Capítulo 19

A comparative review of the main techniques for electromagnetic characterization of materials

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ABSTRACT
This paper presents the main techniques of electromagnetic characterization electromagnetic at microwave frequency. A detailed analysis of these is performed, indicating which materials under test (MUT) can be measured with the specific technique. Additionally, for the dielectric characterization, measurement results are presented with the best technique that suits this case.

Keywords: Dielectric characterization, measurement techniques, microwave frequency.

1 INTRODUCTION

Materials contain charged particles that, when subjected to electromagnetic fields, interact with each other producing currents and modifying the propagation of the electromagnetic wave in these media compared to free space[1]. A set of these interactions produces specific effects on a macroscopic scale that constitute the interaction of the material as a whole with the fields transmitted over it. When these phenomena are represented as a function of this macroscopic electromagnetic behavior, the material, be it a pure substance or a mixture (Ex: soil), is designated as a medium. Therefore, the constitutive relations specify the characteristics of a medium in which fields exist. When the electromagnetic wave does not propagate in a vacuum, but in a linear, isotropic, and non-dispersive medium, the constitutive relationships change in a simple proportionality, common to many materials. In this case, the constitutive relationships become [2]:

$$\vec{D} = \varepsilon \vec{E}$$
$$\vec{B} = \mu \vec{H}$$

It should be noted that $\varepsilon \mu$and are the constitutive parameters used to characterize the dielectric properties of isotropic material. Isotropic materials are all those electrical or magnetic flux densities independent of the polarization of the incident electromagnetic wave and a single permeability or permittivity value is represented.

There are lots of specialized literature presenting dielectric measurements of various materials, in different frequencies and temperatures, by plenty of characterization methods. Unfortunately, most of these works do not present a solution to choose the best characterization method due to a Material Under Test (MUT) you are up to measure in the microwave range.

The dielectric characterization of materials for commercial use can be used in support of new materials development, in quality assurance of manufacturing processes, providing an enterprise product development shorter lifecycles, higher performance, and reduced material scrap.

Every material has a unique set of electrical characteristics that are dependent on its dielectric properties. Accurate measurements of these properties can provide scientists and engineers with valuable information to properly incorporate the material into its intended application for more solid designs or to monitor a manufacturing process for improved quality control. More recent applications in the area of industrial microwave processing of food, rubber, cement, plastic, and ceramics have also been found to benefit from a knowledge of dielectric properties [3].

TABLE I. Dielectric measurement needs, applications in industry, and products.

Industries	Products	Measurement Needs
Traditional		
Electronics Microwaves Communications Aerospace/Defense	Dielectrics Substrates Ferrites Absorbing materials	ε_r and/or μ_r Reflection Γ, Transmission τ High accuracy Wide frequency
Non-traditional, but technical		
Chemical Ceramics Composite Materials	Plastics Adhesives Polymers Paints/Films Semiconductors/Superconductors Ceramics	ε_r and/or μ_r Ceramic sintering/annealing Compositions analysis Temperature dependence Cure/Polymerization Relaxation effects
Non-traditional, not technical		
Food, Packaging Forest, Paper Rubber Cement, Concrete Bio, Medicine, Drugs	Food, Processing, Packaging Wood, Paper, Fiber Rubber Cement Medical therapy	Research, Control, Optimization Microwave processing Heating, Cooking, Drying Moisture content Analysis, Diathermy

The present work aims to present and implemented a model that allows the choice of the most appropriate method for a specific material characterization, given its characteristics or laboratory equipment availability. The main approaches for materials characterization in microwave range wavelengths are presented.

This paper is composed of five more sections besides the introduction. Section II-Materials and Methodology discusses the main factors of interest to determine the most appropriate characterization method and measurement procedures for each type of Material Under Test (MUT), section III results obtained for available samples are discussed, section IV the final considerations and the steps that will give continuity to the work are carried out and in section, V acknowledgments are presented.

2 MATERIALS AND METHODOLOGY

Electromagnetic characterizations methods

Characterization in material science refers to the use of external techniques to probe the internal material structure (geometric characterization) and material properties of the structure (chemical, thermal, electromagnetic, etc)[4]. This article focuses on dielectric properties characterization. Dielectric properties depend on frequency, homogeneity, anisotropy, temperature, surface roughness, and in the case of ferroelectrics and ferromagnetics, applied bias field[5,6,12]. There are some factors of interest to determine the most appropriate characterization method and measurement procedures for each type of Material Under Test (MUT) [7]:

- The frequency range of interest;
- The dielectric loss (high, medium, or low) and expected permittivity range;
- The type of material, for example, hard, malleable, or soft solids, volatile or viscous liquids;
- Specimen machining imperfections and tolerances and their influence on uncertainty;
- Specimen shape and size and their influence on measurement uncertainty;
- Specimen anisotropy and homogeneity and their influence on measurement uncertainty;
- Inhomogeneity and the presence of surface layers on specimens;
- The possibility that the specimen may be made from a magnetic and anisotropic material;
- The required uncertainties. What level of uncertainty can be achieved by available methods;
- The specimen composition (e.g. does the specimen have a laminated structure);
- The availability of suitable methods for machining and grinding specimens;
- The presence of surface inclusions and pores, surface conditions in solid specimens;
- Toxicity, contamination, and evaporation of liquid specimens;
- The cost of machining specimens and performing measurements: cost-effectiveness; and
- The time taken to perform the measurements – labor intensiveness and the labor cost of measurements.

No single technique can measure all materials over an entire set of aforementioned characteristics. Among various techniques some most popular and most important methods for dielectric measurements are briefly discussed:

1) Transmission Line(TL): Uses a sample holder to adapt a MUT to a waveguide. By using specific algorithms sample dielectric characteristics are obtained[8].

2) Transmission in the free space(FE): This method employs an anechoic chamber-controlled environment, in which two microwave antennas are put in a far field condition to irradiate over a sample placed among them. It also uses algorithms to extract constitutive parameters given by sample scattering

parameters[9].

3) Coaxial test method (CT): A coaxial transmission line is terminated with the material that is desired to determine the permittivity, which serves as a load. Permittivity is determined by the spreading parameters measured at the inlet of the line[10].

4) Capacitor Method (CP): The sample is placed between two parallel plates. Once the capacitance of the system is measured, from simple mathematics relations the sample permittivity is calculated[11].

In this paper, a multi-criteria method has been used to the determination best method of dielectric characterization of solid material in a frequency range between 8,2 to 12.4 GHz.

Comparative analysis for dielectric measuring techniques

Given the aforementioned measurement methods and factors affecting its realizations, we can conclude it is a moot point to select the appropriate measuring technique for a dielectric measurement. Thought we would like to suggest a multicriteria method to solve such this problem. It consists of two tables. The first table, described in Table II presents each measurement method, constitutive parameters provided, frequency range, MUT dimensions, and MUT loss characteristics:

TABLE II. Measurement techniques versus parameters provided versus MUT dimensions and losses

Technique	Constitutive parameter provided	Frequency range	MUT dimensions	MUT losses
Transmission Line(TL)	ε and μ	0,1 to 110 GHz	Restricted to sample holder dimensions	High and low losses
Free Space(FE)	ε and μ	5 a 500 GHz	10 λ x 10 λ	High and low losses
Coaxial Test Method(CT)	ε	100 MHz to 18 GHz	Restricted to sample holder dimensions	Low losses
Capacitor Method(CP)	ε	0,8 a 3 GHz	Restricted to sample holder dimensions	Low losses

The second table is Table III, it presents the relation among sample characteristics and the methods available for microwave measurements:

TABLE III. Sample characteristics versus dielectric measurement methods

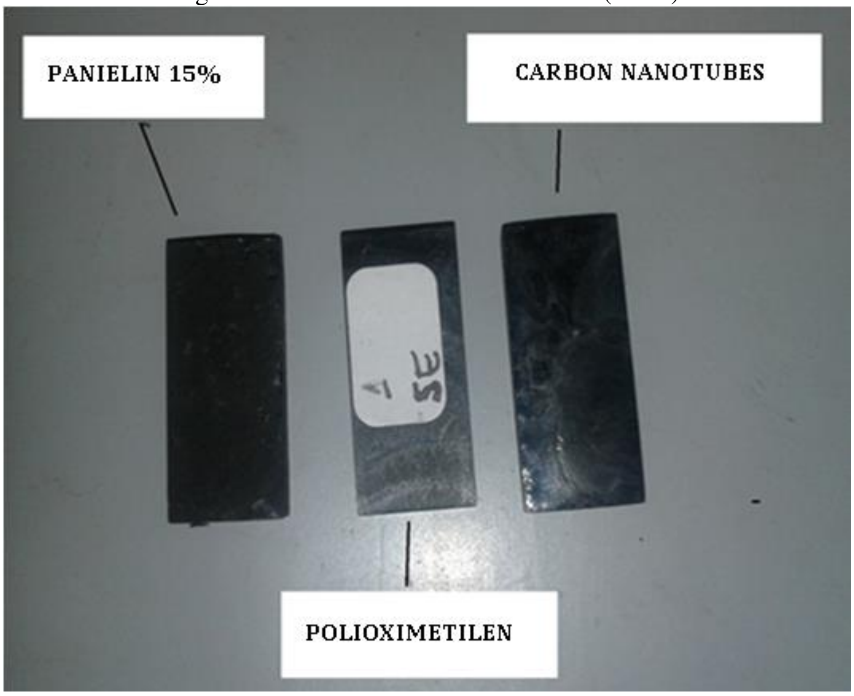
MUT type	Transmission Line(TL)	Free Space(FE)	Coaxial Test Method(CT)	Capacitor Method(CP)
Liquids				
Solids				
Powder and Gases				
Films, slabs, and ink				
Metamaterial				
Anisotropic				

Subtitles	
Able	
Needs adaptations	
Unable	

3 RESULTS

Three 3mm thickness samples of a parallelepiped shape are available for measurements. The first sample obtained consists of resin epoxy LY5052 impregnated with carbon nanotubes (NTC), the second material obtained consists of a composite based on Polyaniline and Carbon Black (Pani/NF) and the third material obtained is composed of a pure composite of dielectric polyoxymethylene. The specimens that have fillers in their mixture were prepared respectively with 15% Pani / NF and 0.5% concentration of NTC in their respective substrates. These samples are shown in figure 1:

Fig. 1. Three Solid Material Under Test (MUT)



In the sequence of the work it will be determined which is the best method of measures to be used in the characterization of the MUT, given the main characteristic of samples collected as below:

TABLE IV. Sample characteristics versus dielectric measurement methods

MUT Characteristics	Data collected
Frequency of interest	8.2 to 12.4 GHz (X Band)
The dielectric loss (high, medium, or low)	High dielectric loss
Type of material: hard, malleable, or soft solids, volatile or viscous liquids	Hard solid
Specimen shape and size	Rectangular (Length:22.86mm, High:10.16mm, Thickness: 3mm)
Inhomogeneity and the presence of surface layers on specimens	Homogenous and a single-layer material

When analyzing the general characteristics of the three MUTs of natural absorbers in the form of cobblestones, obtained according to table IV, it is obtained that the methods of the Transmission Line, Free Space would be able to measure samples without restrictions. However, so that the samples in question could be measured by coaxial test and capacitor methods, they should be machined, generating costs and wasting time performing measurements as described in Table V:

TABLE V. Eligible measuring methods for available MUT

MUT type	Transmission Line(TL)	Free Space(FE)	Coaxial Test Method(CT)	Capacitor Method(CP)
Liquids				
Solids				
Powder and Gases				
Films, slabs and ink				
Metamaterial				
Anisotropic				

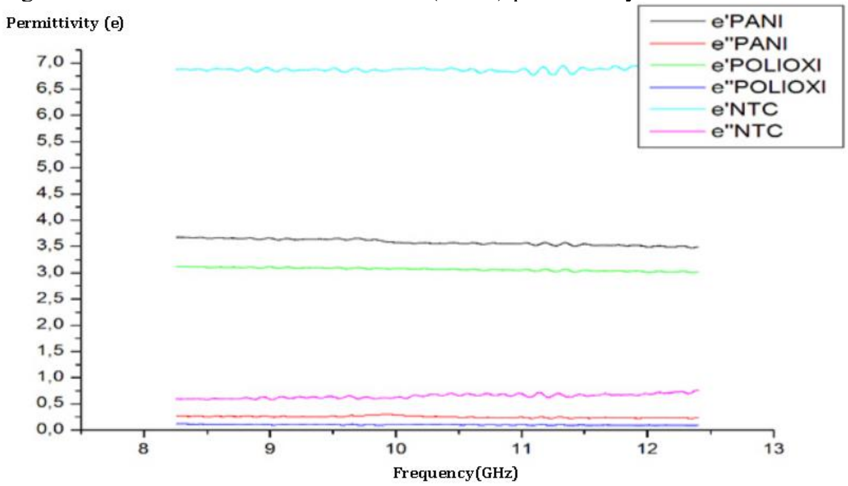
However, as absorbent materials likewise Panielin, Carbon nanotubes, and Polioximetilen are high-loss materials, the Coaxial Test(CT) and Capacitor Method(CP) are not eligible for the given MUT measurement as described in Table VI:

TABLE VI. Eligible measuring methods for available MUT

Technique	Constitutive parameter provided	Frequency range	MUT dimensions	MUT losses
Transmission Line(TL)	ε and μ	0,1 to 110 GHz	Fitted to WR 90 sample holder: 22.86mm x 10.16mm x 3mm	High and low losses
Free Space(FE)	ε and μ	5 a 500 GHz	10 λ x 10 λ	High and low losses
Coaxial Test Method(CT)	ε	100 MHz to 18 GHz	Restricted to sample holder dimensions	Low losses
Capacitor Method(CP)	ε	0,8 a 3 GHz	Restricted to sample holder dimensions	Low losses

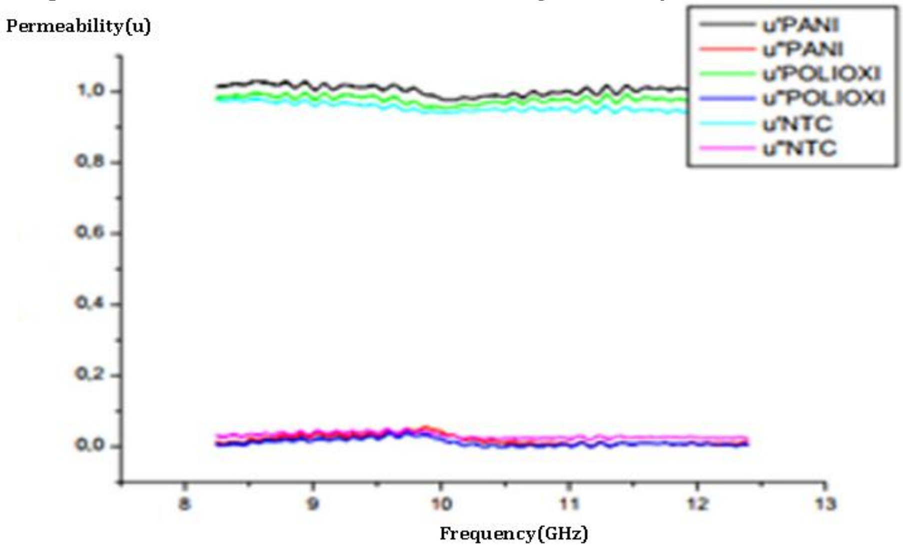
Though, when analyzing the sample's frequency of interest and the MUT dimensions specifications for the method´s employment, Table VI shows that Free Space Method is not eligible for the given MUT. The only method that can be used to measure the available MUT, is the transmission line.Fig.2 and Fig.3 presents MUT measurement done for the three samples in a Transmission Line Setup:

Fig. 2. Three Solid Material Under Test (MUT) permittivity measurements results



From Fig.2 analysis it´s obtained that NTC material MUT built has the highest dielectric losses since it has a complex component permissiveness (e´´) equivalent to 0.55. Among all other materials, NTC is most suitable for electromagnetic absorber materials applications.

Fig. 3. Three Solid Material Under Test (MUT) permeability measurements results



From the permeability results obtained in Figure 3, it is observed that all samples of absorbent materials are non-magnetic and without losses, since they presented magnetic permeability $\mu = 1$, with zero imaginary complex components.

The technique for characterizing materials using the transmission line measurement technique allows for obtaining the permissiveness and permeability of the samples in a simple, reliable, and adequate way for the research purposes carried out in this job.

4 FINAL CONSIDERATIONS

Recently, the dielectric characterization of materials has gained strong significance in industrial applications. These properties provide useful assets for systems design improvement and quality assurance of products. This work presents the main techniques used in the characterization of materials and three solid MUT species characterization. As a secondary objective of this work, a methodology to determine the most appropriate technique for measuring samples is explained so as in reverse, it is presented to students and industry technicians a definition of which kind of MUT can be measured with a given Laboratory setup. These techniques are quite accurate and relatively easy to implement, as long as the sample to be measured has dimensions compatible with the method that seeks to measure it. In the transmission line technique, the sample will need to fit in the sample holder to be coupled to the available waveguide. For free space measurements, the sample must have a length and width greater than 10 λ × 10 λ, to be properly characterized. As a suggestion for future work, it would be valuable studying dielectric characterization of quiral materials, Nonlinear materials characterization, and Digital signal processing techniques to narrow the beam of horn antennas to optimize the use of free space measurement devices without the use of anechoic chambers and lenses collimators to carry out these measures.

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