Permittivity models for determination of moisture content in Hevea Rubber Latex

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Abstract: The commercial open-ended coaxial probe (Agilent 85070E) is the most commonly used sensor to determine the permittivity of wet materials. This paper extends the usability and applicability of the sensor to the estimation of moisture content in Hevea Rubber Latex. The dielectric constant and loss factor were measured using the commercial probe whilst the moisture contents were obtained using the standard oven drying method. Comparison results were obtained between the different dielectric models to predict moisture content in latex. Both the dielectric constant and the loss factor of rubber latex linearly increased with moisture content at all selected frequencies. Calibration equations were established to relate both the dielectric constant and the loss factor with moisture content. These equations were used to predict moisture content in Hevea latex from measured values of the dielectric constant and the loss factor. The lowest mean relative error between actual and predicted moisture contents was 0.02 at 1 GHz when using the Cole-Cole dielectric constant calibration equation.

Keywords: open-ended coaxial probe, permittivity models, Hevea Rubber Latex, moisture rubber content
DOI: 10.3965/j.ijabe.20140705.005

Citation: Yahaya N Z, Abbas Z, Ibrahim N M, Hafizi M H M, Yahaya M Z. Permittivity models for determination of moisture content in Hevea Rubber Latex. Int J Agric & Biol Eng, 2014; 7(5): 48－54.

1 Introduction

Hevea Rubber Latex is a biological product tapped from *Hevea brasiliensis* tree. It consists 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]. In the rubber industry, the price of Hevea latex depends on the percentage of dry rubber content (*DRC*) which can be indirectly estimated from moisture content.

A small quantity of ammonia is normally added to hevea latex sample to prevent coagulation. In the direct method of determination of the *DRC*, acetic or formic acid is also added to the sample to separate the rubber solids from the nonsolid materials especially water. The rubber solid is usually pressed, weighed and dried using an oven. The procedure is repeated continuously until a constant mass is obtained. The conventional oven drying method is time consuming and laborious. The standard method to determine the dry rubber content can also be determined indirectly by measuring the moisture content in latex. The sample is initially dried for more than 18 hours at 70°C, followed by continuous drying at 105°C until a constant mass within 0.005 grams is obtained[1]. The technique is simple but suffered similar drawbacks as the *DRC* direct method.

Received date: 2013-09-08 **Accepted date:** 2014-10-02

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Recently, monopole sensors[3] have also been proposed to determine moisture content in latex. Different percentages of *m.c* latex will give different phase shifts of the monopole sensor. The phase shift is defined as the difference between the phase of the unloaded sensor and the sensor loaded with latex. Unfortunately, the technique is too sensitive where even a slight cable movement will result in a large error in the measured phase of the sensor. The Agilent 85070B Probe Kit consisting of an open ended coaxial sensor and software has been used to calculate the complex permittivity of latex from the measured reflection coefficient[4] as a function of moisture content at 10.7 GHz. However no attempts were made to evaluate the accuracy of the different dielectric models to predict *m.c* in latex.

In this paper, we proposed an extended application of the Agilent 80507E Probe Kit where the moisture content in latex can be predicted from the measured permittivity. The accuracy of the determination of *m.c* in latex was established by comparing the results of *m.c* obtained using the standard oven drying method as well as with different models.

2 Principle

2.1 Complex permittivity measurement using open ended coaxial probe

The Agilent Open Ended Coaxial probe[5] is now considered as the industry de facto standard for measuring the permittivity of liquid materials. The probe utilized an admittance equation to calculate the permittivity from the measured magnitude and phase of the open ended coaxial sensor placed in direct contact with the material under test, i.e.,

$$Y = (1 - \Gamma) / (1 + \Gamma) \tag{1}$$

Where the normalized aperture admittance can be expressed as[6]

$$\tilde{Y}_L = \frac{jk_m^2}{\pi k_c \ln\left(\frac{b}{a}\right)} \int_a^b \int_a^b \int_0^\pi \cos(\phi) \frac{e^{-jk_m R}}{R} d\phi dr dr' \tag{2}$$

where, R is the distance from source point to a corresponding field point,

And k_m can be defined as the wave number in the external medium,

$$R = \sqrt{r^2 + r'^2 - 2rr'\cos(\phi)} \tag{3}$$

$$k_m = \omega\sqrt{\varepsilon_m \varepsilon_0 \mu_0} \tag{4}$$

where, ε_m is the permittivity of the test material obtained through an optimization routine[7] by fitting the calculated ε_m to the Cole-Cole Model[8].

$$\varepsilon = \varepsilon' - j\varepsilon'' = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 - (j\omega\tau)^{1-\alpha}} \tag{5}$$

Where the constants, $\varepsilon_s = 78.6$, $\varepsilon_\infty = 4.22$, $\tau = 8.8\times10^{-12}$ s and $\alpha = 0.013$.

2.2 Dielectric mixture model of rubber latex

The Cole-Cole Model is essentially a simplified form of the Debye Model[9]. Various dielectric mixture models have also been proposed to predict the permittivity of materials with more than two components. The most popular dielectric mixture models are the Bruggemann, and Krazewski-Weiner Models[10]. However, the Kraszewksi-Weiner Model is still among the most commonly used models due to its simplicity requiring only values of volume fraction and permittivity of each component[11]. The Kraszewski-Wiener Model treats latex as a biphase liquid, consisting of water and solid rubber[12]. In Wiener's upper bound formula the relative dielectric permittivity of the mixture is written as

$$\varepsilon^* = V_1\varepsilon_1 + V_2\varepsilon_2 \tag{6}$$

where, ε_1 and ε_2 are the relative dielectric permittivity for water and solid materials respectively, and V the water volume fraction. Kraszewski et al.[10] derived a simplified version of the Wiener's model in the form

$$\sqrt{*} = v_1\sqrt{1} + v_2\sqrt{2} \tag{7}$$

The volume fraction V is related to the *m.c* (wet basis) by

$$V = M_1\left[M_1 + \left(\frac{D_2}{D_1}\right)\right] \tag{8}$$

M_w is the *m.c*; D_1 and D_2 are the relative density of the water and solid rubber respectively and are considered to be constant with $D_1 = 1.0$ and $D_2 = 0.04$[11]. The permittivity of latex sample can be estimated using Equations (7) and (8). The Kraszewski's Model is almost similar in form to the more popular Laudau's model

$$\sqrt[3]{*} = v_1\sqrt[3]{1} + v_2\sqrt[3]{2} \tag{9}$$

and Lichtenecter's model

$$\ln^* = v_1\ln_1 + v_2\ln_2 \tag{10}$$

The notation used here applies to three-component mixtures where * represents the complex permittivity of the mixture, $_1$ is the permittivity of medium 1, $_2$ is the permittivity of medium 2. v_1 and v_2 are the fractional volume of the respective components, where $v_1 + v_2 = 1$ as the latex mixture consists of two components. The volume fraction of water and solid can be expressed as

$$1 = v_{water} + v_{solid} \qquad (11)$$

3 Materials and methods

3.1 Sample preparation

This study was carried out at the Department of Physics, Faculty Science, Universiti Putra Malaysia. Freshly tapped Hevea latex obtained from Research Park of University Putra Malaysia was used in this study in order to determine its *m.c*. The mass of fresh and diluted latex samples were recorded using electronic balance and dried into microwave laboratory oven at 70°C for 18 hours followed by a six-hour further drying at 105°C[13]. The dried samples were allowed to cool at room temperature 25°C before weighing. The process was repeated until a constant mass ±0.5 mg was obtained for each sample. The dried samples were kept at room temperature before weighing again until it reached a constant value. The actual moisture content was determined using the standard oven drying method[1].

$$\text{Moisture content (\%)} = (m_{wet} - m_{dry})/m_{wet} \times 100\% \qquad (12)$$

where, m_{wet} and m_{dry} are the initial and final mass before and after drying.

3.2 Measurement set-up

The experimental setup consists of an open-ended coaxial probe, Hevea latex samples with various moisture contents. The Professional Network Analyzer (PNA) was used to measure the dielectric constant and loss factor of Hevea latex in frequency range between 0.1-5.0 GHz. The calibration procedure was performed using Agilent's open, short and load standards from 0.1 GHz to 5.0 GHz to establish a 50 ohm calibration plane between the sensor and the coaxial cable. For liquid samples air bubbles on the tip of the probe can be a significant source of error.

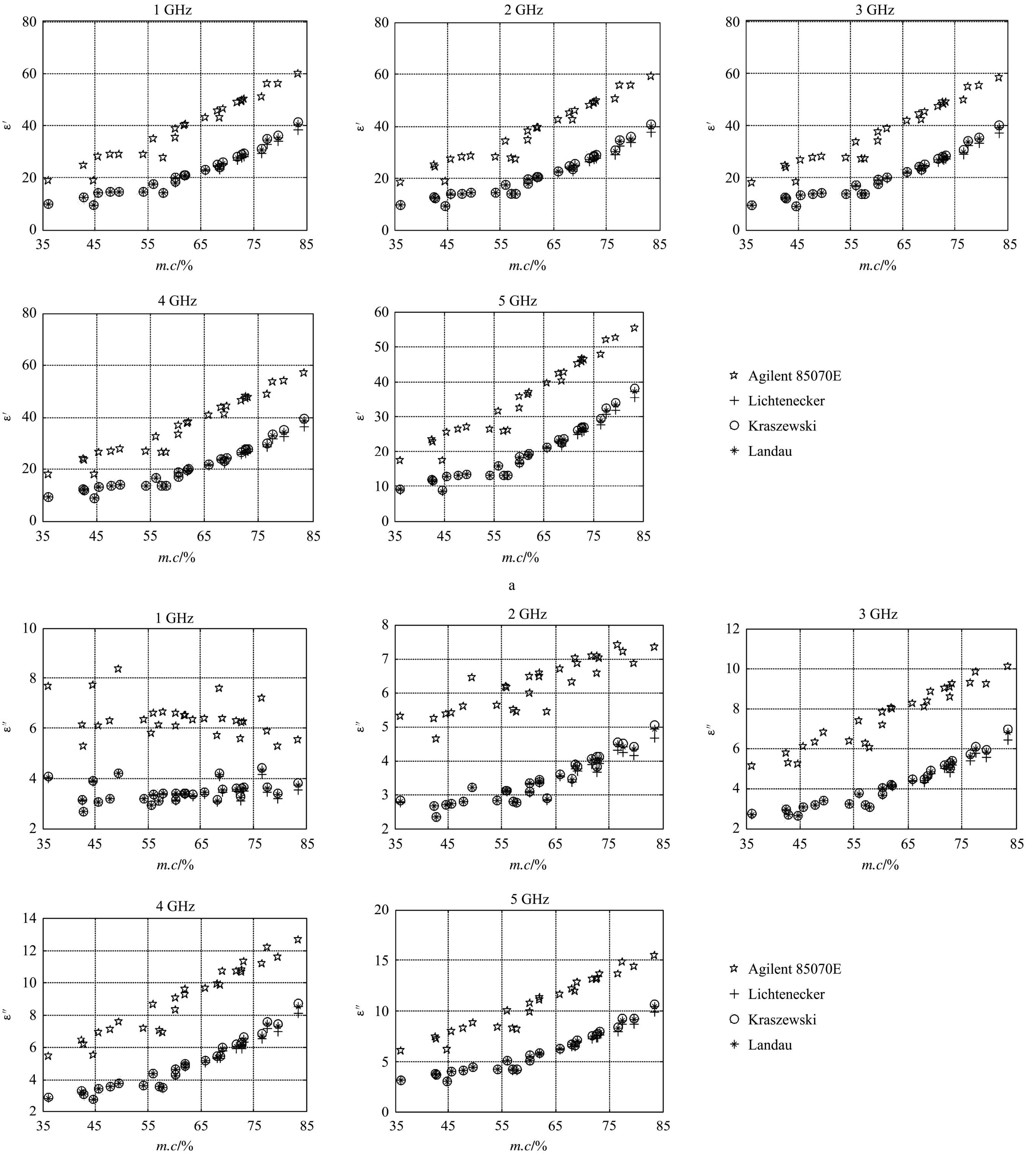
4 Results and discussion

4.1 Variation in the Cole-Cole's dielectric constant and the loss factor with *m.c*

The effect of *m.c* on the values of the dielectric constant, ε' and loss factor, ε'' on Hevea latex samples obtained using the Cole-Cole (Agilent Probe) and the dielectric mixture models are shown in Figures 1a and b, respectively for several selected frequencies. Theoretically, the higher *m.c* in a sample, the higher shall be the permittivity of the sample. It can be clearly seen that the Cole-Cole results for both ε' and ε'' obtained from equation (5) are higher than the Kraszewki, Lichteneker and Landau models. Interestingly, both ε' and ε'' for all the models show almost constant values in the *m.c* region 45% and 65%, typical range of *m.c* of fresh latex. Nevertheless, it can be clearly seen from the graphs that generally both ε' and ε'' vary almost linearly with moisture content for all the frequencies. However, the variation in ε'' with moisture content was less promising as shown in Figure 1b especially at low frequencies 1 GHz and 2 GHz. At low frequencies, changes in loss factor are very small. A rapid change in loss factor is observed when frequency is raised above 3 GHz, especially at higher moisture levels. The high uncertainties in both ε' and ε'' at low *m.c* values were due to non-uniform vibration of bound water molecules[14].

The dynamic range of ε' is much higher than ε'' for the whole range of *m.c*. The *m.c* range between 35% and 85% corresponds to ε' from 20 to 60 for all frequencies for the Cole-Cole results. Also, the dynamic range of ε'' increases with frequency from 7.67 to 5.01 (1 GHz), 5.32 to 7.34 (2 GHz), 5.13 to 10.76 (3 GHz), 5.43 to 14.01 (4 GHz) and 6.05 to 16.33 (5 GHz). The loss factors were almost similar at all frequencies at low *m.c* due to strong binding between hydrogen and oxygen molecules. Higher *m.c*, allows free movement of water molecules and would result in higher energy dissipation with increasing frequency due to the greater effect of dipole polarization.

In general the dielectric constant changes about 9 units for every 10% change in *m.c* in the latex sample when using the Cole-Cole model. This could also mean that the sensitivity of the dielectric constant with respect to *m.c* is approximately 1; i.e every 1% change in moisture content resulted in 0.9 unit change in the value of the dielectric constant. However, the variation in the loss factor with moisture content was less promising as shown in Figure 1b, especially at low frequencies.



a

b

Figure 1 (a) Dielectric constant and (b) Loss factor of Hevea Rubber Latex at various selected frequencies

Empirical models to relate the dielectric constant and loss factor to *m.c* using the different permittivity models are represented by the regression equations listed in Table 1. Both Tables 1 (a) and (b) suggest higher R^2 values could be obtained at higher frequencies approaching free water relaxation frequency as described by the Debye–type relaxation spectral function[15]. The sensitivity which is the gradient of the regression line is defined as the change in the output ($\Delta\varepsilon'$ or $\Delta\varepsilon''$) with respect to the input ($\Delta m.c$). The range of sensitivities, $\Delta\varepsilon'/\Delta m.c$ (Table 1(a)) was from 0.8937 to 0.9382 with almost similar R^2 with a mean value 0.97162. In general, the dielectric constant changes approximately 9 units for every 10% change in *m.c* in the latex sample. This could also mean for every 1% change in moisture content resulted in 0.9 unit change in the value of the dielectric constant.

Table 1 Regression equation, regression coefficient and sensitivity for relationship between (a) dielectric constant and (b) loss factor of Hevea Rubber Latex and moisture content at various selected frequencies

(a)

Frequency /GHz	Regression equation	Regression coefficient, R^2	Sensitivity
1	$\varepsilon'=0.9382\ m.c-18.527$	0.9708	0.9382
2	$\varepsilon'=0.9388\ m.c-19.189$	0.9713	0.9388
3	$\varepsilon'=0.9292\ m.c-19.169$	0.9718	0.9292
4	$\varepsilon'=0.916\ m.c-19.156$	0.9719	0.9160
5	$\varepsilon'=0.8937\ m.c-18.801$	0.9723	0.8937

(b)

Frequency /GHz	Regression equation	Regression coefficient, R^2	Sensitivity
1	$\varepsilon''=-0.0411\ m.c+8.8162$	0.4809	0.0411
2	$\varepsilon''=0.0434\ m.c+3.5483$	0.7866	0.0434
3	$\varepsilon''=0.1042\ m.c+1.1623$	0.9475	0.1042
4	$\varepsilon''=0.1546\ m.c-0.5549$	0.9634	0.1546
5	$\varepsilon''=0.2017\ m.c-1.6942$	0.9691	0.2017

The sensitivities, $\Delta\varepsilon''/\Delta m.c$ (Table 1(b)) were much lower ranging between 0.0411 and 0.2017 with mean R^2 =0.8295. The wider range of $\Delta\varepsilon''/\Delta m.c$ means the change in ε'' with changes in *m.c* should be predicted at a specific frequency. For example, every 1% change in *m.c* will result in a change of 0.201 and 0.0434 in ε'' at 5 GHz and 2 GHz, respectively.

The empirical equations in Table 1 can be used to determine both ε' and ε'' from known *m.c* values. Inversely, the permittivity values obtained using the Agilent Coaxial Probe in conjunction with the Cole-Cole model can be used to predict *m.c* in latex samples by exchanging the ε' and ε'' with *m.c* of *x*-*y* graph. Comparisons between predicted and actual *m.c* using the equations in Table 2 and the oven drying method, respectively are shown in Figure 3.

Variation in Cole_Cole's Loss tangent with m.c

The effect of *m.c* on loss tangent at the selected frequencies can be observed in Figure 2. It is interesting to note that although both ε' and ε'' are proportional to *m.c* as shown in Figure 1, tanδ has an inversely proportional relationship with *m.c* for frequencies above 1.5 GHz.

Table 2 Calibration equation, regression coefficient and sensitivity of relationship between moisture content and (a) dielectric constant, (b) loss factor of rubber latex at various selected frequencies

(a)

Frequency /GHz	Calibration equation	Regression coefficient, R^2	Sensitivity
1	$m.c = 1.0347\varepsilon'+21.127$	0.9708	1.0347
2	$m.c = 1.0346\varepsilon'+21.777$	0.9713	1.0346
3	$m.c = 1.0458\varepsilon'+21.939$	0.9718	1.0458
4	$m.c = 1.061\varepsilon'+22.208$	0.9719	1.0610
5	$m.c = 1.0879\varepsilon'+22.312$	0.9723	1.0879

(b)

Frequency /GHz	Calibration equation	Regression coefficient, R^2	Sensitivity
1	$m.c = -11.69\varepsilon''+137.64$	0.4809	-11.6900
	$m.c = 18.131\varepsilon''-50.124$	0.7866	18.1310
	$m.c = 9.0889\varepsilon''-7.0457$	0.9475	9.0889
	$m.c = 6.2295\varepsilon''+5.9125$	0.9634	6.2295
	$m.c = 4.8048\varepsilon''+10.211$	0.9691	4.8048
		mean	5.3128

(c)

Frequency /GHz	Calibration equation	Regression coefficient, R^2	Sensitivity, ($\Delta m.c/\Delta\tan\delta$)
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

Additionally, the values of tanδ are always lower than 1 for the samples as shown in Figure 2. This is expected as the loss factor. ε'' is always lower than dielectric constant, ε' for all samples[16]. This confirms that water has higher tendency to store energy rather than dissipating the energy at all frequencies below 5 GHz.

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

The calibration equations for determination of *m.c* based ε' and ε'' are listed in Table 2. Comparisons between the predicted and actual *m.c* using the calibration equations and oven drying methods, respectively were made. The most accurate equation to predict *m.c* in Hevea Latex within 2.0% was found based on the measurement of dielectric constant at 1 GHz where the relationship between the predicted (Y) and actual *m.c* (x) was

$$Y=0.9518x \tag{13}$$

The very high sensitivity value 0.9518 indicates almost perfect one-to-one correspondence between predicted and actual *m.c*. The accuracy of the calibration is highly dependable on the accuracy of the permittivity Cole-Cole model adopted by the Agilent 85070E Probe Kit. Figure 2 compares the predicted *m.c*. using all the permittivity models with the actual *m.c*. It can be clearly seen that the Cole-Cole model prediction of m.c is always more accurate than the Kraszewski-Weiner, Landau and Lichtneker models. It is envisaged that higher accuracy in the prediction of *m.c* in latex could be obtained by fitting the calculated ε_m to a more suitable dielectric mixture model. In contrast, the Cole-Cole mode is much more accurate than the kraszewski-Weiner, Landau and Litchneker Models.

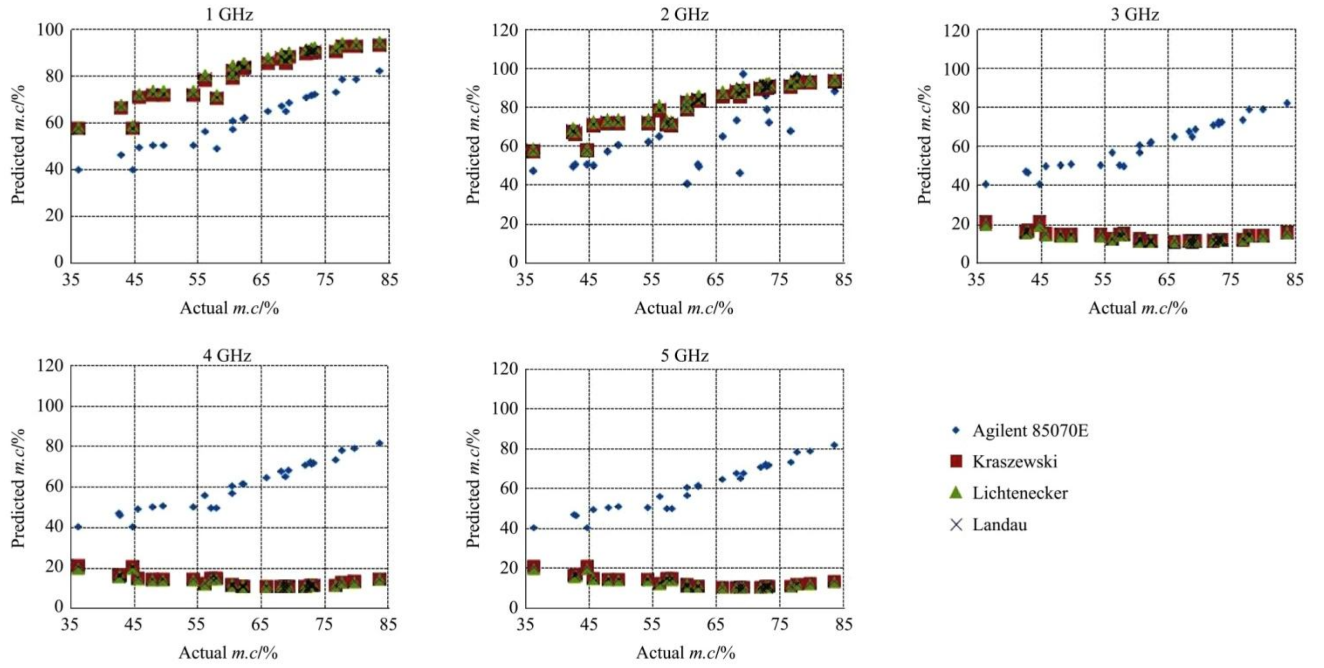


Figure 2 Predicted *m.c* using the empirical equation in Table 2(a) and Table 3(a) and actual *m.c* obtained by the oven drying method for ε' technique

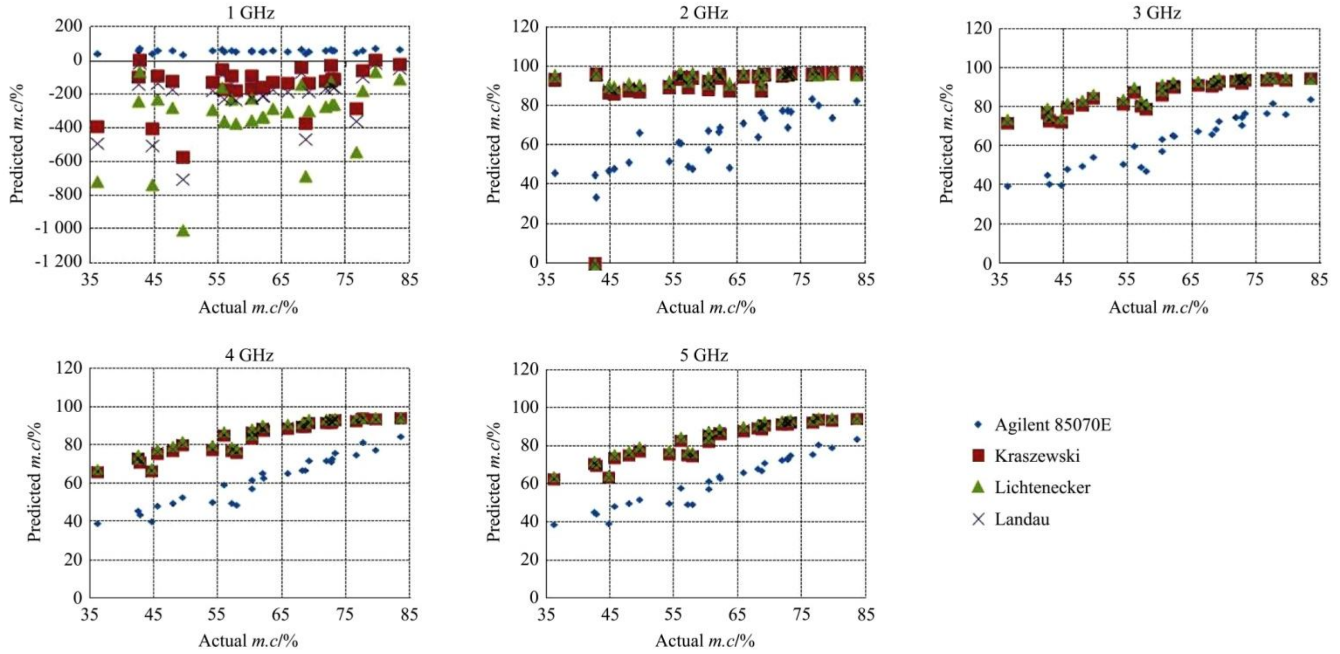


Figure 3 Predicted *m.c* using the empirical equation in Table 2(b) and Table 3(b) and actual *m.c* obtained by the oven drying method for ε'' technique

5 Conclusion

A commercial open-ended coaxial sensor for accurate measurement of moisture content in Hevea latex has been successfully used to determine moisture content in latex based on reflection measurement. The accuracy of the sensor was determined by comparing the predicted moisture content with the actual moisture content using oven drying. The moisture content predicted by the dielectric constant technique has been proved with an mean relative error of 2.0%.

Acknowledgement

The authors acknowledge the financial assistance for this research from Ministry of Science, Technology and Innovation (MOSTI) for National Science Fellowship (NSF).

List of Abbreviations

m.c: Moisture content
GHz: Gigahertz
DRC: Dry rubber content

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