Development of Calibration Equation Based on Complex Permittivity of Hevea Rubber Latex

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ABSTRACT— In rubber industry, the price of Hevea latex depends on the percentage of moisture content. Since water occurs on most materials in nature as a natural component of the material or is introduced during technologies processes, it is quite obvious that mesasurement and control of moisture content have great economic and technical importance. Thus, in this work, measurements of loss tangent have been done using Agilent Dielectric Probe Kit 85070E. The calibration equations have been established between 1 GHz to 5 GHz. It was found to be most accurate at 5 GHz with lowest mean error, 0.07 using the loss tangent method.

Keywords— Complex permittivity, loss tangent, rubber latex, calibration equation

1. INTRODUCTION

The Hevea Rubber Latex is extracted from the bark of *Hevea brasiliensis* by systematic excisions of the external tissues. *Hevea brasiliensis* type is preferred tree because it yields high quality latex over a sustainable period and is amenable to tapping. Hevea rubber latex is a biological product with a complex composition of about 55 % to 80 % water, 15 % to 45 % rubber hydrocarbon and about 2 % to 4 % non-rubber constituents [1]. This composition varies widely according to season, weather, soil condition, clone, tapping system, etc [2]. Apart from water, other non-rubber constituents includes proteins, lipids, quebrachitol (methyl inositol), and inorganic salt (i.e. potassium, phosphate ions and traces of copper, iron, sodium, calcium and magnesium) of about 0.5 % [1].

One of the properties of microwaves is the ability to travel through nonconductive materials. In materials with bipolar molecular structure, (i.e. water), the electric field of microwaves can induce oscillations whilst travelling through the medium. During this process, the microwaves lose some of their energy. This loss in energy increase with the amount of water that medium contains, with the result that as the water concentration increase, less energy will reach the other side of the medium. Water not only absorbs but also reflects some of the microwave energy.

By utilising these two effects (reflection and absorption), either one is able to perform moisture measurements with microwave techniques. During transmission, (where the substance to be analysed is placed between the microwave emitter and detector) the microwave intensity arriving at the detector decreases with increasing of moisture. The moisture content can then be calculated by taking into account the absorption of the dry substance and some geometrical factors.

In reflection the procedure is equivalent, exempt that both microwave emitter and detector are mounted on the same side of the substance. The microwaves do not heat or change in any way the material; due to the extremely low energy emitted (it would need approximately one thousand emitters to cause any measurable effect) [3].

Testing with microwave is dominated by the basic properties of microwaves. Since their penetration in good conducting materials is minimal, they are mainly used to test the non-conducting materials. On the other hand, microwaves are affected by a large number of material properties. In lossless or lossy dielectrics, material composition, uniformity of the material, moisture and contamination content and such diverse properties as porosity are some of the properties that can be measured.

The interest in this work is the interaction of microwaves with materials. This takes the forms of absorption in materials, scattering, attenuation, reflection and transmission. These effects are exploited in various testing arrangements to allow for quantitative measurements in materials.

2. MATERIAL AND METHODS

2.1 Sample preparation and Moisture Content Measurement

Fresh Hevea latex samples were obtained from Universiti Putra Malaysia's Research Park. Moisture content of freshly tapped Hevea latex samples were approximately 42 % moisture content, m.c which were considerably lower than typical values about 65 % m.c reported in the literature [1]. The latex was diluted with ammonia to prevent coagulation. The mass of ammonia has been negligible always less than 0.39 %. Different percentages of moisture content in latex were obtained by diluting the latex with different volumes of deionised water as shown in figure 1. The mass of fresh and diluted latex samples were recorded using a Shimadzu AY220 electronic balance with a \pm 0.1mg precision.

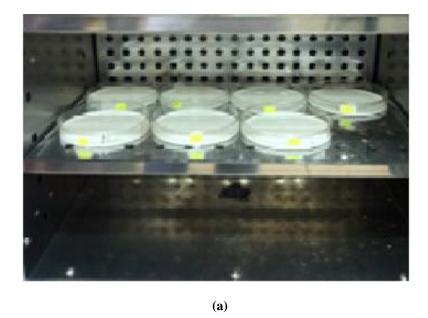


Figure 1: The Hevea Rubber Latex Samples

The actual moisture content in Hevea latex was determined using conventional, standard an oven drying method. Each sample was dried in an oven at 70 $^{\circ}$ C for 18 hours followed by a six hours further drying at 105 $^{\circ}$ C [4]. The dried samples were allowed to cool at room temperature 25 $^{\circ}$ C before weighing. The process was repeated until a constant mass \pm 0.5 mg was obtained for each sample.

Moisture content (%) =
$$(m_{\text{wet}} - m_{\text{drv}})/m_{\text{wet}} \times 100 \%$$
 (4.1)

where $m_{\rm wet}$ and $m_{\rm dry}$ are the initial and final mass before and after drying. Figure 2 show both variation % m.c Hevea Latex before and after drying using a conventional drying oven.



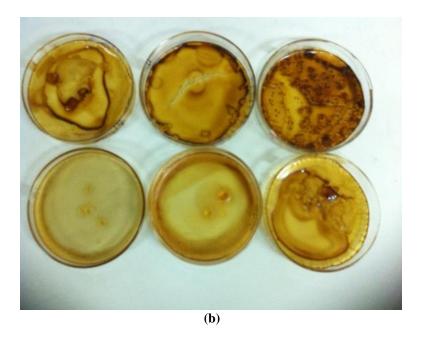


Figure 2: Variation % m.c Hevea Latex (a) before and (b) after drying using a conventional drying oven

2.2 Complex Permittivity Measurement of the Samples

In this work, the permittivities of the samples were measured using the Agilent Dielectric Probe Kit 85070E. Following the manufacturer's recommended procedure, three standard materials were used for calibration, i.e, air, short and distilled water. The measurement accuracy achievable was within ± 5 % and ± 3 % for the real, ε' and imaginary parts of permittivity, ε . The Dielectric Probe Kit automatically determined the complex permittivity of the material under test by measuring both the magnitude and phase of the reflection coefficients.

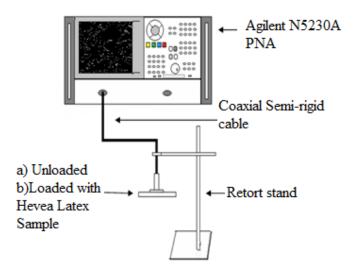


Figure 3: Complex Permittivity Measurement of the different moisture content of Hevea Rubber Latex and Water

3. RESULTS AND DISCUSSIONS

3.1 Loss Tangent, tan δ

The frequency dependence of the loss tangent, $\tan \delta$ at different m.c levels are illustrated in Figure 4 (a) and (b) for frequencies below and above 2.0 GHz, respectively. The loss tangent, $\tan \delta$ follow closely the profile of the loss factor, ϵ " starting with decreased values with frequency from 0.1 GHz to 1.5 GHz for all m.c followed by a slight broad minimum region between 1.5 to 2.0 GHz before drastically increased from 2.0 GHz to 5.0 GHz.

As stated earlier, the loss mechanism below 2.0 GHz in Figure 4(a) was predominantly influenced by ionic conduction. The electric 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 bond with water, both of which result in the generation of heat [5]. Figure 4 (b) suggests a linear increase in $\tan \delta$ with frequencies from 2.0 GHz to 5 GHz. This was due to dipole polarization that is similar to earlier description on the variation in loss factor, ϵ " with frequency. Moreover, the higher the m.c, the higher shall be the $\tan \delta$ values as water is the main contributor in dipole polarizations. Oscillating fields will increase dipoles rotational and vibrational energies resulting in frictional generation of heat energy. It should be noted that molecules are non-polar but asymmetrically charged may behave as dipoles in electric field; however, their responses to microwave energy are negligible [5].

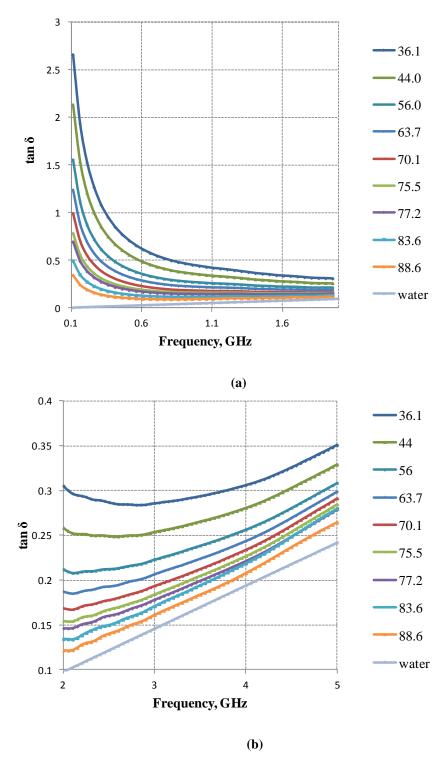


Figure 4: Relationship between (a) Loss tangent, $\tan \delta$ below 2.0 GHz and (b) above 2.0 GHz with frequencies of Hevea rubber latex for differences percentages of m.c

The regression coefficients of the relationship between loss tangent and frequency for water and latex samples below and above 2.0 GHz are listed in Table 1(a) and (b) respectively. Sensitivity analyses ($\Delta \tan \delta / \Delta f$) are also provided but only for linear relationships observed above 2.0 GHz. It can be clearly seen that water has the highest R^2 values for all frequencies. This strongly suggests the influence of the effects of ionic conduction and dipole polarization of water below and after 2.0 GHz.

Table 1: Regression equation and regression coefficient of relationship between loss tangent (a)below and (b)above 2.0 GHz, respectively for differences percentages of moisture content

Moisture content, m.c (%)	Regression equation	Regression Coefficient, R ²	
36.1	$\tan \delta = 2E + 06 f^{0.726}$	0.99	
44.0	$\tan \delta = 767891 f^{0.701}$	0.98	
56.0	$\tan \delta = 75240 f^{0.611}$	0.94	
63.7	$\tan \delta = 75240 f^{0.611}$	0.94	
70.1	$\tan \delta = 23079 f^{0.562}$	0.92	
75.5	$\tan \delta = 5577.1 f^{0.5}$	0.87	
77.2	$\tan \delta = 2798.2 f^{0.472}$	0.84	
83.6	$\tan \delta = 273.15 f^{0.369}$	0.7	
88.6	$\tan \delta = 23.967 f^{0.26}$	0.48	
water	$\tan \delta = 3E-11 f^{1.0208}$	1	
	(a)		

Moisture content, m.c (%)	Regression equation	Regression, R ²		
,				
36.1	$\tan \delta = 1 \times 10^{-20} f^2 - 8 \times 10^{-11} f + 0.4079$	0.9923		
44.0	$\tan \delta = 1 \times 10^{-20} f^2 - 6 \times 10^{-11} f + 0.3238$	0.9976		
56.0	tan δ = 9 x 10 ⁻²¹ f^2 - 3 x 10 ⁻¹¹ f + 0.2338	0.9992		
63.7	$\tan \delta = 8 \times 10^{-21} f^2 - 2 \times 10^{-11} f + 0.191$	0.9996		
70.1	$\tan \delta = 8 \times 10^{-21} f^2 - 1 \times 10^{-11} f + 0.1595$	0.9998		
75.5	$\tan \delta = 6 \times 10^{-21} f^2 - 2 \times 10^{-12} f + 0.1298$	0.9997		
77.2	$\tan \delta = 6 \times 10^{-21} f^2 + 3 \times 10^{-12} f + 0.1146$	0.9997		
83.6	$\tan \delta = 6 \times 10^{-21} f^2 + 1 \times 10^{-11} f + 0.0904$	0.9996		
88.6	$\tan \delta = 4 \times 10^{-21} f^2 + 2 \times 10^{-11} f + 0.0645$	0.9997		
water	$\tan \delta = -9 \times 10^{-23} f^2 + 5 \times 10^{-11} f - 0.0005$	1		
(b)				

3.2 Variation in Loss tangent with m.c

The effect of m.c on loss tangent at the selected frequencies can be observed in Figure 5. It is interesting to note that although both ε ' and ε " are proportional to m.c as shown in Figure 4, however tan δ has inverse proportional relationship with m.c for frequencies above 2.0 GHz. Additionally, the values of tan δ is always lower than 1 for the samples as shown in figure 5. This is expected as the loss factor, ε " is always lower than dielectric constant, ε ' for all samples [6]. This confirms that water has higher tendency to store energy rather than dissipating the energy at all frequencies below 5 GHz [7]. Table 2 shows both of the sensitivity ($\Delta m.c$ / $\Delta \tan \delta$) and regression coefficient, R^2 are increasing at the selected frequency.

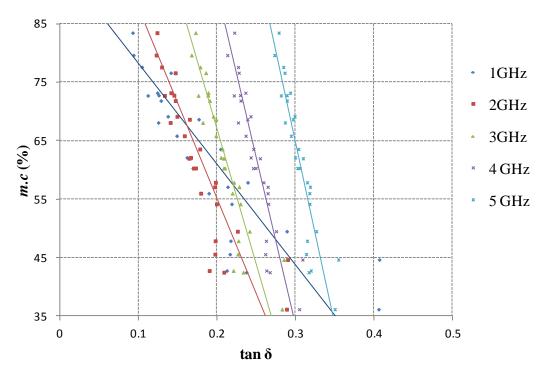


Figure 5: Variation in percentage moisture content, m.c (%) with Loss tangent, $\tan \delta$

3.3 Development of Calibration equation for determination of moisture content at 1 GHz to 5 GHz

The calibration equations at several frequencies for determination of m.c based on tan δ have been listed in table 2. The calibration equations based on tan δ methods were found to be most accurate at 5 GHz with obtained the lowest mean relative error, 0.07. Thus, it can be concluded that the most accurate equation to predict m.c in Hevea Latex is based on the measurement of tan δ at 5 GHz, i.e.

$$m.c = -619.56 \tan \delta + 250.38 \tag{5.1}$$

The very high sensitivity value 0.8818 indicates almost perfect one-to-one correspondence between predicted and actual m.c for tan δ method has been showed in figure 6.

Table 2: Calibration equation, regression coefficient and sensitivity of relationship between moisture content and Loss tangent of Hevea Rubber Latex at various selected of frequency

Frequency	Calibration equation	regression	Sensitivity,
(GHz)		coefficient, R ²	$(\Delta m.c/\Delta \tan \delta)$
1	$m.c = -170.01 \tan \delta + 94.587$	0.7603	170.01
2	$m.c = -321.36 \tan \delta + 119.13$	0.8054	321.36
3	$m.c = -562.39 \tan \delta + 202.4$	0.8491	562.39
4	$m.c = -458.72 \tan \delta + 158.44$	0.8433	458.72
5	$m.c = -619.56 \tan \delta + 250.38$	0.8818	619.56

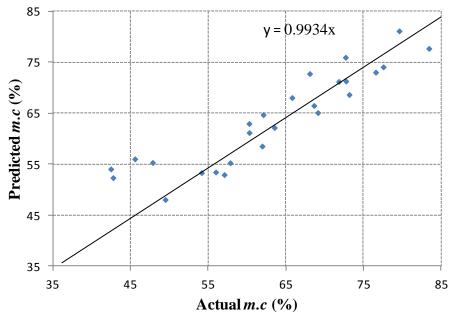


Figure 6: Predicted m.c using empirical equation of tan δ (5 GHz) and actual m.c obtained by using oven drying method.

4 CONCLUSION

The relationships between Loss tangent with frequency and moisture content of hevea rubber have been described. The calibration equation was established to predict moisture content from the measured loss tangent, $\tan \delta$ and was found to be accurate with lowest mean relative error, 0.07 at 5 GHz when compared to actual moisture content obtained from standard oven drying method.

5 ACKNOWLEDGEMENT

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