^{-2} to the order of 10^{2}. The NSE is completely analogous to that of the coefficient of variability, which is used to compare the precision of experiments).

Histograms of the NSE's of ε' and ε'' for measurements made on milks at three different fat contents and on the three edible oils (soya, sunflower, canola) are presented in **Figures 5.8a,b** and **5.9a,b**. The average of the NSE's and 95% confidence limits are noted on the figures since they can be used to estimate confidence limits on the average of ε' and ε'' at a given combination of frequency and temperature. Although the same procedure was used in investigations of the data sets on ethanol-hexane mixtures, neem oil and neem pulp, the data sets for these substances were not complete for reasons mainly due to inconsistent values of ε' & ε'' obtained at certain test conditions. Thus, although the histograms for these materials are also presented with the averages and confidence limits of the NSE's (**Figures 5.10a,b to 5.12a,b**), their associated statistics cannot be considered reliable and are not to be used for estimates of precision at this stage.

There are two clear features in these histograms. First, they are distinctly skewed to the left. The vast majority of NSE's corresponding to the dielectric constant (Fig.a) of each set are of magnitude less than 1% of the mean value of ε' at a given combination of temperature, frequency and subclass (eg. milk fat percentage, fatty acid composition of edible oil, etc.). In itself, this is very encouraging in terms of the level of precision one might expect in estimating the dielectric constants of homogeneous materials such as the milks

and edible oils. Furthermore, these tendencies are quite similar for high (milk), intermediate (edible oils) and low (hexane-ethanol mixtures) loss materials. The greatest degree of variability in the distribution of NSE's corresponding to ε' occurs in the cases of neem oil and neem pulp which were the least homogeneous of the substances tested. Of all the liquid samples tested, the neem oil was the least homogeneous (**Fig. 5.11a**), although it was certainly more homogeneous than the neem kernel pulp (**Fig. 5.12a**). Visually, the neem oil could be described as a turbid or cloudy slurry of oil with some suspended particles, not resembling at all the highly refined commercial edible oils tested.

The second important feature of the histograms is that the variability of the NSE's of ε" measured on a substance is clearly greater than that of the NSE's of ε' on the same substance (except in the most extreme case of NSE variability which was that of neem pulp – here the variabilities are about equal). The clearest example is the difference for milk (see **Fig. 5.8a,b**). The NSE's corresponding to ε" cluster mainly over the range 0-1.8% of its mean, whereas those corresponding to ε' cluster over a narrower region (0-0.8%) of the mean. The question arises as to why the loss factor exhibits greater variability than the dielectric constant under the same range of conditions.

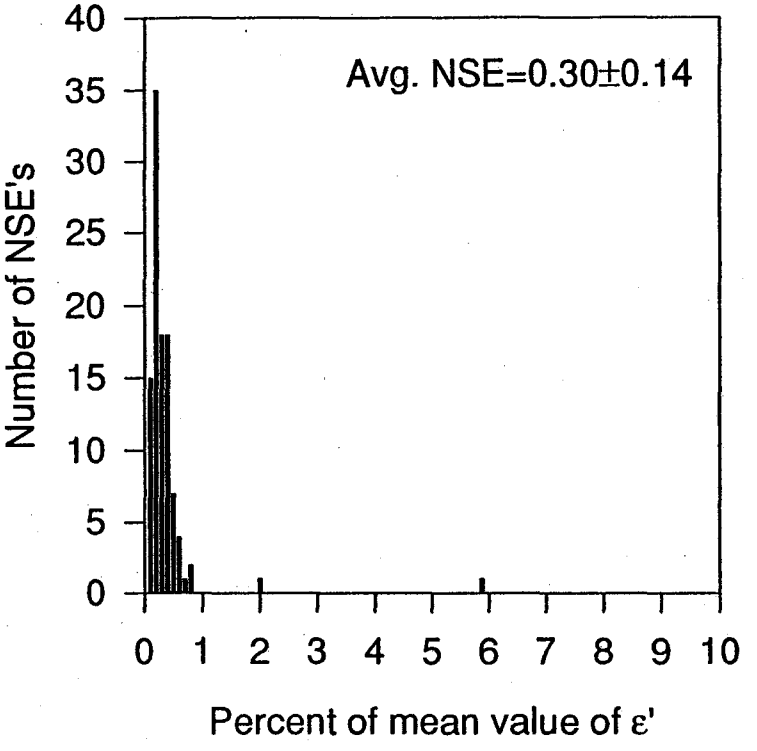
The difficulties associated with accurate determination of ε' have been discussed in terms of the effects of introducing a sample into the cavity on the validity of equation by Chen et al. (1996). One of the assumptions underlying this equation is that the empty cavity Q_o and the Q_s for the cavity with the sample, are measured at the same frequency and at the same coupling conditions The authors demonstrate that the degree of coupling between cavity and transmission line is altered by introduction of the sample, as is the frequency of the signal. They present experimental results that indicate the significance of re-tuning and adjusting the coupling on the calculated loss factors. These considerations do not apply to calculation of the dielectric constant. Thus, with respect to **Figures 5.8a,b-5.12a,b** which involve samples with different properties and or at different temperatures, it may be that the NSE's associated with the loss factors are more scattered because of a wide range of interactions of sample with the coupling coefficient and resonant frequency. **Table 5.6a** represents the extracted and measured values of ε' & ε" of selected and available values reported in the literature for temperatures below 0°C (-30 to 0°C) for certain foods & chemicals. It is observed that majority of samples (except oils) exhibited a sharp increase in their epsilon (ε' & ε") values obtained in the range of -20°C & 0°C, with other techniques and similar conditions (freq., tempr.). It is difficult to compare the absolute values obtained in this research as the test conditions vary from one study to the other.

Table 5.6a **Extracted dielectric data obtained using other techniques**

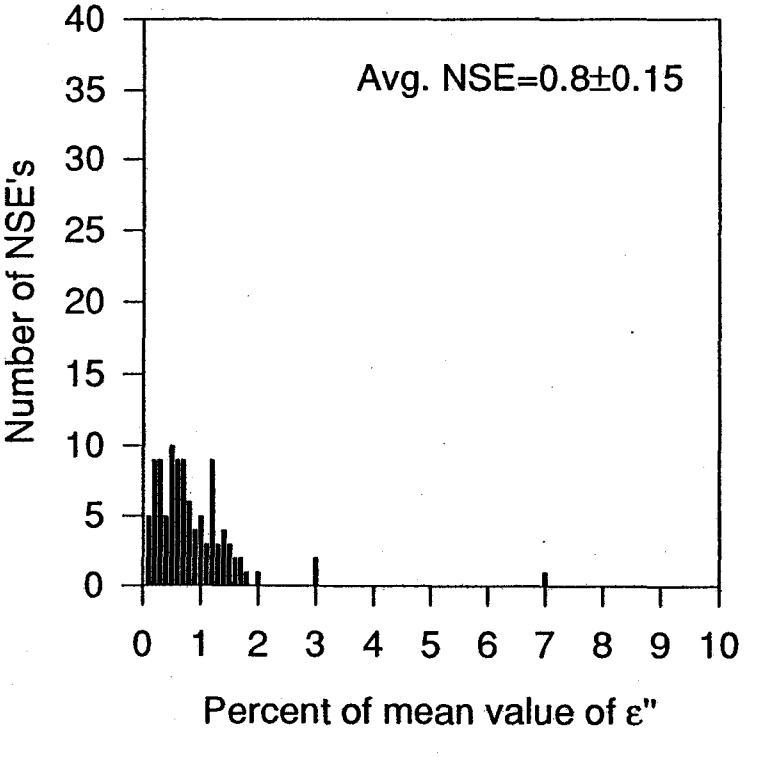
#	Sample	Frequency (MHz)	Temperature (celsius)	D. constant	D. loss factor	(Literature) Reference
1	Pure water	2450	-30	3.2	0.027	Giroux (1987)
		2800	-20	4.9	Not reported	Calay etal. (1998)
		2450	22	73	21.9	Giroux (1987)
2	Ice	2450	-12	3.2	0.003	Schiffman, 93
3	Milk powder	2450	-10	2.24	0.045	Giroux (1987)
4	Cooked peas	2800	-20	4.1	0.29	"
		2800	3	64.3	22	Giroux (1987)
5	Cooked Fish	900	-20	4.5	0.61	
	(m.c.=75%)	900	-10	5.5	1.1	"
		900	3	54.2	17.1	"
6	Cooked carrot	2800	-20	4.2	0.34	Rao & Rizvi (1995)
	(m.c.=88%)	2800	3	75.6	25.4	
7	Mashed potato	900	-20	3.8	0.28	"
	(m.c. =81%)	900	3	71.5	18.9	
		3000	-20	4.6	0.32	
		3000	3	66.7	25.5	
8	Vegetable soup	2800	-20	3.9	0.29	Giroux (1987)
	(m.c.=85%)	2800	-10	5.1	0.9	
		2800	0	74.7	23.9	
9	Corn oil	3000	-10	2.57	0.103	"
	(refined)	3000	20	2.63	0.149	
10	Gravy	3000	-20	6.8	1.6	"
		3000	-10	10	3.9	
		3000	0	44.3	22.4	
11	Milk	2450	-30	3.84	0.1447	Measured values
	(1% fat)	2450	-20	37.25	49.75	using CPT
		2450	-10	68.6	64.55	
		2450	0	72.1	27.35	
		915	-30	66.2	14.25	"
		915	-20	72.35	14.1	
		915	-10	31.2	15.25	
		915	0	42.9	9.28	

Table 5.6a **Extracted dielectric data obtained using other techniques**

#	Sample	Frequency (MHz)	Temperature (celsius)	D. constant	D. loss factor	(Literature) Reference
12		915	-20	72.62	15.75	"
		915	-10	33.75	16.35	
		915	0	41.65	9.97	
13	Milk	2450	-30	3.95	0.1447	
	(3% fat)	2450	-10	65.82	56.75	"
		2450	0	69.45	25.65	
		915	-30	66.5	14.05	
		915	-10	31.1	15.62	
		915	0	41.15	9.98	
14	Soya oil	2450	-25	6.25	0.117	"
		2450	-10	7.02	0.159	
		2450	0	11.85	0.303	
	Sunflower oil	2450	-25	6.27	0.121	"
		2450	-10	7.04	0.171	
		2450	0	11.75	0.32	
	Canola oil	2450	-25	0.08	0.058	"
		2450	-10	0.81	0.08	
		2450	0	11.35	0.249	
15	Tylose	2450	-20	5.1	2.5	Chamchong et al. '98
	(3% salt)	2450	-10	14.5	7.81	
		2450	0	60.56	34.65	
16	Methanol	2450	-20	14.75	12.59	Gabriel et al. 1998
		2450	-10	17.85	14.25	
		2450	0	20.9	16.46	
17	Propanol	2450	-20	6.33	1.34	"
		2450	-10	6.94	1.65	
		2450	0	6.97	2.08	

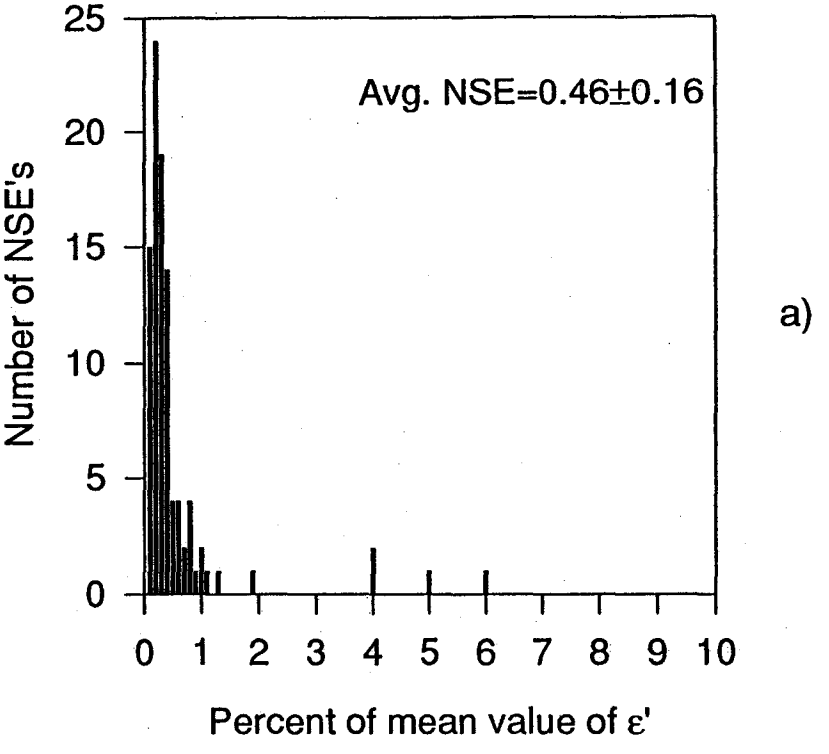


a)



b)

Figure 5.8 NSE frequency and % mean value of ε' and ε" of milk



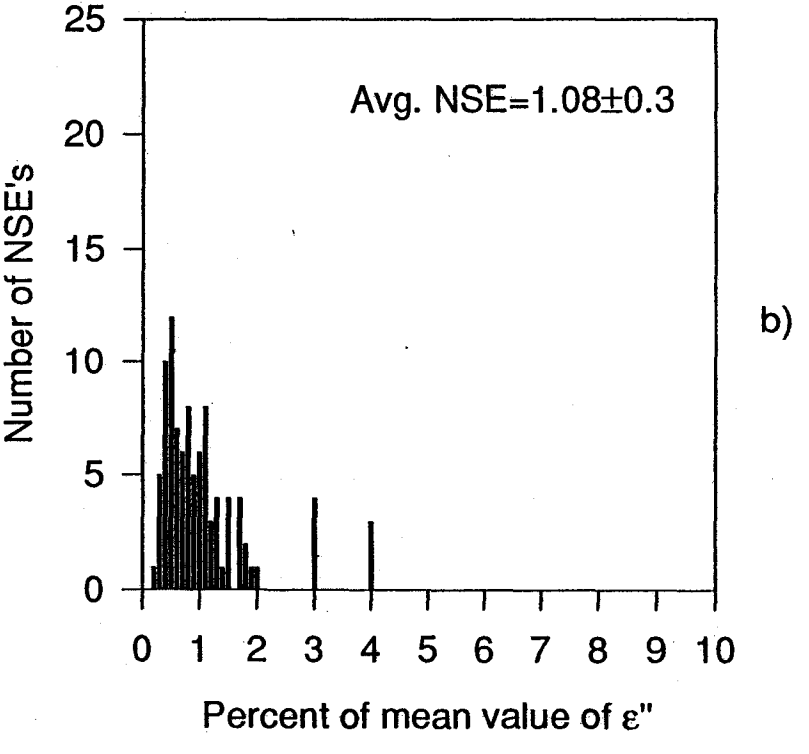
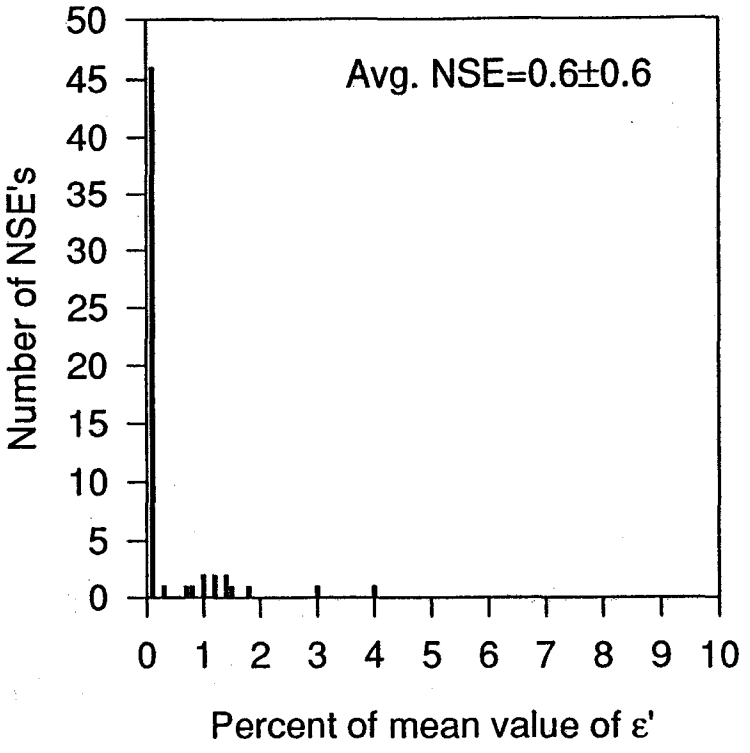
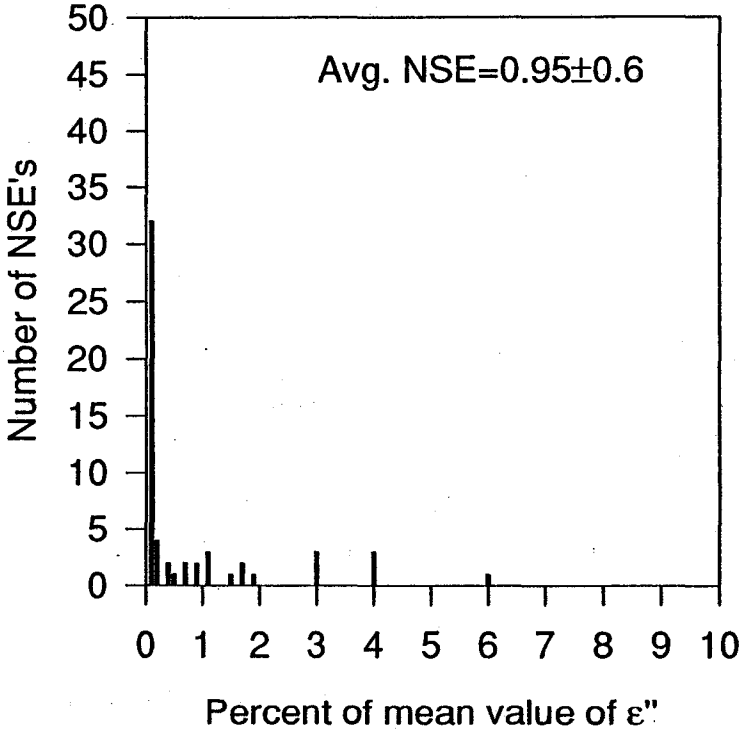


Figure 5.9 NSE frequency and % mean value of ε' and ε" of oils



a)



b)

Figure 5.10 NSE frequency and % mean value of ε' and ε" of Ethanol, hexane mixture

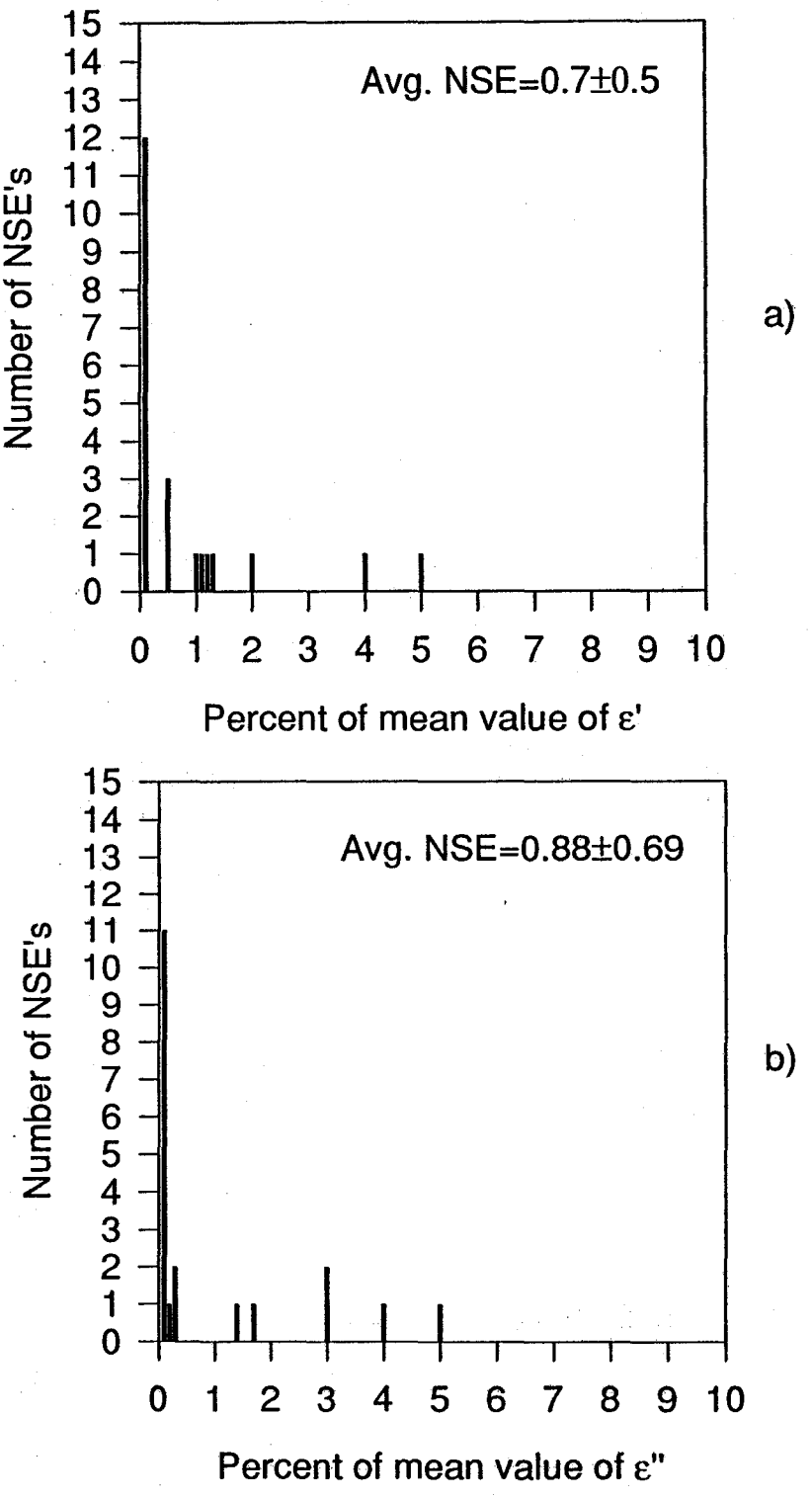


Figure 5.11 NSE frequency and % mean value of ε' and ε" of neem oil

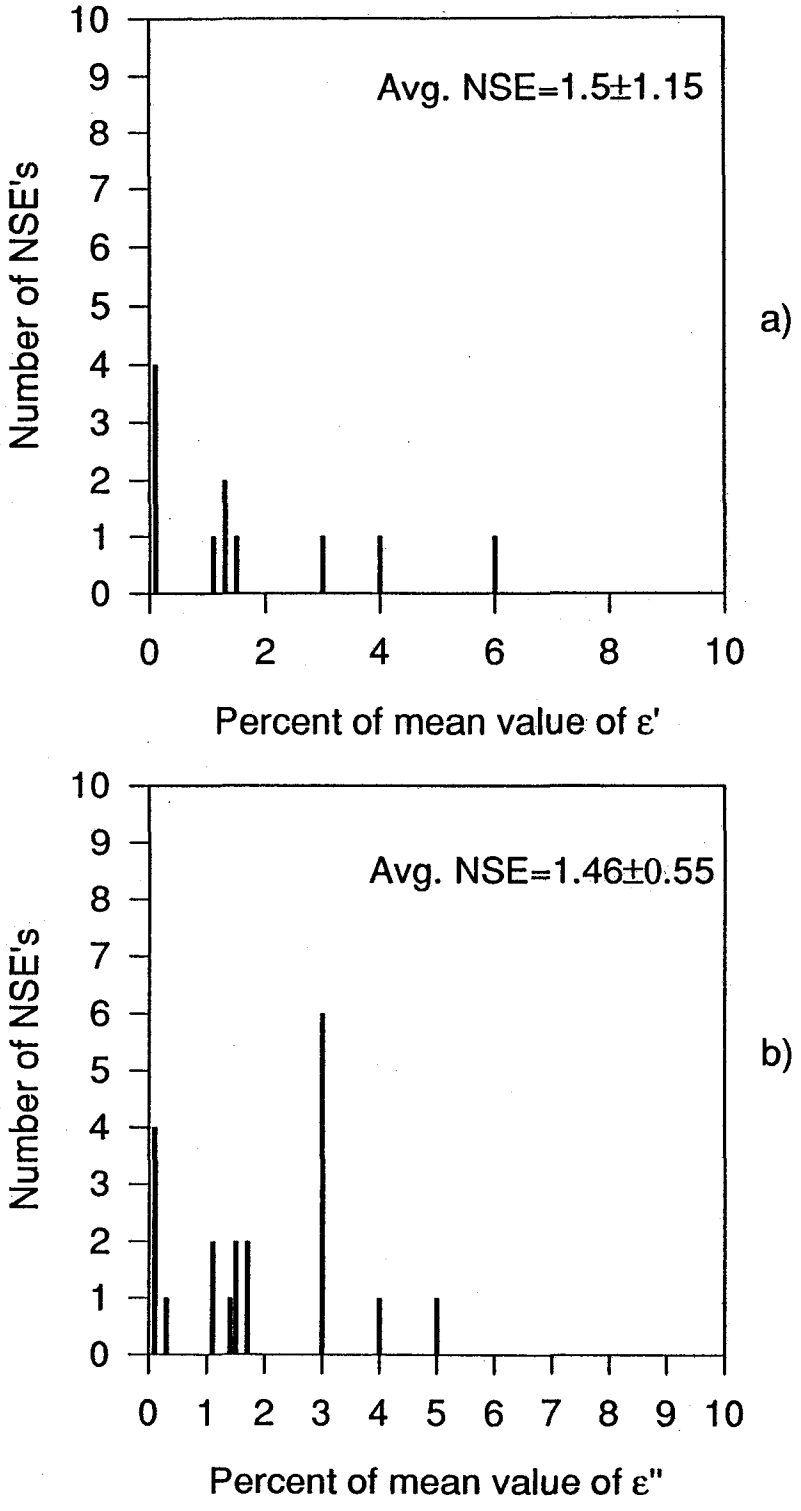


Figure 5.12 NSE frequency and % mean value of ε' and ε" of neem pulp

The average NSE's are reported in the figures with twice their own standard errors relative to all the data corresponding to a given class of substance (milk, edible oil, etc.). This gives the 95% confidence limits on the mean NSE's. The average SE's and their standard errors are reported in **Table 5.7a,b** for various materials. The implicit assumption is that they are minimally dependent of temperature, frequency or composition (eg. percent milk fat, fatty acid distribution for the edible oils, ratio of ethanol to hexane) and can therefore be used as global error 'estimates' for the different class of substances. The idea here is that for a specific combination of substance composition (milk fat %, fatty acid composition of edible oil, ethanol:hexane ratio), frequency and temperature, the upper and lower confidence limits of the appropriate mean SE can be associated with the mean value of the dielectric property in question. To be conservative, the error to attach to the mean value of the dielectric property at a specific combination of factors is twice the upper limit of the SE.

Table 5.7 Standard error and confidence limits for the classes of substances studied.

(a) dielectric constant (ε')

Sample	Avg. SE	Upper limit	Lower limit	Max. range*
Milks	0.16	0.18	0.14	±0.36
Edible Oils	0.03	0.036	0.024	±0.072
Ethanol, hexane mixture	0.1	0.15	0.05	±0.3

*applicable to means at specific combination, SE = standard error

(b) dielectric loss factor (ε'')

Sample	Avg. SE	Upper limit	Lower limit	Max. range*
Milks	0.13	0.15	0.11	±0.30
Edible Oils	0.003	0.004	0.002	±0.008
Ethanol, hexane mixture	0.024	0.036	0.012	±0.072

*applicable to means at specific combination, SE = standard error

5.5.1 Dielectric Properties of Various Substances with Permittivity Analyzer

5.5.1a Homogensied Milk

The dielectric constant and the dielectric loss factor for milk at three levels of milk fat and two frequencies are presented in **Figures 5.13** and **5.14.** The dielectric constant (Figure 5.13a) and the dielectric loss factor (5.14a) exhibited similar responses to temperature and % fat over the range studied, both being very small in the range of –28 to -30°C and then rising sharply (2450 MHz) upto 40°C (data points); after which it decreases gradually. In the case of ε", there is a sharp decrease from -10°C to 0°C and a slow downward trend thereafter. It is not clearly understood why there is a huge difference for loss factor of milk at –10 and –5°C (2450 MHz). If compared to the value of loss factor for water at –10°C (37), it is seen that it is far different than the value of 75 obtained here. This difference is attributable to the differences in physical status of water and milk. It is speculated that milk cannot be considered frozen at these temperatures whereas water would definitely be considered frozen. However, the loss factor drops thereafter. From the literature it is found that ε" value for milk to be around 18 (Kudra et al., 1992; Mudgett et al., 1974) at 20°C which agrees with the value obtained in this study for the same condition (Figure 5.14a).

The ε' and ε" values at varying temperatures for 915 MHz can be seen in Figures 5.13b and 5.14b. The data here also shows inconsistencies below 0°C as expected and there is no literature data available to compare. However, both the ε' and ε" follows an increasing trend with temperature which are opposite to the trend observed for 2450 MHz.

5.5.2 Edible Oils

The dielectric constant and dielectric loss factor for three different edible oils (soya, sunflower and canola) at two frequencies (915 and 2450 MHz) are presented in **Figures 5.15 and 5.16**. All the three oils, viz, soya, sunflower and canola have shown similar responses at microwave frequency of 2450 MHz. Whereas at 915 MHz, the values of ε' (Fig. 5.15b) and ε" (Fig. 5.16b) for all the three oils were found to be inconsistent below 25°C, possibly due to contribution of the constituents (saturated fatty acids) of the sample. As we know, oils and fats are made up of fatty acids of glycerol and come from both plant and vegetable sources and have important functional and nutritional properties in agri-foods. Monoglycerides and diglycerides decompose at temperatures ranging from 160°C to 190°C (Potter et al., 1996).

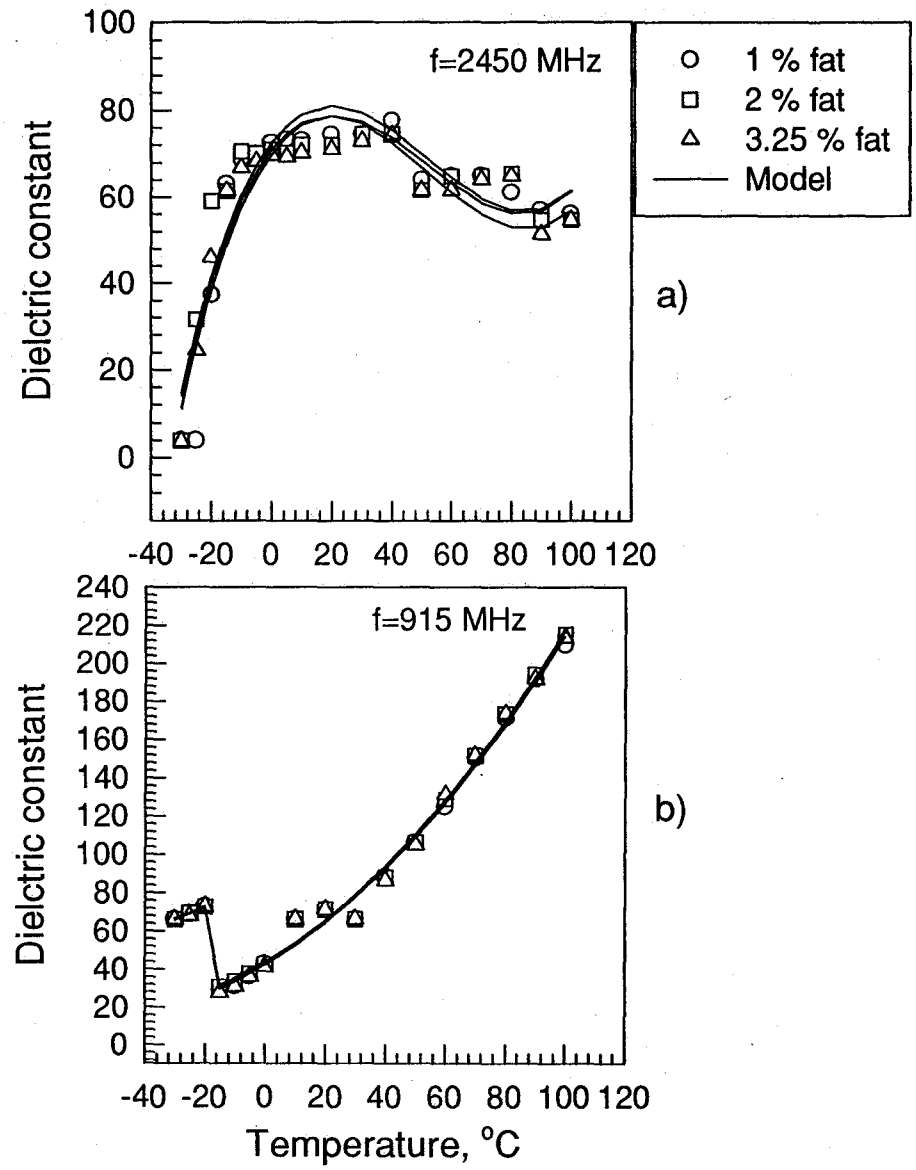


Figure 5.13 Functional relationship of dielectric constant vs. temperature of milk (1%, 2%, and 3,25% fat) at 2450 MHz and 915 MHz.

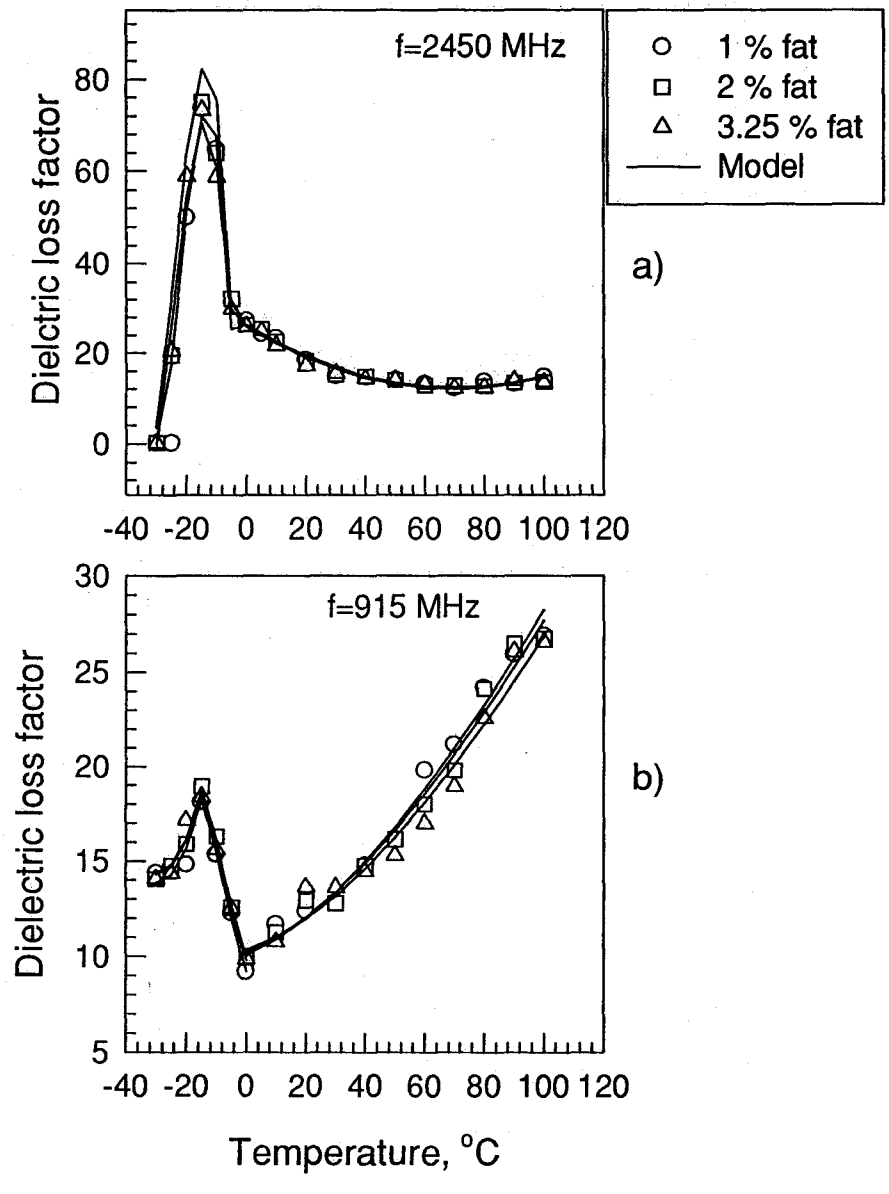


Figure 5.14 Functional relationship of dielectric loss factor vs. temperature of milk (1%, 2%, and 3,25% fat) at 2450 MHz and 915 MHz.

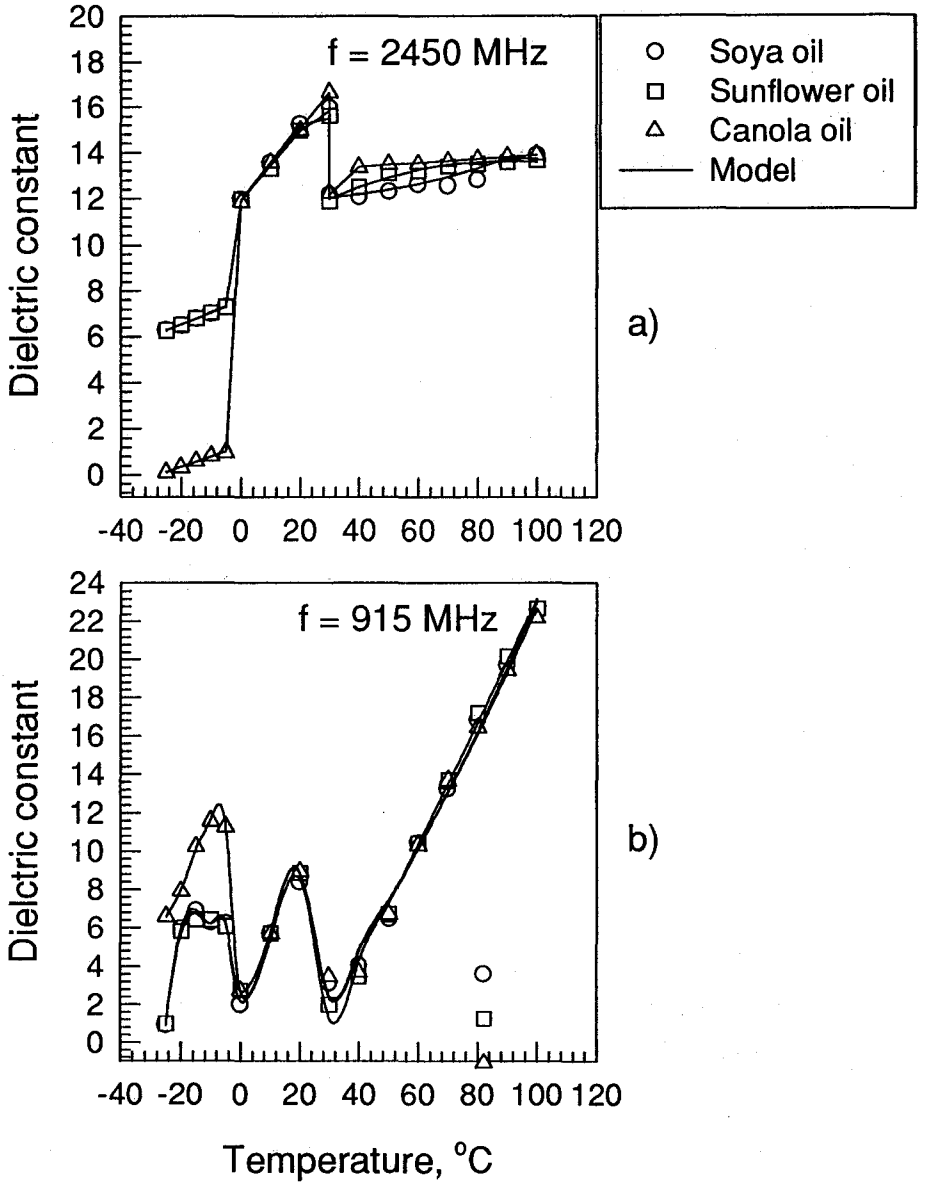


Figure 5.15 Functional relationship of dielectric constant vs. temperature of oils (soya, sunflower and canola) at 2450 MHz and 915 MHz.

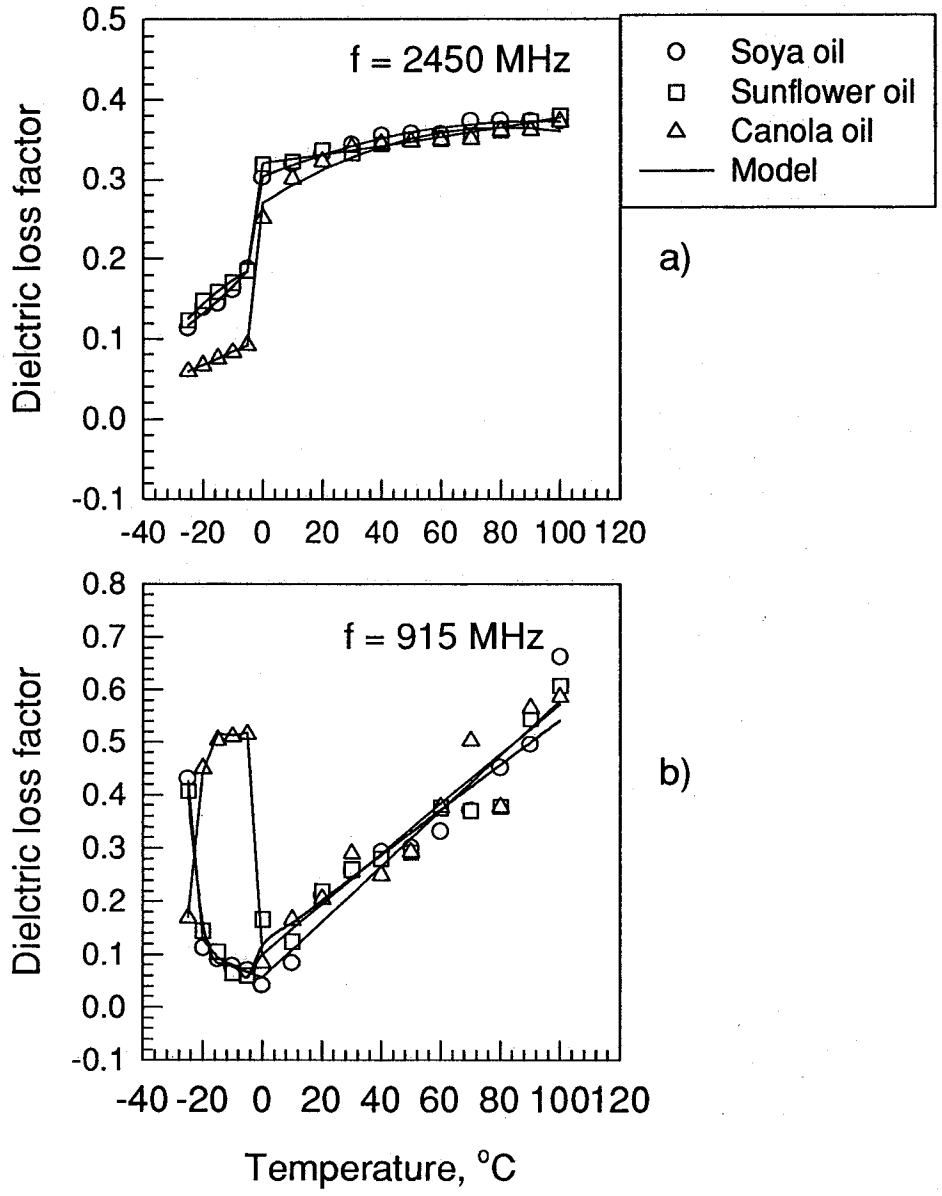


Figure 5.16 Functional relationship of dielectric loss factor vs. temperature of oils (soya, sunflower and canola) at 2450 MHz and 915 MHz.

Since the dielectric properties of different oils exhibited radically different responses to frequency and temperature in this study, future efforts should be concentrated on studying the fatty acid compositions and determining what additives may be present due to the types of extraction and preservation methods used for each oil in order to explain the odd behavior. Pace et al.(1968) reported data on 11 commercial cooking oils and this data compares well with the data obtained here at similar frequencies and temperature ranges (25 to 100°C). Canola oil behaved differently than the other two types in the 0 to -20°C at 915 as well as at 2450 MHz The data below sub-zero temperatures for these oils is not easily available in the literature. When suspected factors are identified, specific studies should be performed to elucidate the mechanisms. This however is outside the scope of this study.

5.5.3 Ethanol, hexane mixtures

Organic solvents such as ethanol and hexane were mixed in various proportions (10, 30, 50, 70, 90 and 100% ethanol) and the dielectric properties were determined at both frequencies. The behaviour of ε' and ε" of ethanol and the mixture of ethanol and hexane, at temperatures ranging from 22°C to 62°C was determined. All the measurements were carried out at both 915 and 2450 MHz frequencies. The flash point (65°C) of these mixtures did not allow permittivity measurements beyond the presented range as the sample boiled off before the insertion into the cavity. For 2450 MHz, the values of ε' decreased as the % of ethanol decreased (as shown in **Figure 5.17a**). The same trend can be observed with respect to ε". Samples of pure ethanol (100%) exhibited higher values for both ε' and ε". The trends of ε' with temperature were identical for ethanol concentrations of 10 to 30% and were flat from 37 to 62°C. At higher concentrations of ethanol, ε' increased with temperature. The dielectric loss factor, ε'' increased with temperature for mixtures containing greater than 50% ethanol by volume (**Figure 5.17b**). However, as the ratio of ethanol in the mixture decreased below 50%, the dielectric loss factor stayed essentially the same for different temperatures. These results also show that, over the temperature range studied, both the dielectric constant and loss factor decreased linearly as the volumetric ratio of hexane in the mixture increased. The data compares well with the literature data reported by Punt (1997), using VNA based measurement approach and the data reported by Gabriel et al.(1998), and Giroux (1987).

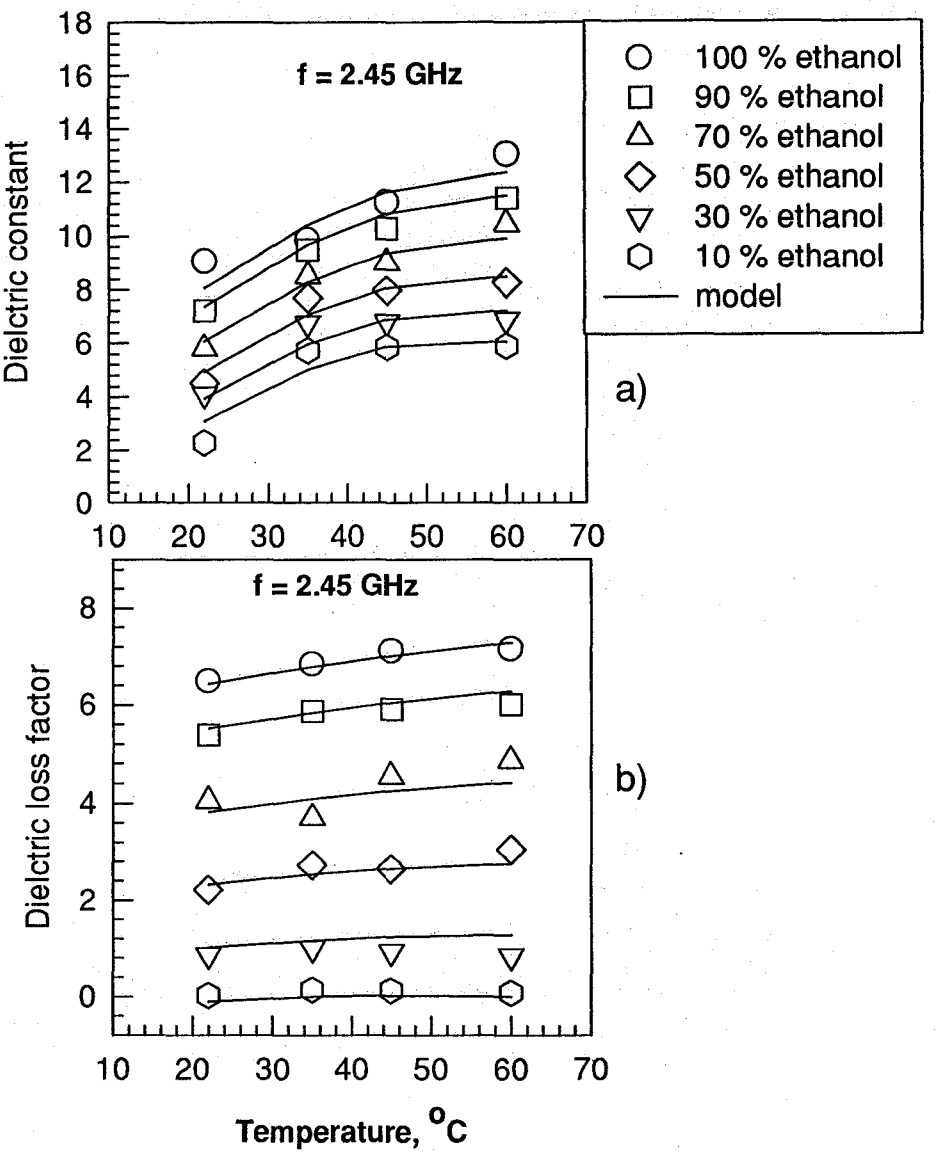


Figure 5.17 Functional relationship of dielectric constant and loss factor vs. temperature of ethanol and hexane mixtures at 2450 MHz.

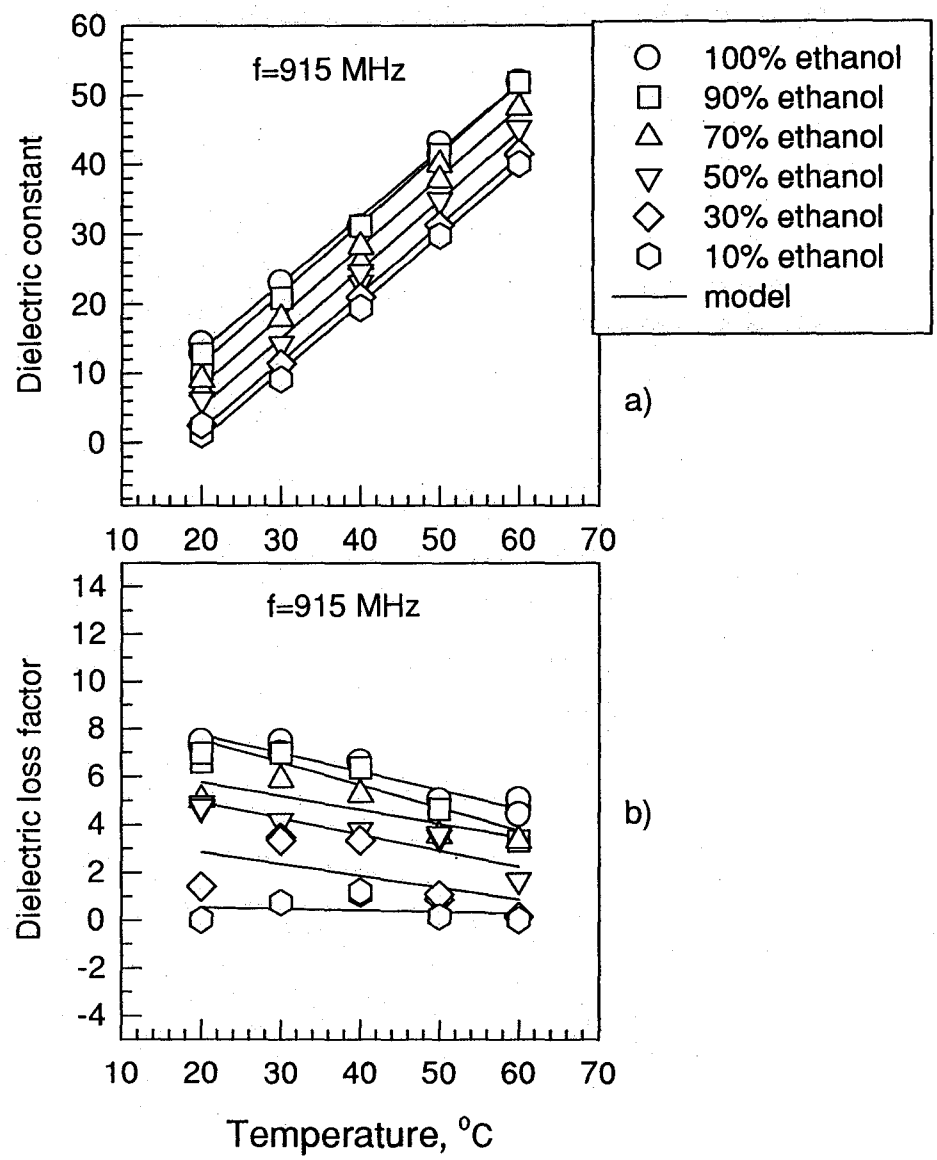


Figure 5.18 Functional relationship of dielectric constant and loss factor vs. temperature of ethanol and hexane mixtures at 915 MHz.

In the case of 915 MHz, the ε' increased linearly with temperature and decreased with decrease in ethanol concentrations (**Figure 5.18a**). The trends of ε' with temperature were identical for all the concentrations. However, ε'' decreased with temperature (as shown in **Figure 5.18b**) and also with decreasing ethanol concentrations. The trends of ε'' with temperature were identical for ethanol concentration in the range of 50-100%; however, ε'' increased slightly in the range of 20°C to 40°C (for 10 - 30% ethanol concentrations). The dielectric loss factor values did not change much in the range 40 - 50°C; however, it decreased gradually thereafter until it reached the flash point (65°C). There is no literature available for mixtures of ethanol and hexane measured at 915 MHz and similar conditions.

5.5.4 Behaviour of Neem Oil and Pulp

Plant based materials such as neem oil and pulp were tested for their dielectric constant and loss factors, at 2.45 GHz frequency and 0-100°C temperature range (**Figure 5.19a,b**). The preparation of neem pulp sample was very difficult because of the nature of the bio-ingredients of neem, as reported by Dai et al.(2000). Both dielectric constant and dielectric loss factor increased linearly with temperature; however, the values of oil was lower than that of pulp as there was no contribution of moisture and organic substances contained in the pulp. The ε' ranged from 1.5 to 6.0 for oil and 3.5 to 10 for the pulp. The ε'' of pulp increased with temperature. The relationship was non-linear as shown in Fig. 5.19(b).

5.6 Economic aspects and advantages

In this research study, the permittivity analyzer was developed and found to be simple, easy and flexible to operate. It is a unique, dedicated equipment and portable compared to the commercially available Vector Network Analyzers (VNAs).

5.6.1 VNA vs Permittivity Analyzer (PA) Comparison

The components and their specifics are discussed in the Materials and Methods Chapter. All the essential electronic components that are directly responsible to measure dielectric parameters are commercially available. The approximate costs of electronic components used for Permittivity Analyzer are shown below:

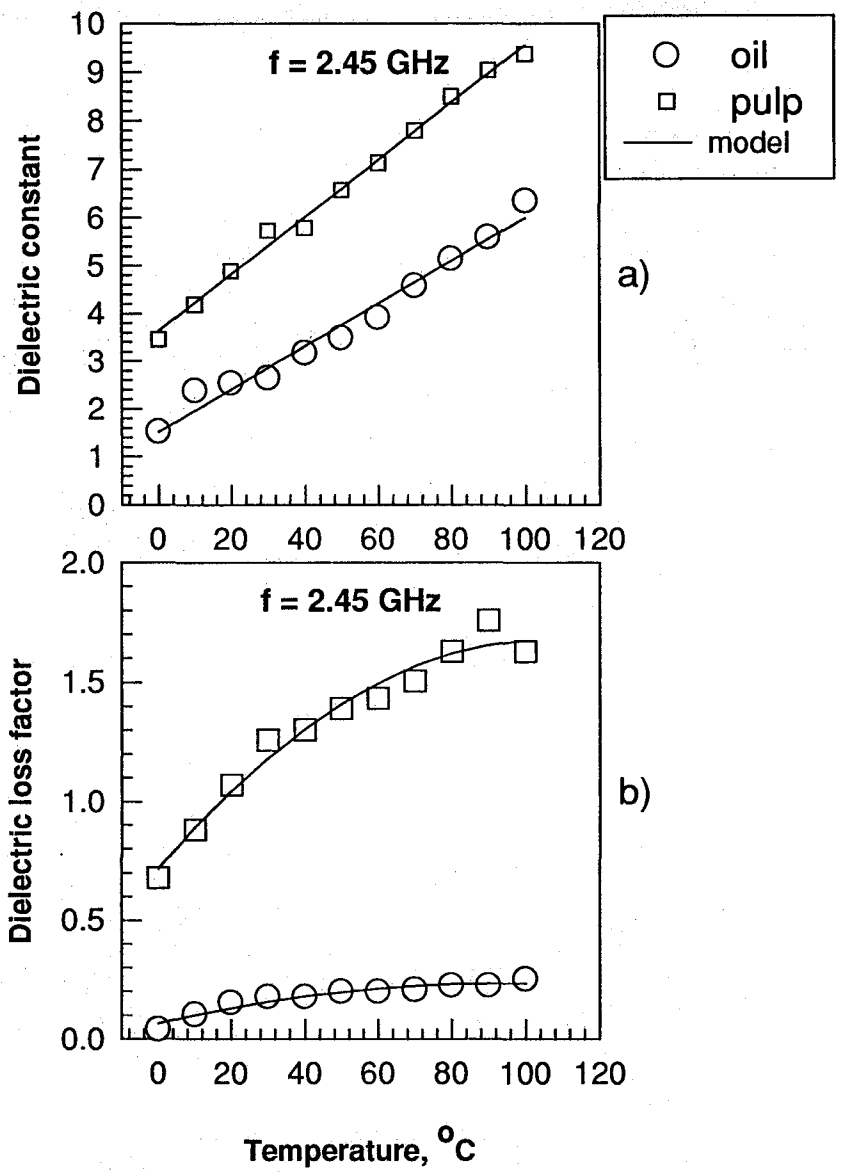


Figure 5.19 Functional relationship of dielectric constant and loss factor vs. temperature of neem oil and pulp at 2450 MHz.

- 2 MW Synthesizers - $2000
- Micro controler - $200
- Amplifier - $250
- Detector - $150
- Power supply - $100
- Outer Casing - $50
- Cable and filter - $200

Total = CAD$2950 (excluding software and computer expenses)

The cost for VNA based measuring system is shown below::

- 8753D HP VNA Network Analyser- $50000 minimum
- GPIB card- $500
- Amplifier -$250
- Power supply - $500
- Cable and filter -$200

Total = CAD$51,450

Since the resonant cavities are not commercially available the cost involved in the design and development (fabrication) need to be considered. However, this depends on the selection of the material and nature of experimentation. The material and the machining costs are similar for both VNA and PA measurements.

For variable temperature measurements, the cost towards the refrigerated re-circulator and suitable thermocouple sensors are to be added which are the same for both VNA and PA. Other parameters to consider are:

- Weight
- Portability
- Size
- Speed of measurement
- Accuracy

Concerning the weight, the permittivity analyzer is a better equipment compared to VNA. The total weight of VNA is around 60 pounds, and the dimension is around 90 cu cm; whereas permittivity analyzer studied here weighs only 10 pounds, and the size is only 15 cu cm. This added advantage will make a big difference for field application. While it is extremely difficult to use the VNA for field measurement, the Permittivity Analyzer can

find the best option as most appropriate equipment for in-situ measurements of dielectric properties. The scanning time and speed of measurement for both systems are relatively the same.

In this unit (PA), it was not easy to achieve better than 40 dBm of output microwave power. Suitable amplifier was integrated in the cavity loop to increase the sensitivity of the detector. The frequency resolution of PA is 10 kHz and this range is sufficient to carry out dielectric measurements; however the effect of this parameter influences the precision of dielectric constant. This negative point can be compensated by increasing the size of sample under test. In the perturbation theory the dielectric constant is a direct function of sample volume over cavity volume.

In conclusion, we can see that the analyzer is a dedicated equipment for permittivity measurement at two frequencies and has a capability to measure in the range of -30 and $+200^{0}$C. It is also a perfect tool for field application. VNA is suitable for extensive laboratory measurements where more accuracy is needed not only for measuring permittivity but for various other microwave parameters needed in the fields of telecommunications, bio-medical and space research, etc.

VI SUMMARY AND CONCLUSIONS

This research is aimed at developing and integrating the permittivity analyzer to measure the dielectric constant and dielectric loss factors at two ISM approved frequencies (915 and 2450 MHz) and various temperature capabilities. The permittivity measurement is based on the perturbation concept and uses circular cavities operating in a standard TM_{010} mode. As per the ASTM guidelines and design principles, two resonant perturbation cavities were designed and fabricated for 915 and 2450 MHz frequencies, respectively. The initial coupling of microwave antenna and tuning of the cavities was performed with the aid of Vector Network Analyzer (HP 8753D). Both the cavities exhibited high Q-factor (>4000) under empty cavity condition and thereby indicating an accurate and reliable measurement.

Choice of the sample holder (micropipette / teflon tube) and the sampling procedure depend on the nature of the material (low or high loss) and the experimental conditions. The micropipettes used in all the measurements were made of borosilicate glass (ε'=4.2, ε''=0.008). An empty sample holder is always placed at the geometric centre of the cavity (where the electric field intensity is maximum for such a mode) to allow calibration of the tube for subsequent measurements with the sample. After both the cavities are tuned and the assembly of electronic components are complete, certain standard reference materials (water, air, alcohol) were repeatedly tested for their ε' (water=78.4,air=1) and ε'' (water=10.98, air=0) at 2.45 GHz and 23°C. Similar measurements were also made at 915 MHz frequency. Suitable control software was developed to carry out the measurement procedure and analysis. Appropriate mathematical steps and perturbation formulae were included in the main control software for complete permittivity analyses at both designated frequency schemes. Calibration of the equipment was made with the vector network analyzer tuning functions and performance was verified by repeated testing of standard materials such as distilled water, alcohols and air. The ε' and ε'' of selected agricultural and food materials ranging from homogenized milk, edible oils and organic solvents, were tested and validated at 915 and 2450 MHz and at appropriate temperature ranges. All the samples tested were homogeneous in nature except the neem oil and neem pulp.

The permittivity analyzer's performance was evaluated in terms of the data

obtained. The first issue was the stability of measurements taken on the same sample under the same conditions in sequence. The variability aspects of measurements taken on different samples of material of the same batch with respect to the different temperature levels and frequencies is addressed and validated in terms of values reported in the literature. The stability of quadruplets of dielectric constant, considered over all of the materials, temperatures and frequencies tested was excellent. In 95% of the tests, all numbers of a quadruplet were identical. When the members were not identical, variations were nevertheless quite small (<1% of the average reading). Overall, a nominal error of less than 2% can be attributed to the system's circuitry and cavity conditions. To check the variability across samples at different frequencies and temperatures, the standard error (SE) of the quadruplet averages was determined and then normalized by dividing by the mean of the samples at that combination (usually 4 or 6 samples). The average of the NSE's and 95% confidence limits are calculated.

Milks and edible oils exhibited odd behavior at lower temperatures (<5°C) and could not be easily explained. The greatest degree of variability in the distribution of normalized standard errors corresponding to ε' occurs in the case of neem oil and neem pulp which were the least homogeneous of the substances tested.

Control software was upgraded and appropriate communication codes for the measuring system and the PC were identified. Entire permittivity analyses procedure is encrypted in a user friendly module in Visual Basic 5.0 in which the data can be translated, stored and plotted. The actual measurement can be carried out with the basic operating system (Windows 95+) and independent of any software since the application of this program is an executable format independent of software.

There are economic as well as several other advantages of this dedicated equipment over the conventional VNA based system in terms of measuring and analyzing dielectric properties at a set frequency and variable temperature combinations.

In conclusion, a dual frequency (915, 2450 MHz) permittivity analyzer is designed and developed to measure the dielectric constant and dielectric loss factor at varying temperatures using cavity perturbation concept. The equipment was tested and validated for its repeatability aspects. Behavior of certain class of agri-food materials were studied at both frequencies and at varying temperatures.

VII RECOMMENDATIONS FOR FUTURE WORK

The permittivity analyser design and development presented in this research needs additional experimental work and validation studies. Detailed proximate analysis of agri-food samples are necessary to fully understand the microwave material interaction mechanisms. Standard protocol need to be established for selection of sample holder, micro-sampling, and sample volume calculation. High lossy samples are recommended to be filled using 10 µL or even smaller capacity micropipettes where as bigger (50 or 100 µL) sized tubes are suggested to be used for oils and other low loss materials. For a TM_{010} mode cavity, the geometric center of the cavity should be precisely located during the fabrication of the resonant chambers as the electric field intensity is maximum in this region. Although shape of the cavity is not important, it is suggested to further investigate the dielectric properties measurement aspects obtained by a rectangular cavity for the same mode. Even though this research did not focus on measuring dielectric properties of powders, particulate and semi-solids, it is possible to determine the dielectric parameters of such materials in the designed equipment of this study. Choice of sample holders, filling volume, sealing factors, etc. need to be standardized for such particulates.

This newly configured analyzer can be further adapted for both free space and transmission type of measurement with suitable modification of electronic design and computer programming.

Sample selection is very important and the working temperature range should be examined for standard calibration materials before unknown samples are tested for dielectric constant and loss factor. Proper insulation of the resonant cavity ensures rapid and accurate measurements as the condensation effect, if any, can be eliminated.

Measurement of power levels, heating rate and its control will be useful for future research on molecular structural changes in agri-foods subjected to amplified signals.

There is a need for a comprehensive data hand-book (source) of the values of ε' and ε'' obtained using other techniques for array of agricultural and food materials tested under several experimental conditions (frequency, temperature, moisture content, density, proximate composition, etc.) and is worthwhile to investigate in future.

VIII CONTRIBUTIONS TO KNOWLEDGE

The work presented here has made an original contribution to the body of knowledge surrounding the development of dielectric properties (permittivity) measurement aspects in the agricultural and food sectors. The main points of this contribution are:

(i) The permittivity analyzer (PA) has been fully integrated to operate at two major ISM approved frequencies (915 and 2450 MHz) and varying temperatures (-30 to 200°C).

(ii) Resonant perturbation cavities (TM_{010}) are designed, fabricated, tested and validated for both frequencies.

(iii) Suitable control software is written using Visual Basic 5.0 communication capability codes and an user-friendly module is developed.

(iv) Permittivity Analyzer is a dedicated equipment, stand-alone, easy to operate, flexible and portable.

(v) Relationships of ε' and ε'' on temperature and frequency for homogenized milk, edible oils and organic solvents are obtained using the newly developed PA and summarized.

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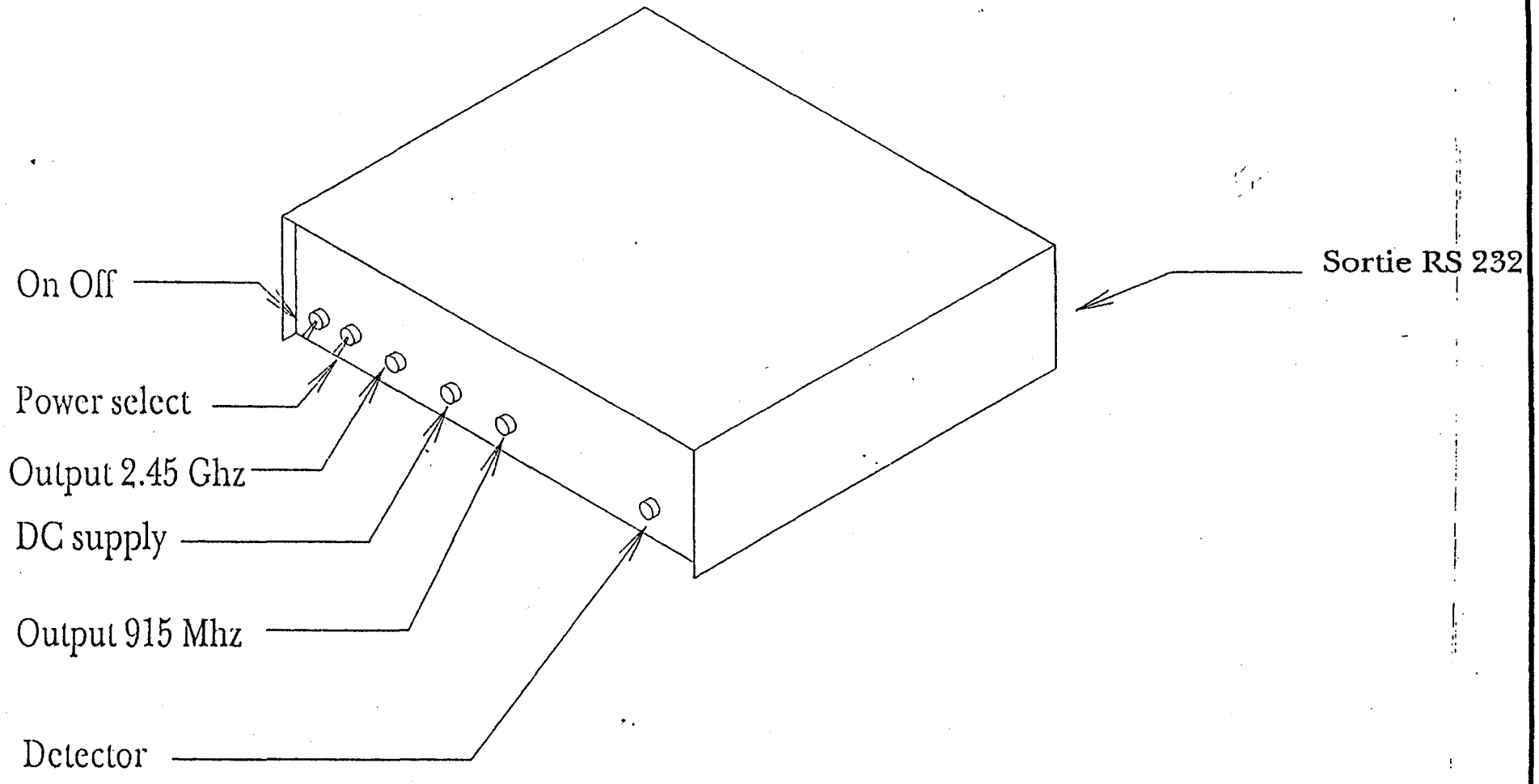
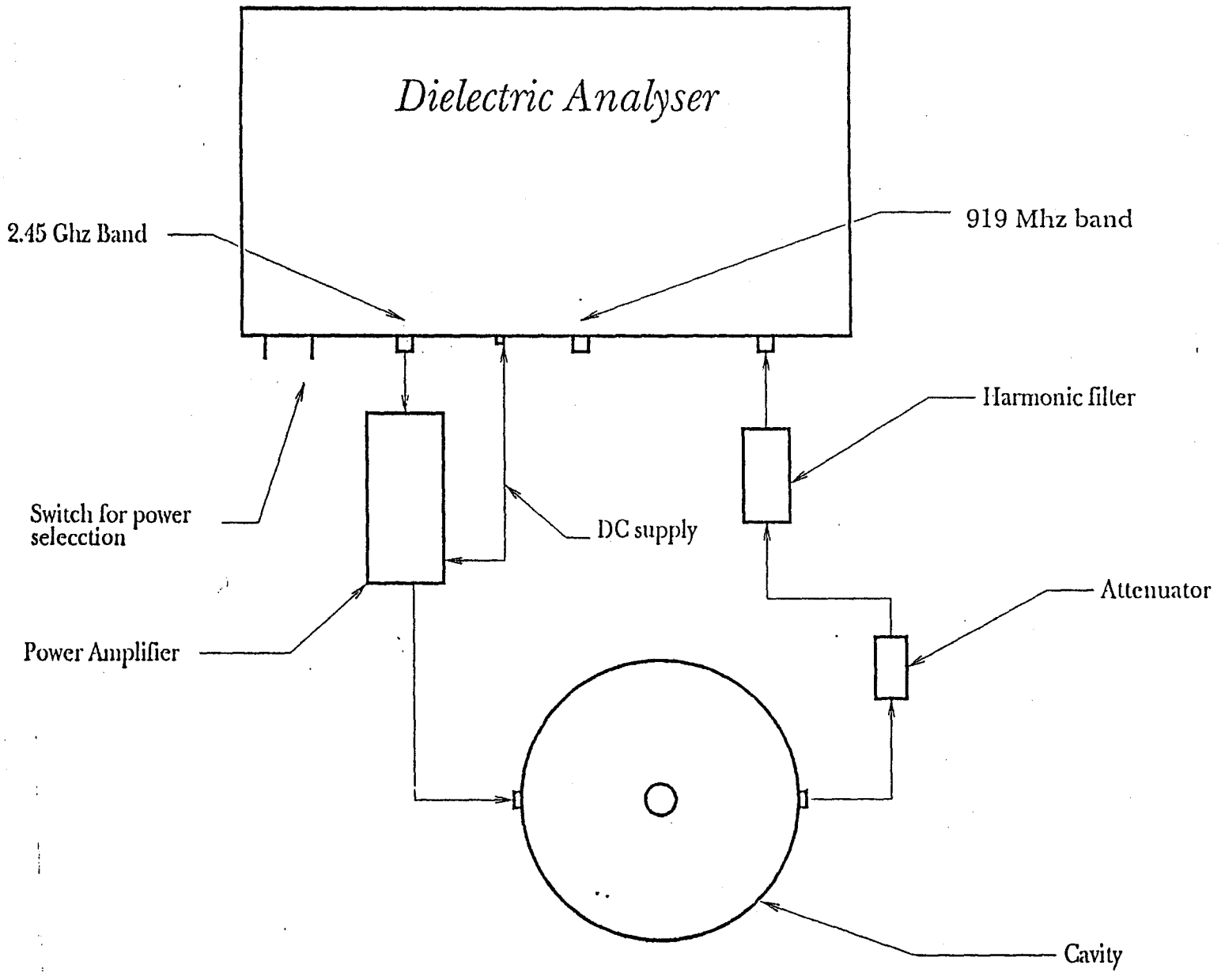
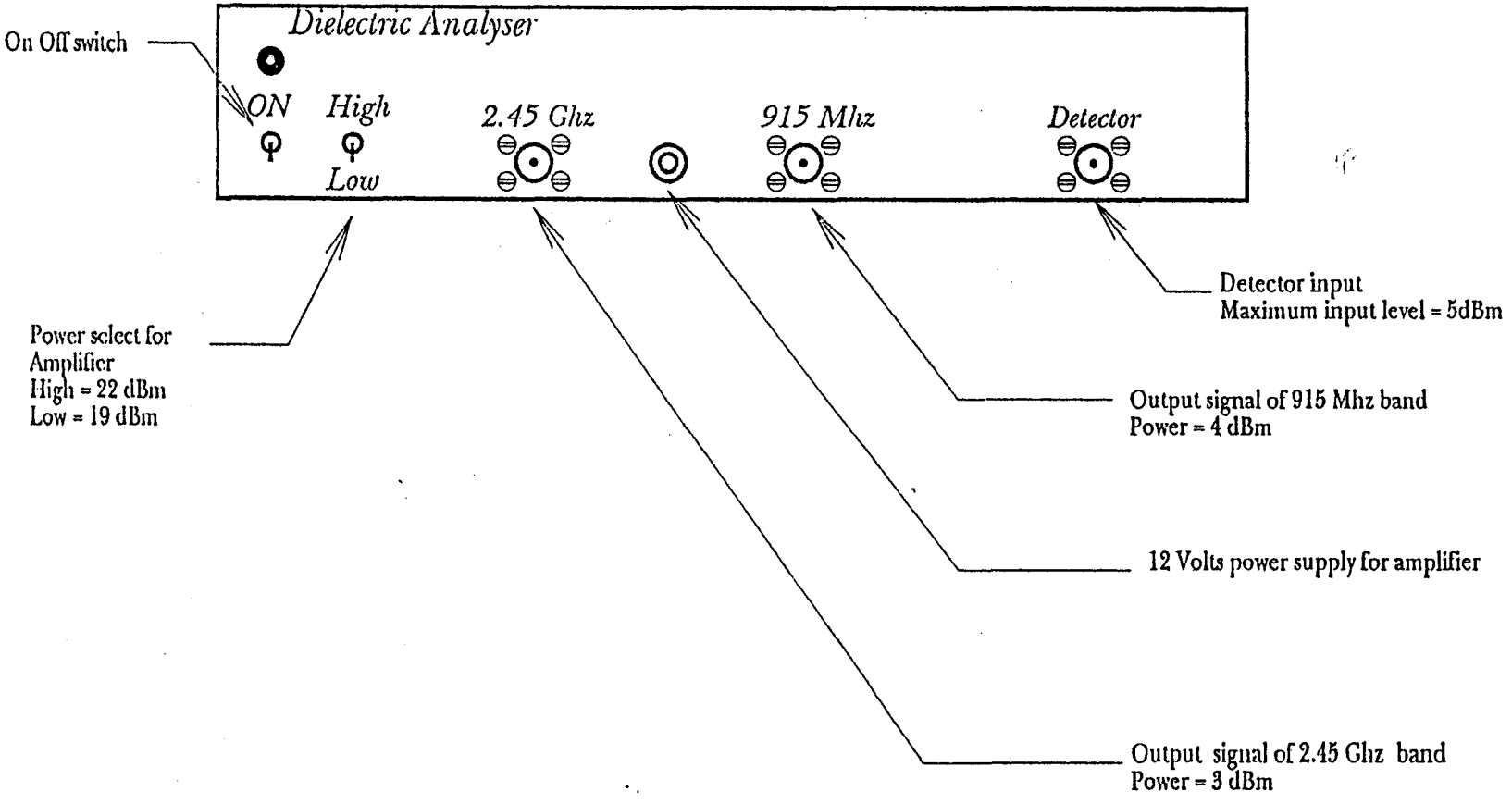
Appendix

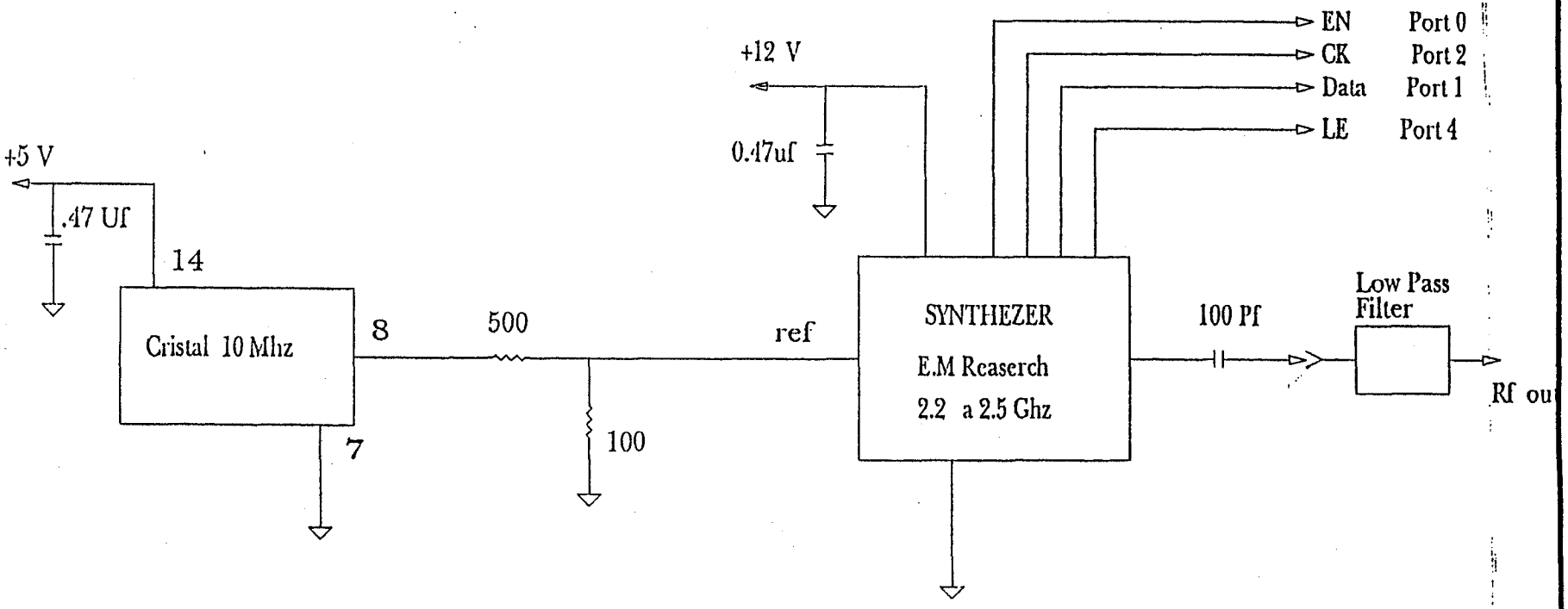
(Line diagrams of the dual frequency permittivity analyser assembly and circuit diagrams of the 915 and 2450 MHz system components)

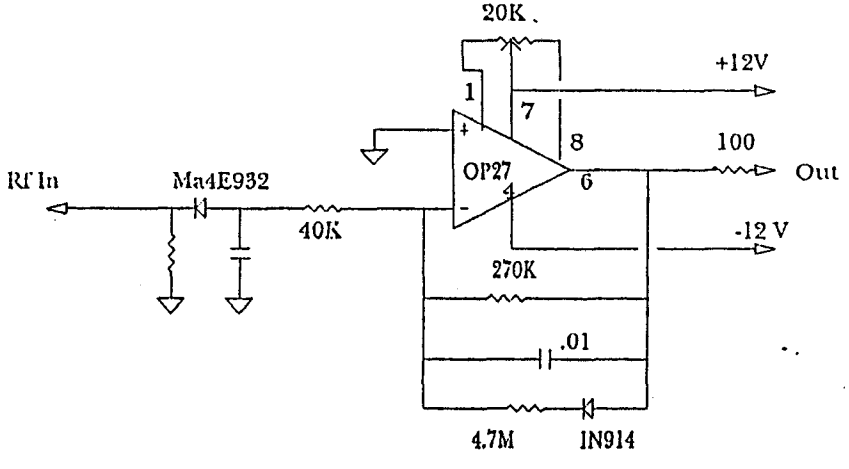
Dielectric Analyser



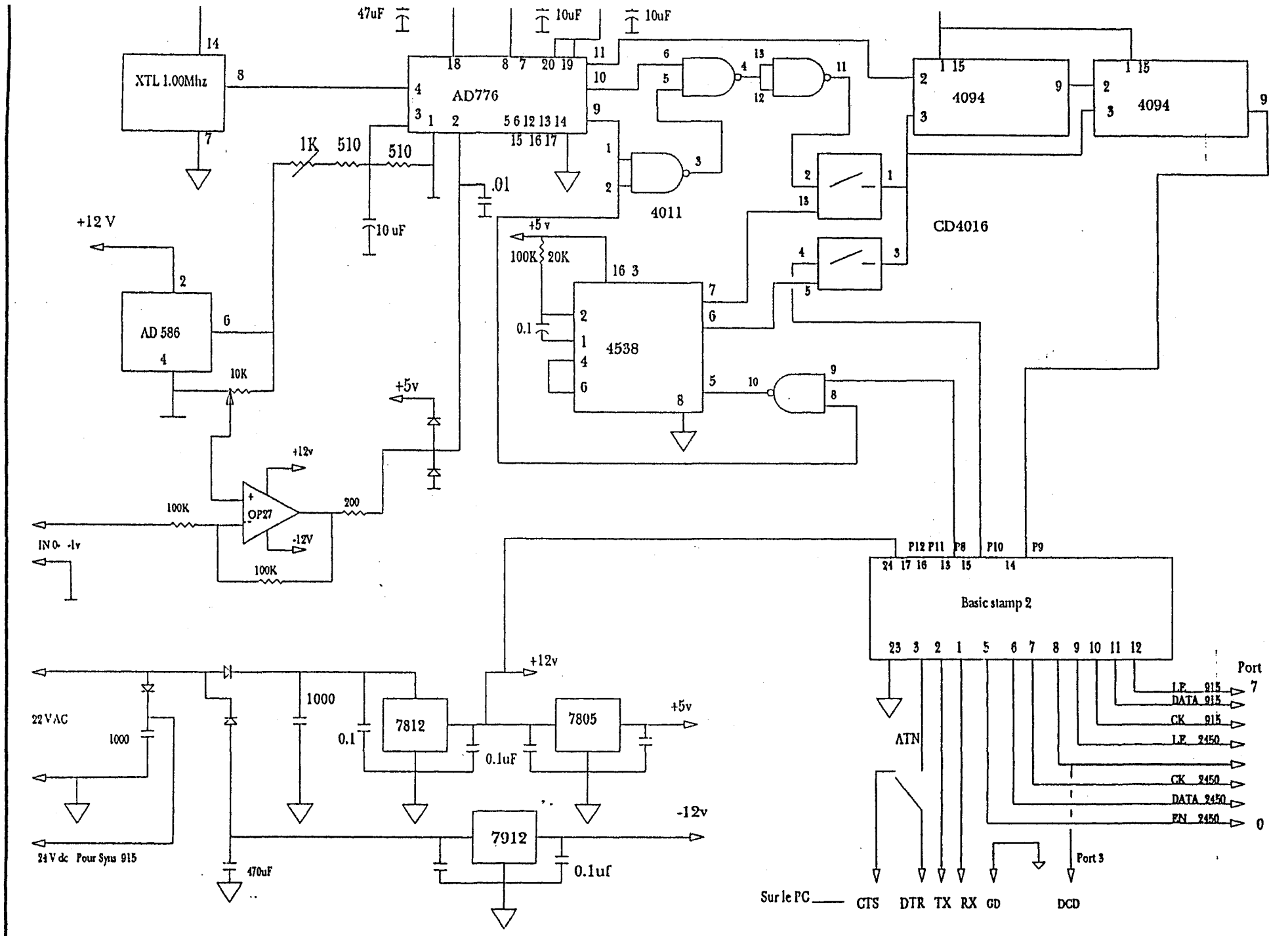

Dielectric Analyser
2.45 Ghz Band
919 Mhz band
Harmonic filter
Switch for power selecction
DC supply
Attenuator
Power Amplifier
Cavity

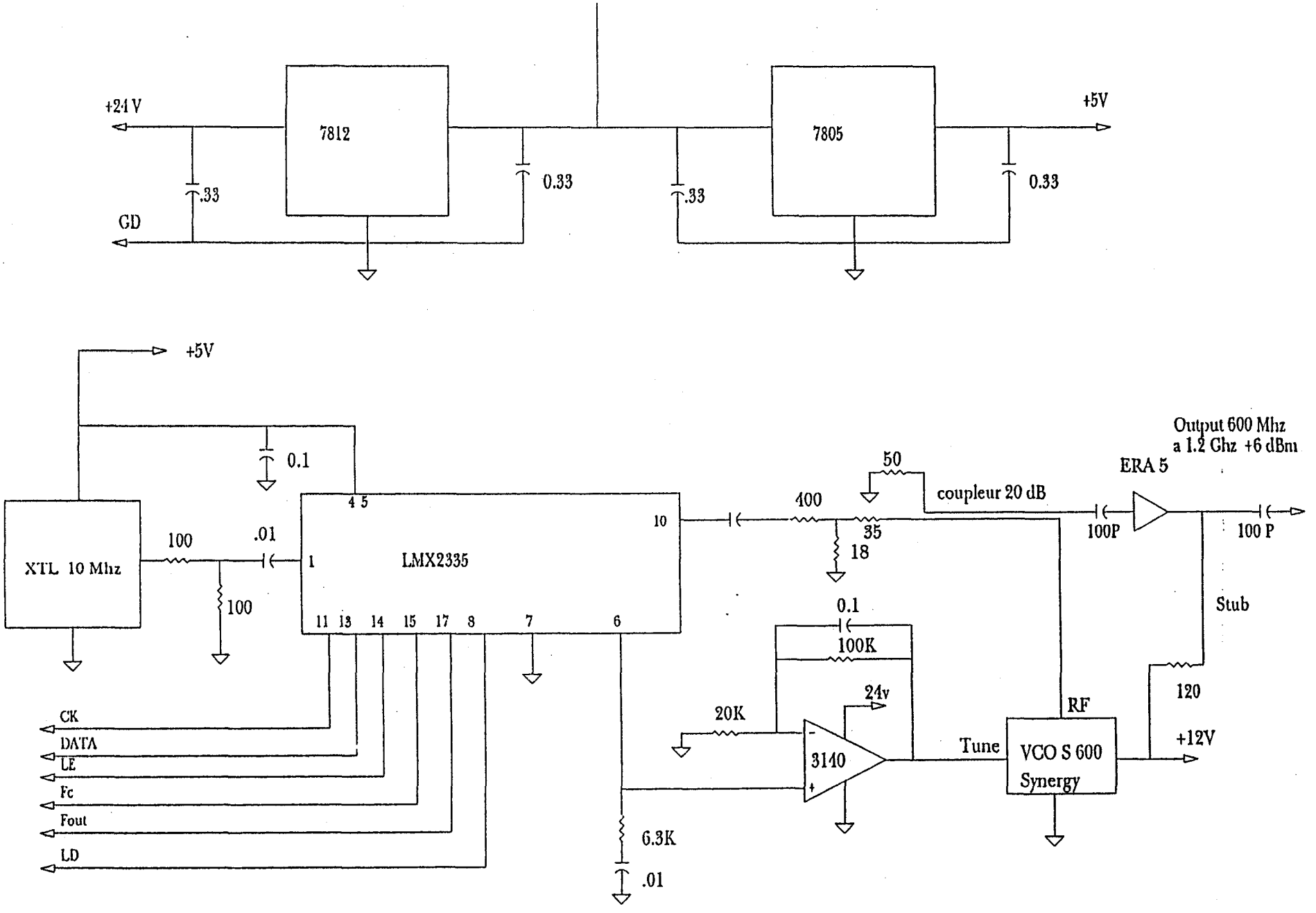

Dielectric Analyser
On Off switch
ON
High
Low
2.45 Ghz
915 Mhz
Detector
Power sclect for
Amplifier
High = 22 dBm
Low = 19 dBm
Detector input
Maximum input level = 5dBm
Output signal of 915 Mhz band
Power = 4 dBm
12 Volts power supply for amplifier
Output signal of 2.45 Ghz band
Power = 3 dBm


+5 V
.47 Uf
14
Cristal 10 Mhz
8
7
500
100
ref
+12 V
0.47uf
EN
CK
Data
LE
Port 0
Port 2
Port 1
Port 4
SYNTHEZER
E.M Reaserch
2.2 a 2.5 Ghz
100 Pf
Low Pass
Filter
Rf ou

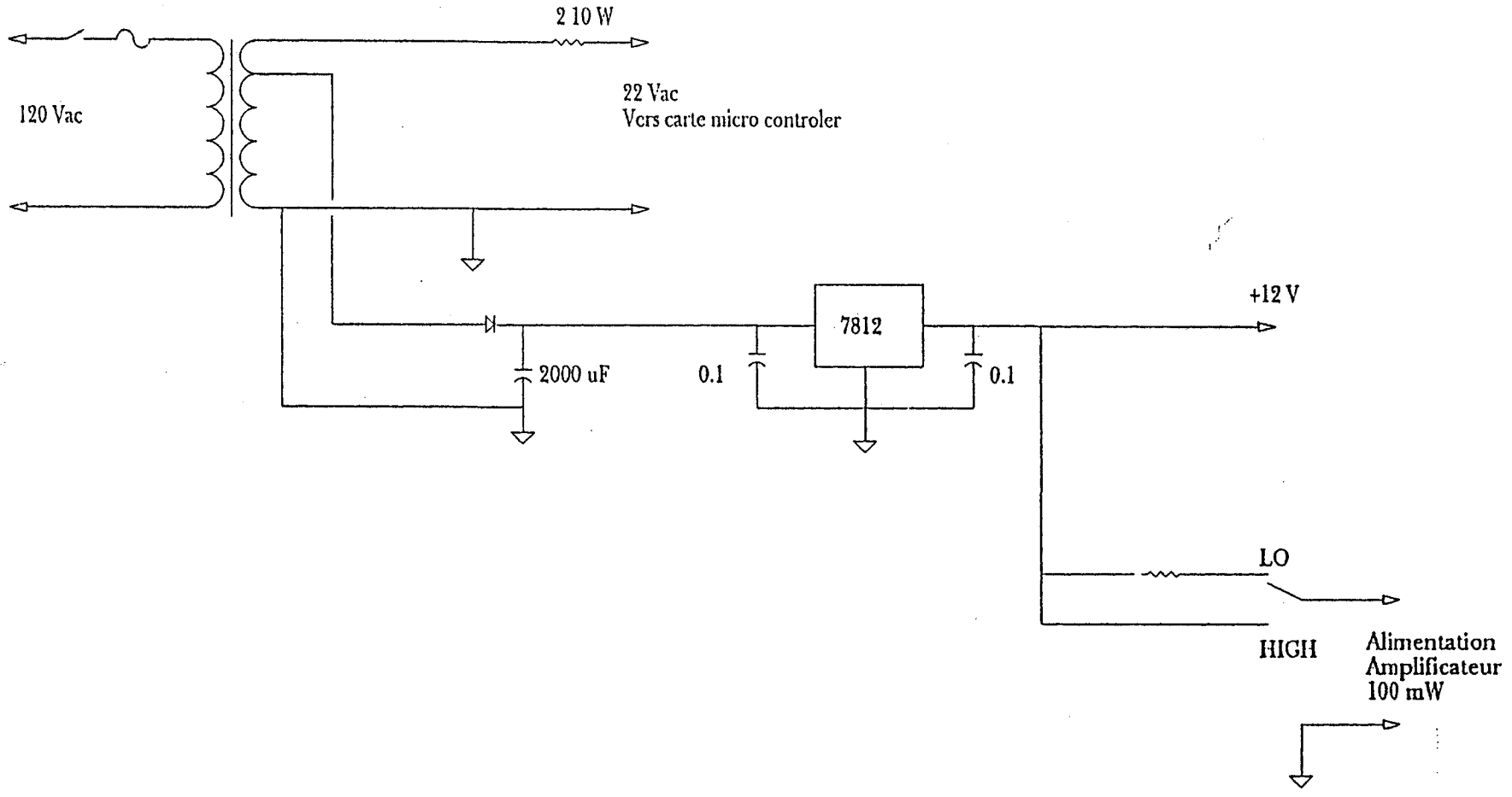

20K
+12V
1
7
8
100
OP27
6
Out
Rf In
Ma4E932
40K
-12 V
270K
.01
4.7M
IN914

Gautel


XTL 1.00Mhz
AD776
47uF
10uF
10uF
1K
510
510
.01
10 uF
4011
CD4016
4094
4094
+12 V
AD 586
10K
+5v
100K
20K
4538
0.1
+5 v
+12v
OP27
100K
200
-12V
100K
IN 0- -1v
Basic stamp 2
P12 P11 P8 P10 P9
ATN
Port 7
LE 915
DATA 915
CK 915
LE 2450
CK 2450
DATA 2450
EN 2450
0
Port 3
Sur le PC
CTS
DTR
TX
RX
GD
DCD
22 VAC
1000
1000
0.1
7812
7805
+5v
0.1uF
-12v
7912
0.1uf
24 V dc Pour Syns 915
470uF


+24 V
7812
.33
GD
0.33
.33
7805
+5V
0.33
+5V
0.1
XTL 10 Mhz
100
.01
100
4 5
1
LMX2335
10
11 13 14 15 17 8 7 6
CK
DATA
LE
Fc
Fout
LD
6.3K
.01
400
50
coupleur 20 dB
35
18
100P
ERA 5
Output 600 Mhz
a 1.2 Ghz +6 dBm
100 P
Stub
120
+12V
0.1
100K
24v
20K
3140
Tune
RF
VCO S 600
Synergy

Alimentation de l'analyseur dielectrique



Gautel

SYSTEME DE MESURE DE PERMITTIVITE À 915 Mhz