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Original Article

# Design and implementation of microwave attenuation measurements to estimate the dry rubber content of natural rubber latex

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# Abstract

An accurate, quick and practical method for measuring the dry rubber content (DRC) in natural rubber latex is proposed and investigated experimentally, based on microwaves. The concept exploits the microwave absorption of water molecules to infer the DRC. However, various design aspects had to be experimentally investigated. In this study, rectangular waveguide antennae were used for microwave transmission. The latex was placed between two antennae, and the signal was analyzed by a vector network analyzer (VNA). A calibration curve was fitted with least squares regression for sample DRCs ranging from 10.26% to 60.63%. The best linearity with 0.9996 correlation between attenuation and DRC was obtained at 2.36 GHz. To validate this measurement technique, 17 fresh local rubber latex samples were measured both with the slow reference gravimetric determination of DRC that involves oven drying, and with the microwave transmission technique. The measurements had a 0.43 mean error in %DRC, and a 0.9047 correlation to the gravimetric DRC. The study supports potential design and construction of a prototype DRC measurement device for field use.

Keywords: dry rubber content, latex, microwave, waveguide

# 1. Introduction

Natural rubber latex is a natural product collected by tapping rubber trees, particularly Brazilian rubber trees (*Hevea Brasiliensis*) that are native in Amazonia. Although there are many plants that provide latex, rubber trees produce large enough quantities with sufficient quality for industrial use in commercial products. The elastomer properties of natural rubber are critical for products such as tires, gloves, hospital materials, and construction materials, among others.

Natural rubber latex (NRL) contains several rubber and non-rubber ingredients suspended in cytoplasm. When the latex is dehydrated, the dry rubber remains. Its initial

\*Corresponding author. Email address: pattara.a@psu.ac.th content (DRC) in the latex varies from approximately 20% to 50%. The definition of DRC is the mass in grams of dry rubber in 100 g of fresh latex (Rejikumar *et al.*, 2010). There are in latex some 2-4% of non-rubber substances, including lutoids, proteins, lipids, carbohydrates, and inorganic salts (Nair *et al.*, 1993). The DRC determines the market value of NRL, and it varies by the strain and age of rubber trees, the season, tapping intensity, and chemical stimulation, among other factors.

DRC determination methods are either direct or indirect measurements. The standard method uses direct determination (ISO 126:1972 Dry rubber content, BS 1672: part 1:1950 and ASM D 1076-80). This standard method anneals the NRL sample with a dry heat oven, after thickening the NRL with 2% acetic acid to form a rubber sheet. The DRC is determined from sample weights before mixing in the acid and after the drying. This method is accurate but time consuming and sometimes there are mass losses during oven drying, due to sample disintegration. Attempts have been made to shorten the measurement time by using a microwave oven, but this method appears sensitive and not robust (Julrat et al., 2012). The indirect approaches use measured physicochemical properties of the rubber latex, for example density (Metrolac) (Tillekeratne et al., 1988), equivalence point of titration (Alex et al., 2003), light reflectance (Zhao et al., 2010), microwave resonant frequency (Somwong et al., 2008), microwave reflection (Khalid, 1992; Mohammadi et al., 2011), and microwave transmission (Khalid, 1982; Chung, 2007; Abbas et al., 2001). Among these methods we chose to focus only on microwave techniques. Determining the microwave resonant frequency requires a complex experimental set-up, so we chose the simpler microwave reflection technique that measures the dielectric permittivity of rubber latex, which is strongly related to the DRC. This technique uses a vector network analyzer (VNA) connected to an openended coaxial probe, which both transmits the microwave signal to the sample and receives the reflected signal at the same probe. The reflection coefficient, which depends on the dielectric permittivity of the latex, is the ratio of reflected to incident signal. This method requires that the latex sample is sufficiently thick, and that the flat probe contacts the sample surface without air bubbles or gaps. Robustness of the instrument is also an issue, as the probe might be damaged when dipped into the sample. The transmission technique, on the other hand, does not require dipping, touching the sample, or a precise probe angle, all of which complicate the reflection technique (Trabelsi and Nelson, 2003). For these reasons, we selected the transmission technique. Khalid (1982) was among the first determining the DRC of fresh latex by microwaves. The attenuation of 10.7 GHz microwaves in latex was measured, and this measurement approximated the standard method with a 0.9980 correlation coefficient and 0.7% standard deviation. This technique reduces the measurement time to three minutes from the 8-16 hours of the gravimetric standard method.

The objectives of our research were to design a rectangular waveguide antenna, to study the relationship between the DRC in latex and the transmission of microwaves, to select the best frequency for the DRC determination of latex, and to develop a model that predicts the DRC of latex from its microwave transmission.

# 2. Materials and Methods

#### 2.1 Principle of measuring DRC

Microwaves are electromagnetic waves in the frequency range from 300 MHz to 300 GHz, or equivalently wavelengths from 1 mm to 1 m. In a hydrated material the microwaves interact with the water molecules that are strongly polar electric dipoles. The interaction strongly orients the water molecules consuming the input microwave energy, and the magnitude of energy loss depends on the

amount of water (Hongliang, 2010). The other substances in rubber latex have much lower dielectric constants than water, so the measurement of water amount is not disturbed by them at the microwave frequencies. By assuming direct proportionality of the total solids content and DRC, the DRC can be estimated from microwave attenuation. The attenuation of microwaves in a lossy medium follows exponential decay (Pengfei *et al.*, 2010), similar to Beer's law for light absorption, as in equation 1.

$$P_2 = P_1 e^{-\alpha x} \tag{1}$$

In Equation 1,  $P_1$  is the input microwave power (mW),  $P_2$  is the microwave power after decay (mW),  $\alpha$  is the decay constant (mm<sup>-1</sup>), and x is the thickness of the medium (mm). The absorbance is also defined similarly as for light absorption

$$A = 10 \log\left(\frac{P_1}{P_2}\right) \tag{2}$$

#### 2.2 Rectangular waveguide antenna

An antenna is a device that can convert electrical energy into electromagnetic waves. It is an important part of a radio system. In transmission, the transmitter generates radio waves and sends signals to the antenna, which radiates the electrical energy as electromagnetic waves (radio waves). In reception, an antenna intercepts electromagnetic waves and provides an electrical signal to an amplifier (Halit, 2013). Microwave transmission requires directional or beam antennae, which are intended to transmit or intercept the electromagnetic waves in a particular direction, or with a directional pattern that focuses the microwaves to the sample. We chose to use a rectangular waveguide antenna. A waveguide antenna transports electromagnetic energy from one spatial region to another, and typical waveguides are metal tubes of rectangular or circular cross section. These can direct the microwave power to the sample, and can handle high power. However, the waveguides only function properly above a certain frequency, called the cutoff frequency. The shape and size of the cross section determine the cutoff frequency of a waveguide. The cutoff frequency for a rectangular cross-section is given by Sadiku's equation (Sadiku, 1995):

$$f_c = \frac{1}{2\pi\sqrt{\mu\varepsilon}}\sqrt{\left[\frac{m\pi}{a}\right]^2 + \left[\frac{n\pi}{b}\right]^2}$$
(3)

where  $\mu$  is the permeability of the medium (H/m),  $\varepsilon$  is permittivity of the medium (F/m), *a* and *b* are side length of the rectangular cross section (m), *m* and *n* denote the number of half-cycle variations in the *x-y* cross section of the waveguide. The cutoff frequency for the TE<sub>10</sub> mode is obtained with *m* = 1 and *n* = 0:

$$f_c = \frac{v}{2a} \tag{4}$$

where v is the velocity of plane wave in the medium (m/s), a is the larger side length of the rectangular cross section (m).

The frequency range from 2.0 to 3.0 GHz was chosen because it gives microwave attenuation directly proportional to the water amount. The width *a* of the waveguide was obtained from Equation 4 as approximately 70 mm (Nutchanat, 2009). The rectangular waveguide was designed with a 70 mm x 70 mm rectangular hollow box section of 3 mm thick aluminum. The probe in the waveguide was a 2 mm diameter copper wire, soldered into an N-Socket and cut to about 36 mm (1/4 free space wavelength) including the protruding bit of the N-Socket, shown in Figure 1.

#### 2.3 Measurement set-up and procedure

A diagram of the experimental setup, used to measure the microwave attenuation through a latex sample, is shown in Figure 2. A pair of rectangular waveguide antennae sandwich a horizontal polystyrene sample holder. The waveguides are connected to an 8510B Vector Network Analyzer



Figure 1. The rectangular waveguide antenna (a) and the probe housed within the waveguide (b).



Figure 2. Schematic of the measurement set-up.

via RG58 coaxial cables and N-Type male adapters. The latex sample holder is vertically between the transmitting and receiving waveguides. The sample holder is a rectangular container, made of polystyrene with 2 mm wall thickness. The width of the sample holder must be large enough to avoid microwave transmission on the sample surfaces, and the sample thickness must be less than the maximum penetration depth of microwaves into the sample. The horizontal cross section of the sample holder was 80 mm ' 80 mm. The network analyzer was calibrated in transmission mode with a through connection of the transmitting and the receiving antennae, to measure the  $S_{11}$  and  $S_{21}$  parameters for reflection and transmission. The microwave attenuation by water in the latex samples is obtained by measuring  $S_{21}$  as follows (Kim and Kim, 2002):

$$S_{21} = |S_{21}|e^{j\Phi}$$
(5)

$$4 = -20 \log |S_{21}| \tag{6}$$

$$\Phi = 2\pi n + \phi \tag{7}$$

where A is the attenuation in dB, F is the total phase, f is the phase shift, and n is an integer.

Since the attenuation depends only on the moisture content in the latex samples, when the wavelength (or frequency) and the sample thickness are held fixed, it is possible to estimate the DRC from the measured  $S_{21}$  value, read from the VNA.

### 3. Results and Discussion

# 3.1 Antenna efficiency

Antenna efficiency is the ratio between the radiated power and the total input power, with the differential power loss converted to heat. The resistance in the antenna's conductors and the dielectric and magnetic core losses in the antenna contribute to the total losses.

The standing wave ratio (SWR) is the amplitude ratio between maximum and minimum amplitudes of a standing wave. The SWR is commonly used to estimate the antenna efficiency in a transmission line with radio transmitters and receivers. An impedance mismatch can reflect the radio waves back into the input cable and damage the input source, and the SWR determines the power reflected from the antenna. In an ideal transmission line none of the input power would be reflected, which corresponds to SWR = 1. The SWR of a transmission line can be measured with an SWR meter, or it can be calculated from the reflection coefficient G as follows (Beatty, 1959):

$$SWR = \frac{1+|\Gamma|}{1-|\Gamma|} \tag{8}$$

where  $|\Gamma|$  is the absolute magnitude of the reflection coefficient.

The reflection coefficient can be obtained from  $S_{11}$  measured by the VNA. The SWR of our rectangular waveguide antenna is shown in Figure 3, for various microwave frequencies, with a water sample and various latex samples. It can be seen that at the frequencies from 2.2 to 2.3 GHz, the SWRs are among the lowest comparing to all samples, and for latex with DRC 60.63% the lowest SWR = 4.56 was obtained at 2.2 GHz.

#### 3.2 Relationships between attenuation and DRC in latex

The experiments covered the DRC range from 10.26% to 60.63% with latex samples obtained by diluting concentrated rubber latex with water. The 20 ml of latex was filled in the holder which makes the sample thickness of 4 mm, and the frequency range was 2.0-3.0 GHz, using the two rectangular waveguide antennae and the HP8510B vector network analyzer. The preliminary measurements are summarized in Table 1. The dependence of attenuation on DRC was fitted by linear regression with



Figure 3. SWR of the rectangular waveguide antenna, with various samples, for microwave frequencies from 2.0 GHz to 3.0 GHz.

Table 1. Summary of preliminary measurements.

Frequency (GHz)	Correlation		
	k <sub>0</sub>	$k_{1}$	R <sup>2</sup>
2.00	15.595	-0.1630	0.9651
2.10	16.183	-0.1456	0.9671
2.20	14.902	-0.1271	0.9793
2.30	14.231	-0.1366	0.9979
2.36	15.334	-0.1571	0.9996
2.40	16.250	-0.1647	0.9988
2.50	19.248	-0.1785	0.9899
2.60	21.539	-0.1620	0.9721
2.70	24.277	-0.1491	0.9628
2.80	26.992	-0.1342	0.9464
2.90	28.853	-0.1328	0.9633
3.00	33.143	-0.1146	0.9416

$$A = k_{\mu} D + k_{\mu} \tag{9}$$

where A is attenuation, D is DRC (%),  $k_1$  is slope, and  $k_0$  is intercept at 0% DRC. The fit parameters and the coefficients of determination R<sup>2</sup> are given in Table 1. The correlation was maximized experimentally, and the maximum was found at 2.36 GHz. At this frequency the relationship between DRC and attenuation was almost linear over the DRC range tested:

$$A = -0.1571D + 15.334 \tag{10}$$

Hence, the 2.36 GHz frequency was selected to estimate the DRC of latex, as shown in Figure 4. The calibration Equation 10 can be inverted to:

$$D = \frac{A - 15.334}{-0.1571} \tag{11}$$

# 3.4 Validation of new DRC estimates against the gravimetric standard method

Figure 5 shows the averaged DRC estimates from Equation 11, using measurements at 2.36 GHz, and the DRC values determined by the standard gravimetric oven-drying method, for 17 local rubber latex samples collected in the



Figure 4. Relationship of attenuation to DRC for latex calibration samples, at 2.36 GHz frequency.



Figure 5. New DRC estimates from microwave determinations were validated against the standard gravimetric method for 17 samples of natural rubber latex.

Songkhla Province, Thailand. The correlation coefficient between the two methods was 0.9047, and the new estimates in percent DRC had a mean error of 0.43. The errors are mainly attributed to the variations in the chemical composition of natural rubber latex, especially in liquids other than water, in the moisture content of air, and in the room temperature, among other possible factors.

#### 4. Conclusions

The standard gravimetric determination of dry rubber content (DRC) in collected natural rubber latex is too slow for practical field use at latex collection points. Alternative rapid and sufficiently accurate methods would be welcome. In this study we designed and implemented microwave transmission experiments using rectangular waveguide antennae made of aluminum, sandwiching a sample holder in a vertical stack. The microwave energy is guided to pass through the latex samples, and the design prevents electromagnetic interference from outside the waveguides. The temperature increment of the sample is neglected because of the low power and short exposure of the microwave radiation. The microwave attenuation by the samples was measured using a VNA, and the linearity of the relationship between attenuation and DRC was best at 2.36 GHz. The calibration samples were made by water diluting concentrated rubber latex so it had DRC from 10.26% to 60.63%, and a linear standard curve was fitted to estimate DRC from attenuation. Thus, calibrated microwave transmission technique was compared to the slow standard gravimetric oven-drying method, showing a good accuracy of the DRC estimates across samples of natural rubber latex. The mean error in %DRC was 0.43. The results demonstrate microwave attenuation as a promising approach to quickly estimate the DRC of natural rubber latex, and could support the development of novel commercial instruments for such measurements.

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# References

Abbas, Z. and Roger, D.P. 2001. Complex permittivity measurement at Ka-Band using rectangular dielectric waveguide. IEEE Transactions on Instrumentation and Measurement. 50, 1334-1342.

- Alex, R., Premalatha, C.K., Nair, R.B. and Kuriakose, B. 2003. Measurement of dry rubber content of fresh natural rubber latex by a titration method. Journal Rubber Research. 6, 221-230.
- Beatty, R.W. 1959. Magnified and Squared VSWR Responses for Microwave Reflection Coefficient Measurements. IRE Transactions. 7, 346-350.
- Chung, B.K. 2007. Dielectric constant measurement for thin material at microwave frequencies. Progress in Electromagnetics Research. 75, 239-252.
- Halit, E. 2006. Wireless sensors and instruments: networks, design, and applications. CRC Press, U.S.A.
- Hongliang, W. 2010. The indirect measurement for the water Ratio of Fabric. Basic Science Journal of Textile Universities. 2, 271-275.
- Julrat, S., Chongcheawchamnan, M. and Khaorapapong, T. 2012. Single-Frequency-Based Dry Rubber Content Determination Technique for In-Field Measurement Application. IEEE Sensors Journal. 12, 3019-3030.
- Khalid, K.B 1982. Determination of dry rubber content of hevea latex by microwave technique. Pertanika Journal of Science and Technology. 5, 192-195.
- Khalid, K.B. 1992. Microwave dielectric properties of hevea rubber latex. Proceeding of Asia-Pacific Microwave Conference, Adelaide, Australia, August 11-13, 1992, 611-616.
- Kim, K.B. and Kim, J.H. 2002. Measurement of grain moisture content using microwave attenuation at 10.5 GHz and moisture density. IEEE Transactions on Instrumentation and Measurement. 52, 72-77.
- Mohammadi, A., Khalid, K., Bafti, P.S. and Homaiee, M. 2011. Development of microcontroller-based microwave system to measure solid content of hevea rubber latex. Measurement Science Review. 45, 571-579.
- Nair, N.U., Thomas, M., Sreelatha, S., Simon, S.P., Vijaya Kumar, K.R. and George P.J. 1993. Clonal Variation in lipid composition in the latex of *Hevea brasiliensis* and its implications in latex production. Indian Journal Natural Rubber Research. 6, 143-145.
- Nutchanat, S. 2009. The Study for the development of the device for measuring of the dry rubber content in latex. M.Sc. Thesis, Prince of Songkla University, Thailand.
- Pengfei, L., Bo, W., Jiakun, L. and Luhua, N. 2010. Research on moisture content measurement of fabric based on microwave transmission. IEEE Computer Society. 3, 120-123.
- Rejikumar, R. and Philip, J. 2010. Estimation of dry rubber content in natural rubber latex by differential scanning calorimetry. Journal of Thermal Analysis and Calorimetry. 101, 1047-1050.
- Sadiku, M. 1995. Elements of Electromagnetics. 2<sup>nd</sup> edition. Oxford University Press, U.K.
- Somwong, T. and Seewattanapon, S. 2008. Application of microstrip ring resonator for correlation of dielectric and frequency in rubber latex. Proceedings of 31<sup>th</sup> Electrical Engineering Conference, Nakhon Nayok,

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Thailand, October 29-31, 2008, 769-772.

- Tillekeratne, L.M.K., Karunanayake, L., Kumara, P.H.S. and Weeraman, S. 1988. A rapid and accurate method for determining the dry rubber content and total solid content of NR latex. Polymer Testing. 5, 353-358.
- Trabelsi, S. and Nelson, S.O. 2003. Free-space measurement of dielectric properties of cereal grain and oilseed at

microwave frequencies. Measurement Science and Technology. 14, 589-600.

Zhao, Z.M., Jin, X.D., Zhang, L. and Yu, X.L. 2010. A novel measurement system for dry rubber content in concentrated natural latex based on annular photoelectric sensor. International Journal of Physical Sciences. 5, 251-260.