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Dielectric measurement of bitumen: A review

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Abstract: Bitumen is a rheological, viscoelastic, thermoplastic and extremely complex material that has hydrocarbons as its predominant molecules with slight traces of others. Bitumen is semi-solid at room temperature, liquid at high temperature and solid at a very low temperature. Till date, a complete understanding of its properties have not been fully known. Various techniques/methods have been developed by many researchers to study the behavior of this complex material. Most of the techniques are proved to be effective in evaluating some of the properties but very costly. This paper presents a review on some of the work conducted on investigating the behaviors of bitumen using microwave technique. This technique has been shown to be simple, less time consuming and cost-effective in evaluating both physical and rheological properties of bitumen when compared with other techniques.

1. Introduction

Microwave and radio-frequency signals are currently used as diagnostics tools in many fields with wide variety of applications. These comprise of wireless communication systems, mobile phone, radar, telemetry, medicine, biology, agriculture, industrial process control, forest and wood-working industry, civil engineering, radiometry, physics, chemistry, remote sensing and cooking food [1-3]. The efficient usage of electromagnetic energy in these applications can be achieved if its interaction with the associate material is known and this depends on the electromagnetic properties of the material [1]. According to Ma and Okamura [4], microwave measurement methods are dynamic and their parameters can be measured instantaneously, and the waves have no effect on the Material Under Test (MUT) in any way [5].

Microwaves are electromagnetic waves like radio, visible light, and X-ray waves but with a different wavelength from the other types of electromagnetic waves. For example, visible light has a wavelength of between 4 x 10^{-7} m (violet) and 7 x 10^{-7} m (red), while microwaves have wavelengths between 3 mm and 3 m, and these match to frequencies of between 100 MHz and 100 GHz [6]. Microwaves range also defined as follows [7-9]:

i. Oxford American dictionary: Electromagnetic waves of frequency 600 MHz to 300 GHz (50 cm to 1.0 mm)
ii. Webster's new world dictionary: Electromagnetic waves of frequency 300 MHz to 300 GHz
iii. IEEE standard dictionary: Electromagnetic waves of frequency 1.0 GHz to upwards

A microwave is an effective problem-solving tool for the determination of material parameters as it interacts with materials and structures in a microscopic scale [10]. Musil and Zacek [11] highlighted the importance of microwaves for the determination of material parameters which is closely related to microparameters of the materials. The researchers further state that the properties of materials medium and the electromagnetic field are quantified by the permittivity and permeability. Bartley and Begley [12] conducted an experiment based on an improved free-space calibration and reported that the dielectric property (permittivity) of a material is fundamental and unique, and independent in measurement techniques. The permeability (μ), permittivity, (ε) and conductivity (σ) are the most important electromagnetic characteristics of materials [13].

The principle of electromagnetic wave was also used in the field of highway engineering to study the behaviour of bitumen, other bituminous materials and bituminous concrete. Bitumen dielectric response depend on frequency and temperature, and is strongly related to its rheological behaviour [14]. Most of the studies carried out showed that the technique is promising. Microwave complex permittivity of various bitumen from different sources were determined using cavity perturbation technique by Xiao, J. A [15]. The results of the study showed a considerable difference in the complex permittivity of different metamorphic degrees of bitumen; the complex permittivity increases from the weak thermo metamorphosed bitumen to high thermo metamorphosed bitumen. The results also indicated a good correlation between the complex permittivity and Carbon-Hydrogen (C/H) ratio with a coefficient of 0.945. Weak metamorphic bitumen is composed of additional isomeric fatty alkanes with high content of hydrogen. Fundamentally, the atoms, electrons and molecules are bounded tightly together, as a result, it is difficult for them to move with the frequency of an applied external electric field. Consequently, their polarization ability is very low, and as such, their complex permittivity is small. On the other hand, high metamorphic bitumen has large amount of carbon concentration because they are predominated by aromatic compounds. Due to the strong polarization ability of aromatics, they follow the frequency of the external electric field, and as a result, their complex permittivity is high.

In this paper, summary of some of the researches carried out on investigating the behaviours of bitumen and bituminous concrete using microwave technique are presented, with a view to encourage researchers to explore more on this technique because of its potential benefits in the field of Highway Engineering.

2. Dielectric Measurement of Materials

The basic principle of materials testing is the interaction between materials and electromagnetic waves. The materials electromagnetic properties are consist of dielectric permittivity (ε), magnetic permeability (μ), and electrical conductivity σ [16]. The permittivity (ε) describes the behaviour of a material when an electric field passes through it, while the permeability (μ) describes the behaviour of a material when a magnetic field passes through it. Because permittivity and permeability are complex numbers, sometimes are referred to as complex permittivity and complex permeability. In determining the reflection or attenuation properties of the component materials of the microwave buffers, complex permeability and permittivity play a significant role [17].

Material's dielectric properties are characterized by the complex permittivity, ε* and consists of two components. The real part, ε' is referred as the permittivity or dielectric constant, and the imaginary part, ε'' is referred as the loss factor. Over the years, several techniques have been developed for dielectric measurement and Table 1 shows some of the commonly used techniques with their merits and demerits [18].

Table 1. Commonly used dielectric measurement techniques.

Method	Measure	Merit	Demerit
Coaxial Probe	ε_r	▪ Broadband frequency ▪ Simple and convenient (non-destructive) ▪ Best for semi-solids or liquids ▪ Simple sample preparation ▪ Isotropic and homogeneous material ▪ High accuracy for high-loss materials	▪ Air gaps causes errors ▪ Repetitive calibrations
Transmission line (waveguide)	ε_r, μ	▪ High frequency ▪ Support for both solids & liquids ▪ Anisotropic material	▪ Cannot use below few GHz, due to practical sample length limitation ▪ Sample preparation is difficult (fills fixture cross section)
Free Space	ε_r, μ	▪ Wide frequency range support ▪ Non-contacting ▪ Easy sample preparation ▪ Moderate accuracy for high-loss & low-loss ▪ Best for large flat and solid materials ▪ Useful for high temperature	▪ Diffraction problem (from material edges) ▪ Low end limited by practical sample size
Resonant Cavity	ε_r, μ	▪ Support for both solids & liquids ▪ Most accurate method ▪ Suitable for low loss materials ▪ No repetitive calibration procedure ▪ High temperature capability ▪ Best for low loss materials	▪ Measurements at only single or at resonant frequency ▪ Suitable for small size samples
Parallel Plate	ε_r	▪ Higher accuracy ▪ For thin, flat surface samples ▪ Suitable for high-loss materials ▪ Measurements relatively easier	▪ Support for low frequency (best results) ▪ Electrode polarization effect
Planar Transmission	ε_r	▪ Simple, cost-effective and rapid ▪ No special sample handling ▪ Can used for Solid/Liquid ▪ High temperature measurements	▪ Low quality factor ▪ Air gap causes error

The dielectric response of materials depends on temperature and frequency [19]. The complex permittivity represents the polarization and conduction of materials in an external electric field [20] and the total polarizability of a material can be expressed by the relative permittivity, ε_r, as shown in equation 1 [14].

$$\varepsilon_r = \varepsilon^{'} - i\varepsilon^{'} \qquad (1)$$

Where,

$\varepsilon^{'}$ = the material's dielectric constant, a measurement of how much polarizability is present in a material in the electric field.

$\varepsilon^{''}$ = the dielectric loss factor, a measurement of how dissipative a material is in the electric field.

$i = \sqrt{-1}$ = complex operator.

A dielectric value is the capacity of a material to store and permit electromagnetic energy passages when an electrical field is imposed upon it [21]. The researchers also added the dielectric value as a measure of the material's ability to become polarized, and thus respond to electromagnetic wave propagation. According to the reseachers, material dielectric properties are related to the dipole moment and are affected by the frequency of applied electric field, temperature, and composition of materials. Every material have different electrical characteristics and these characteristics are dependent on the material dielectric properties [18]. The scholars stated that, valuable information is provided by these properties which help engineers and researchers in utilizing such information in design, material characterization or for monitoring process quality.

3. Applications of Microwave in Highway Engineering

Microwave has a great potential to generate alterations in physicochemical and physical properties of bitumen [22], because bitumen properties change when it is subjected to microwave energy. Based on this, efforts have been dedicated previously by researchers toward the development of electromagnetic application in highway engineering and some are presented below.

The dielectric behaviour of hot-mix asphalt mixtures in the range of 200 MHz – 13 GHz frequencies by means of open-ended coaxial probe method at room temperature (25° C) [21]. The dielectric constants of the constituent materials of the mixtures consisting of bitumen, filler and aggregate were first investigated separately, and afterwards the dielectric constant of 69 prepared samples of the compacted asphalt mixtures. The dielectric constant of the bitumen was observed to be about 2.8 at frequency below 7 GHz. However, when the frequency was increased to 13 GHz, there was gradual decrease in the dielectric constant to 2.6. The researchers stated bitumen at molecular level consists of many different hydrocarbons like aromatic and asphaltene which contribute to polarity. When a material is subjected to an external electric field the dipole rotation at low frequencies follow the field with ease and thereby causes an increase in the dielectric constant of the material. But with further increase in frequencies, the field changes very quickly and because the dipole cannot go along with the field, the dielectric constant decreases. There was direct relationship between the density of the compacted asphalt mixtures and dielectric constant, and concluded that as the increase in density increases the dielectric constant [21].

A research was performed to compare two aging techniques (the microwave versus the RTFO-PAV) using two different approaches, High-Performance Size Exclusion Chromatography (HPSEC) (gravimetry and UV absorption), and Bending Beam Rheometer (BBR) [22]. It is noticed that when an irregular organic molecule with dipoles are exposed to microwave radiation, the molecules undergo excitation rotation and this rotation is dependent on the dielectric constant of a material. As a result of the radiation frequency is comparatively high and bitumen's dielectric constant is low, molecules of the bitumen cannot rotate rapidly like the applied electromagnetic field, and thus an out-of-phase component of the dielectric constant (dielectric loss) is dissipated as heat. The use of microwave radiation as a substitute tool for laboratory aging is promising in view of its ability in increasing the Molecular Size Index (MSI) (a behavior synonymous to aging) of bitumen [22]. The researchers also stated that Microwave energy aging is also very simple and quick (less than 3 hours as compared to combine 1.5 hours in RTFO and 20 hours in PAV). Similar findings was also reported on the aging of modified bitumen using two different approaches, microwave and RTFO-PAV [23]

A dielectric spectroscopy measurement on clean bitumen in different grades and Wax Modified Bitumen (WMB) both in a low-frequency range (10^{-2} – 10^{6} Hz) [14]. It was found that the dielectric response of the pure bitumen is dependent on temperature and frequency [19, 21]. Although their result showed no remarkable changes in the dielectric constant between different grades of bitumen of the same source, however, regular variations in the dielectric loss tangent (tan δ) between the grades was noticed, and this indicated that there was a good relationship between the dielectric and rheological responses.

A study was carried out on bitumen blended with rubber of different surface areas using Microwave Spectroscopy (MS) [24]. Generally, rubber if not treated soaks up maltenes (the light fractions) from bitumen when blended at high temperatures. This changes the properties of the bitumen and makes it to be hard and brittle, thereby decreasing pavement service life. Thus, the researchers first treated the rubber with two separate oxidizing solutions. After the rubber oxidation, the non-oxidized and oxidized rubber were cross-linked in the bitumen. The researchers stated that in order to ascertain the effectiveness of the treatment, the treated rubber will soak up a smaller amount of maltenes (light fractions) in comparison with the untreated rubber. Therefore, the lesser the amount of absorption, the higher the proportion of maltenes (light fractions) in the rubber – bitumen blends closer to the neat bitumen, and the closer the rubber – bitumen blends dielectric properties similar to neat bitumen samples. The mixing of bitumen and rubber was carried out for 1 hour at 170 – 180° C in beakers with various percentages of polyethyleneimine (3, 6, and 9 %) to attain optimal chemical treatment. Eleven (11) samples were prepared and the rubber – bitumen mixtures were allowed to cooled and analyzed using microwave spectroscopy analysis. The microwave spectroscopy analysis was carried out by transmitting electromagnetic waves to each of the samples at frequency of 2.329 – 2.347 GHz. Thereafter, the mixtures were cured in an oven at 170 – 180° C for another 6 hours and again analyzed using MS analysis. Electromagnetic waves response curves for the mixtures before and after curing were plotted and compared. Some of the curves were very close to that of the neat bitumen while some were far away from it [24]. The researchers further stated that those curves close that of the neat bitumen were curves of rubber – bitumen mixtures that have similar dielectric properties with the neat bitumen. The experiments were repeated on the samples and the results found were similar. The researchers concluded that Microwave Spectroscopy (MS) has proven to be promising technique from this study and has advantages over existing techniques like: ASTM D2007 (which requires large volume of absorbents and solvents, long analysis periods, and constant supervision); ASTM D (which is very complex, and requires plenty time, solvents and effort); Fourier transform infrared, high performance liquid chromatography, ultraviolet visible spectroscopy, and thermogravimetric analysis (which require days in analyzing bitumen and finding out the complete information about its physical and chemical behaviour as well as its composition); flash chromatography (which requires a lot of sample preparation and can takes hours in completing the analysis of each sample). Some of the advantages include: very simple in terms of sample preparation; only one solvent is required during sample preparation; takes minutes instead of hours to complete sample analysis; the technique is also nondestructive.

A research on the relationship between bitumen penetration, viscosity and dielectric constant values were conducted [25]. In this work, three (3) different grades of bitumen were used and these include 80 – 100, 60 – 70, and PG 76. Each of these bitumen grades was blended with cellulose oil palm fiber (at 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0 %), ethylene vinyl acetate (at 0, 2, 4, 6, 8, and 10 %) and tire rubber powder (at 0, 2, 4, 6, 8, and 10 %). The dielectric properties of the pure and modified bitumen were measured using microwave free-space method at X-band frequency of 10 GHz, while the penetration and viscosity values of the pure and modified bitumen were determined using the conventional methods. The researchers prepared 48 samples and tested the samples at five (5) distinct temperatures (25, 30, 35, 40 and 45° C) for each of the penetration, viscosity and dielectric constant tests. Thereafter, the graphs of penetration against dielectric constant and viscosity against dielectric constant were plotted. From the graphs, there was decrease in dielectric constant as the penetration increases, and on the contrary, there was decrease in dielectric constant as viscosity decreases [25]. Therefore, the researchers concluded that the decrease in the dielectric constant was as a result of temperature increase which increase the penetration and decreases viscosity.

4. Recommendation

Various techniques have been developed in the past to study material's dielectric properties which are dependent on temperature and frequency range. It has been shown that there are several factors to justify which technique to be selected and some of the factors include the nature of the material, measuring temperature and frequency range. Thus, it is necessary to consider these factors to get a more accurate result. Some of the techniques were used to investigate the properties of bitumen and reported to be simple, very quick and cost-effective compared to conventional techniques. The use of microwave techniques if fully developed, one equipment can be used to conduct many tests. For example, instead using RTFO and PAV machines to aged bitumen, one microwave equipment can be used. Also, in the case of penetration, softening point and viscosity tests instead of using separate equipment, one microwave equipment is enough.

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