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EXPERIMENTAL DETERMINATION OF THE PERFORMANCE OF RICE HUSK-CARBON NANOTUBE COMPOSITES FOR ABSORBING MICROWAVE SIGNALS IN THE FREQUENCY RANGE OF 12.4–18 GHz

Yeng S. Lee[1], Fareq Malek[2], Ee M. Cheng[3], Wei-Wen Liu[4], Kok Y. You[5], Muhammad N. Iqbal[1], Fwen H. Wee[1], Shing F. Khor[2], Liyana Zahid[1], and Mohd F. B. H. Abd Malek[6]

[1]School of Computer and Communication Engineering, Universiti Malaysia Perlis (UniMAP), Pauh Putra Campus, Arau, Perlis 02600, Malaysia

[2]School of Electrical Systems Engineering, Universiti Malaysia Perlis (UniMAP), Pauh Putra Campus, Arau, Perlis 02600, Malaysia

[3]School of Mechatronic Engineering, Universiti Malaysia Perlis (UniMAP), Pauh Putra Campus, Arau, Perlis 02600, Malaysia

[4]Institute of Nano Electronic Engineering (INEE), Universiti Malaysia Perlis (UniMAP), Kangar, Perlis 01000, Malaysia

[5]Radio Communication Engineering Department (RaCED), Faculty of Electrical Engineering, Universiti Teknologi Malaysia, Skudai, Johor 81310, Malaysia

[6]MISC LNG Liaison Office Japan, MISC Berhad, Yokohama, Japan

Abstract—Composites of rice husks and carbon nanotubes (RHCNTs) are an innovation in improving the absorption of microwave signals. Rice husks, which are an agricultural waste material, have been found to possess a significant propensity for absorbing microwave signals. Studies have shown that both rice husks and carbon nanotubes (CNTs) have high percentages of carbon. Thus, in this paper, we present the results of our experimental study in which we varied the ratios of rice husks and CNTs in the composite materials and determined the dielectric properties of the composites and measured their abilities to absorb microwave signals. The experimental microwave

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* Corresponding author: Yeng Seng Lee (Leeyengseng@gmail.com).

absorber was fabricated using rice husks and CNTs, which increased the dielectric constant and the loss factor. Complex permittivity was measured using an Agilent dielectric probe. The RHCNT composites were investigated to determine their reflection loss and absorption performance as microwave absorbers. For the fabricated microwave absorber, we used the rectangular waveguide measurement technique to study reflection loss, transmission loss, and absorption performance in the frequency range of 12.4–18 GHz. Carbon has an essential role in the absorber due to its ability reflect/absorb microwave signals. Thus, we compared the abilities of a pure rice-husk (PRH) absorber and RHCNT composites absorbers to absorb microwave signals.

1. INTRODUCTION

Rice husks are one of the most readily-available agricultural wastes in Malaysia. The husks are rice-paddy byproducts, and they contain cellulose, hemicellulose, lignin, and ash (anon. 2008). Normally, rice husks from a paddy are burned, which creates ash at the paddy field and causes air pollution [1]. Such environmental pollution would be reduced if the rice husks were used in other applications rather than burned. Recently, several research projects involving rice husks have shown that the properties of rice husks make this waste material potentially useful as a microwave absorber and for other applications [2–6]. This is because 35–37% of the rice husks is carbon, which has the ability to attenuate/absorb microwave radiation [5]. Several researchers have stated that carbon has an important role in microwave absorption [2–6]. Figure 1 shows the physical look of rice husk and CNTs.

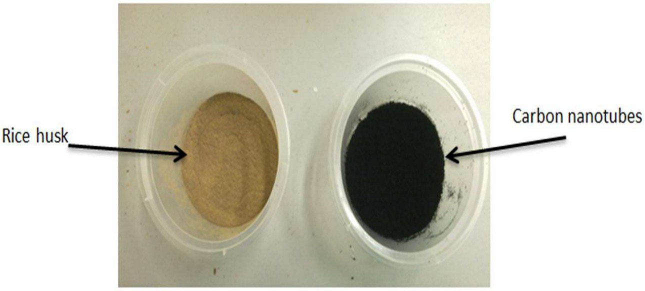


Figure 1. Rice husk and CNTs.

Carbon is a good semiconductor material that is very suitable for transforming microwave energy to thermal energy because microwaves are impeded as they pass through the carbon. When microwaves pass through the absorber, an electric field is produced at the surfaces of the absorbers. When this occurs, the electrical energy is transformed into thermal energy and is dissipated.

Recently, materials that can absorb microwaves have been made from carbon nanotubes (CNTs), and they have been investigated extensively because of their ability to absorb the energy from electromagnetic waves and convert it to heat so that it can be dissipated [7]. CNTs contain a high content of carbon, which is considered to be a lossy conductor material that can help absorb microwave energy [8–13]. In addition, several researchers have used CNT composites to develop materials that can be used as shields against electromagnetic radiation [14–18]. In CNTs, there is no signification magnetic loss contribution in absorption [19]. CNTs have been classified into two different categories, i.e., single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) [20]. The difference between SWCNTs and MWCNTs is SWCNTs show single-layer of graphene sheet whereas MWCNTs consist of multi-layer of graphene sheets [21]. CNTs also can be used in many of fields, including chemical, mechanical, and electrical applications, due to their excellent characteristic properties [22–25]. In terms of theory, the structure of MWCNTs is very complex and much more difficult to confirm that that of SWCNTs. Some of the specific properties of CNTs allow them to store or absorb considerable energy [26, 27]. The electromagnetic properties of pure CNTs are very difficult to measure directly because it is hard to form them in the required shape [7]. Therefore, CNTs must be combined with other materials to form composites in order to to measure their ability to absorb microwave radiation.

In this work, we were interested in studying the dielectric properties associated with various compositions and thicknesses of RHCNT composites and the effectiveness of the composites for absorbing microwave radiation. The dielectric properties were measured using Agilent Technologies 85070 software, which will be discussed in Section 4.1. The performances of different compositions of RHCNT composites for microwave absorbers were investigated.

2. THEORY

The dielectric properties of dielectric constant (ε') and dielectric loss factor (ε'') can be derived from transmission line theory, which can determine the strength of reflected/transmitted microwave signals from a sample material [28]. However, several measurement techniques are available for the determination of dielectric properties, including the coaxial line method and the waveguide method [29, 30].

When microwaves are propagated throughout a material, some of the energy is reflected, some is absorbed, and some is transmitted. This phenomenon can be defined in terms of the dielectric properties. The complex permittivity of material, e, is expressed as:

$$\varepsilon = \varepsilon' - j\varepsilon'', \tag{1}$$

where the real part, ε', is the dielectric constant, and the imaginary part, ε'', is the dielectric loss factor. The dielectric constant (ε') defines the ability of a dielectric material to store electromagnetic energy, whereas he dielectric loss factor (ε'') represents its ability to convert electromagnetic energy to heat (energy that can be dissipated) [31, 32]. The dielectric properties also determines the reflected energy from the surface of material, transmitted energy into material, and absorbed energy in the material.

The materials used in microwave absorbers are designed to attenuate/absorb microwave energy. Radar absorbing materials (RAMs) are designed to attenuate or absorb microwave energy with the absorbed energy being converted to heat. Attenuation of microwave energy occurs due to the dielectric loss and/or magnetic loss of a microwave absorber. Dielectric loss is found in the imaginary component of the complex permittivity and acts on the electric field (E). Magnetic loss is found in the imaginary component of the permeability and acts on the magnetic field (H). Normally, dielectric loss of microwave absorbers causes the absorption of the electric field portion of an electromagnetic wave, in which the synthesized absorber use carbon particles as a loading medium to create the proper complex permittivity. In most cases, microwave absorbers that use dielectric loss are electrically conductive.

Waveguides are the most efficient way to transfer electromagnetic energy. A propagating electromagnetic wave is reflected, S_{11}, and transmitted, S_{21}, by placing the material inside the waveguide. Technique to measure tnormal microwave materials using rectangular waveguide consider the properties of the microwave radiation, such as permeability and permittivity. The standard thru-reflect-line (TRL) calibration technique is used to avoid any unwanted losses/reflection in the inner wall of the waveguide and to achieve a zero reference plane for the measurements [33]. Waveguide measurement produces more accurate results and less radiation losses than free-space measurements, but it has frequency limitations. By using the rectangular waveguide method, the rectangular sample should fit into the inner dimension of the waveguide at the frequency being measured. Normally, the thickness of the sample should be less than a quarter of the wavelength for rectangular waveguide measurements. TRL calibration is needed for accurate measurements, particularly of measurements of reflected energy. Fitting the sample properly inside the waveguide is very important, because a poor fit will cause inaccurate results. Figure 2 shows the possible paths of an electromagnetic wave on a surface of material under testing (MUT) which the incident radiation will be reflected, absorbed, or transmitted.

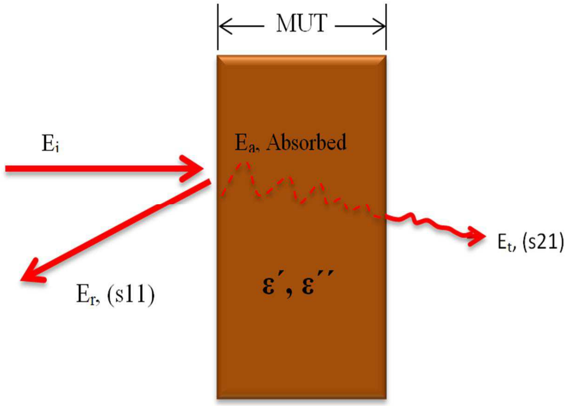


Figure 2. Diagram of electromagnetic wave path in an absorber material.

The MUT is placed between the waveguides, and a measurement is performed. The formula to calculate the absorption of the material is:

$$E_a = E_i - E_r - E_t \tag{2}$$

where E_a, E_i, E_r, and E_t are the incident, reflected, absorbed, and transmitted energy, respectively [34, 35].

In realworld applications, an ideal absorber should fully absorb the signal that interact with the absorber without fail, but this would never occur. The main properties that enable dielectric materials to act as microwave absorbers are the dielectric constant, ε', and the loss factor, ε''. Normally, researchers use a perfect electric conductor (PEC) such as a metal back plate to measure reflectivity of the microwave absorber [36–38]. In this research, the ratio of transmitted signal to incident signal, S_{21} was take into the account, because no metal back plate was placed behind the MUT.

3. METHODOLOGY

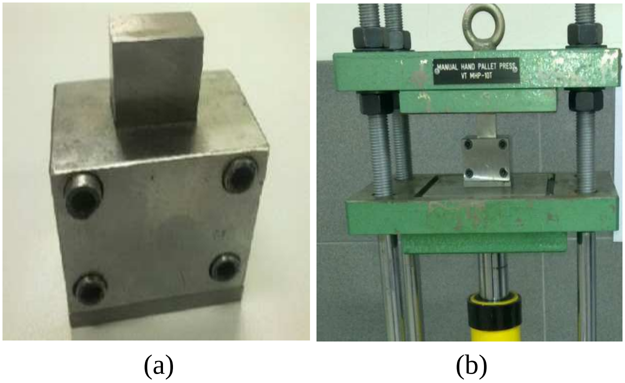
3.1. RHCNT Microwave Absorber Preparation

In this work, low-cost, natural materials, such as rice husk-CNT composites, were prepared by mixing MWCNTs, polyester and methyl ethyl ketone peroxide (MEKP). Figure 3 shows the mixture of the rice husks and the CNTs. The polyester bonding agent was used to bind the rice-husk and MWCNTs physically without forming any new bonding. The harding agent (MEKP) was used to harden the mixture and to facilitate the fabrication process. The dimensions of the rectangular RHCNTs are 2.6 × 1.5 mm. The resin: MEKP weight ratio was set at 5 : 1 of the total weight of the RHCNTs used for fabrication. The mixture was prepared in rectangular shape, which can be measured by using a WR-62 waveguide and an Agilent commercial dielectric probe in the frequency range from 12.4 to 18 GHz. Figure 4 shows the mould that was used to fabricate the microwave absorber and the position of mould mounted at the hydraulic press, respectively.

Figure 3. Mixture of rice husks and CNTs.

The hydraulic press was used to press the sample materials to form compacted materials. The materials had a rectangular shape. Two metal plates were placed at the top and bottom of the sample materials to protect them when they were being removed from the mould. After the rice husks and CNTs were mixed to form the RHCNT materials, the RHCNTs were placed into the mould. Then, the mould was placed and pressed for three minutes by a force of six tons at the hydraulic press. Finally, a rectangular shape of RHCNT composites microwave absorber was formed.

The rectangular RHCNT microwave absorber was measured using the Agilent dielectric probe. Then, the RHCNT microwave absorber was cut into a smaller size, i.e., 15.7 × 7.8 mm ± 0.5 mm. Finally,



(a) (b)

Figure 4. (a) The mould used to fabricate the microwave absorber. (b) The position of mould mounted at hydraulic press.

the RHCNT microwave absorber was placed into the waveguide and measured using a pair of WR-62 waveguides.

3.2. Dielectric Properties Measurement

The dielectric properties of the RHCNT microwave absorber were measured over the frequency range of 12.4 to 18 GHz using a commercial dielectric probe in conjuction with an Agilent E8362B P-series Network Analyzer (PNA) [39]. The dielectric probe with Agilent Technologies 85070 software was calibrated before measurement to avoid any systematic errors. Three calibrations were performed, i.e., air (open), short (metal), and water. Figure 5 shows the dielectric properties of RHCNT microwave absorber were measured using dielectric probe.

3.3. Reflection and Transmission Measurement

An Agilent PNA was used to measure the reflection loss, S_{11}, and the transmission loss, S_{21} of different sample materials placed in a waveguide sample holder. Two low-loss, coaxial cables were used to connect with the WR-62 waveguide adapters as the source (port 1) and the receiver (port 2). One and two ports were used to measure the reflection loss, S_{11}, and the transmission loss, S_{21}, of the sample materials, respectively. The measurement setup is shown in Figure 7. The TRL must be calibrated before any sample can be measured. After the calibration, the sample material was placed in the sample holder of waveguide and measured over the frequency range from 12.4

Figure 5. Dielectric probe was used to measure the dielectric properties of the RHCNT microwave absorber.

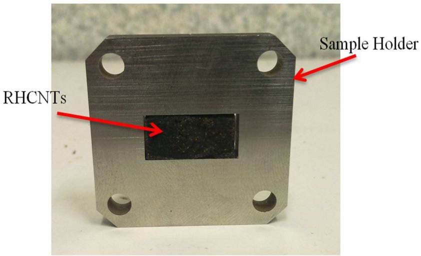


Figure 6. Sample holder and RCHNT microwave absorber.

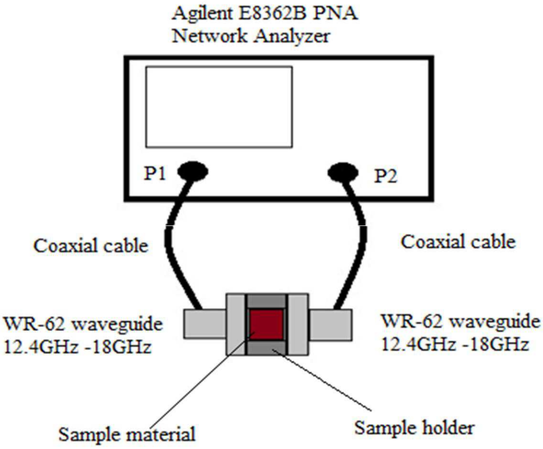


Figure 7. Measurement setup diagram.

to 18 GHz. Figure 6 shows the RHCNT microwave absorber load into sample holder.

Two waveguide ports, i.e., port 1 and port 2, were used as transmitting and receiving ports, respectively. A pair of standard, rectangular, WR-62 waveguides were connected to an Agilent PNA network analyzer with coaxial cable. A waveguide sample holder was used to hold the sample in the correct place. Different thicknesses of rice husks were tested with different quantities of CNTs added to determine reflection loss, S_{11}, and transmission loss, S_{21}, as shown in Figures 9(a), 9(b), 10(a), and 10(b).

The mixtures of RH and CNTs in various ratio were investigated to determine their complex permittivity, and their abilities to absorb microwave radiation. Sample with thicknesses of 1, 2, 3, 4, and 5 mm were investigated in the frequency range from 12.4 to 18 GHz.

4. RESULTS AND DISCUSSION

The dielectric properties (real part, ε', and imaginary part, ε'') of the material are important parameters that must be of concern when modeling a microwave absorber. This is because the dielectric properties define the ability of the material to store electromagnetic energy, convert it to heat, and then dissipate the heat. The results of the dielectric properties presented in this paper are the average values of the five measurements that were made for each sample. Tables 1 and 2 show the comparison of the dielectric properties of PRH and different rice husk: CNTs weight ratio for frequencies range from 12 to 18 GHz. The data in Tables 1 and 2 show that the dielectric constants and loss factors tend to increase with the increasing of the quantity of CNTs in the RHCNT composites.

Table 1. Dielectric constants, ε', for PRH and different RHCNTs over the frequency range of 12 to 18 GHz.

Material	Frequency (GHz)						
	12	13	14	15	16	17	18
PRH	2.409	2.4180	2.416	2.372	2.370	2.412	2.405
Rice husks + 1% CNTs	2.799	2.8214	2.830	2.777	2.776	2.821	2.801
Rice husks + 2% CNTs	2.887	2.9064	2.906	2.844	2.834	2.872	2.850
Rice husks + 3% CNTs	3.345	3.3773	3.384	3.317	3.321	3.381	3.346
Rice husks + 4% CNTs	3.787	3.8191	3.812	3.715	3.708	3.778	3.750
Rice husks + 5% CNTs	3.969	4.0023	4.011	3.924	3.925	3.995	3.950

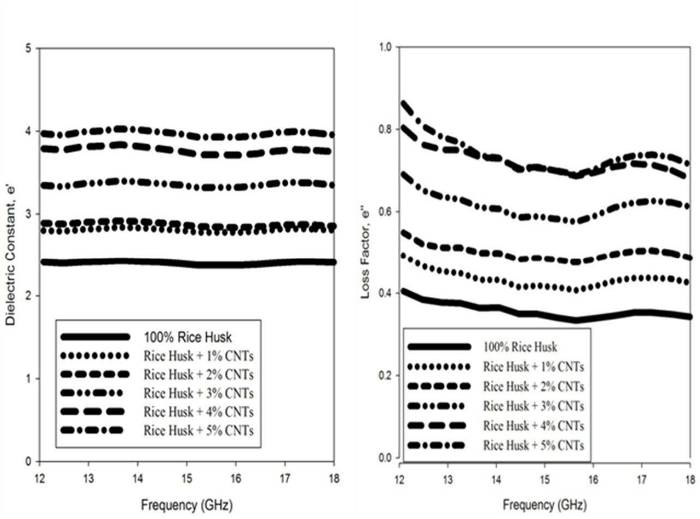
Table 2. Loss factor, ε'', for PRH and different RHCNTs over the frequency range of 12 to 18 GHz.

Material	Frequency (GHz)						
	12	13	14	15	16	17	18
PRH	0.405	0.375	0.364	0.340	0.3389	0.353	0.341
Rice husks + 1% CNTs	0.492	0.448	0.432	0.413	0.416	0.436	0.424
Rice husks + 2% CNTs	0.549	0.511	0.4981	0.482	0.484	0.505	0.486
Rice husks + 3% CNTs	0.690	0.629	0.606	0.582	0.590	0.625	0.610
Rice husks + 4% CNTs	0.804	0.749	0.728	0.698	0.695	0.713	0.678
Rice husks + 5% CNTs	0.863	0.766	0.730	0.700	0.703	0.738	0.713

The measured dielectric properties of the RHCNT microwave absorber are shown in Figures 8(a) and 8(b). The dielectric constant and the loss factor of PRH and RHCNTs vary depending on the frequency. Figure 8(b) shows that the loss factors of PRH and RHCNTs decreased as the frequency increased. For example, the loss factor for RHCNTs with 5% CNTs was 0.863 at 12 GHz, whereas it was 0.713 at a frequency of 18 GHz. If carbon loading is high, then the conductivity of material will increases [40]. The loss factor, ε'' measure of material's dissipation or loss of energy, the relation of conductivity (σ) and loss factor (ε'') are shown in Equation (3) :

$$\varepsilon'' = \frac{\sigma}{2\pi f} \tag{3}$$

where the f is the frequency. The loss factor has values that generally less than the dielectric constant value [41]. According to Equation (3), loss factor is proportional to conductivity. By increasing the conductivity of material, the loss factor value will be increased [41]. Hence, the dielectric constant value will also increase, because the dielectric constant value are always larger than loss factor.



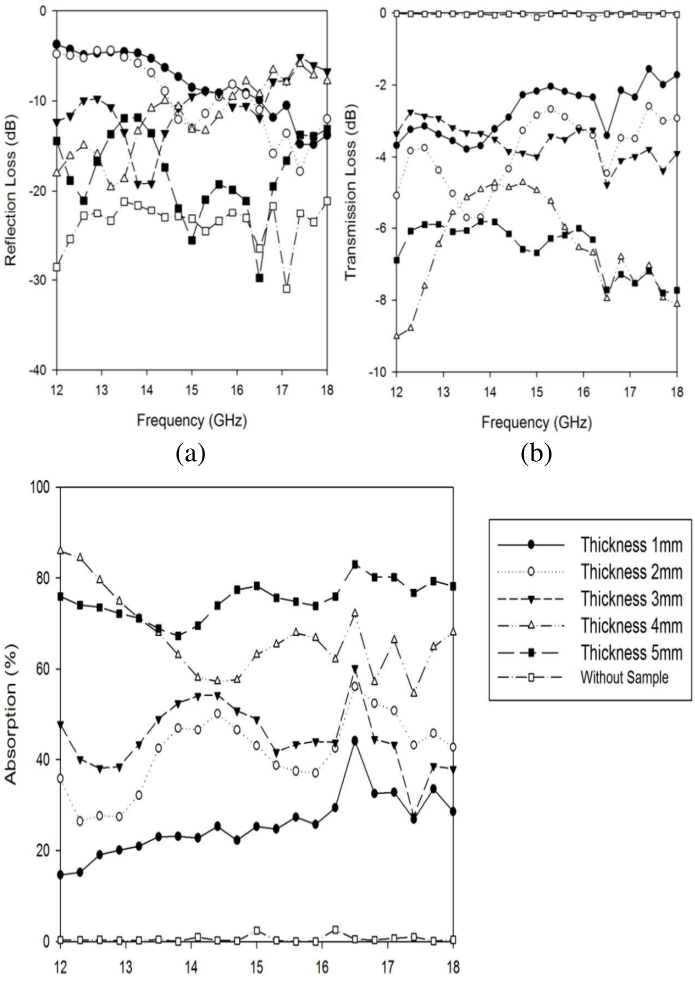
(a) (b)

Figure 8. (a) Dielectric constant, ε', and (b) loss factor, ε'', values measured with an Agilent technologies dielectric probe.

Table 3. Average absorption percentage point of different thicknesses of a PRH microwave absorber.

Thickness of PRH Material (mm)	Average absorption, %
1	25.77
2	41.61
3	45.25
4	66.49
5	75.21

In Table 3 shows the average absorption percentage point of different thicknesses of a PRH microwave absorber. From Figure 9(c), we can see clearly that the absorption ability increased as the thickness of the PRH microwave absorber increased. The PRH absorber with a thickness of 5 mm had the highest percentage absorption, whereas the 1-mm-thick PRH absorber had the lowest percentage absorption. If a microwave absorber is very thin, electromagnetic waves can propagate through the absorber, and very little of the signal will be trapped. Hence, more signal will be absorbed when the thickness of the microwave absorber is increased, as shown in Figure 9(c). Therefore, a lesser signal will be received at the receiver (transmission loss, S_{21}), as shown in Figure 9(b).

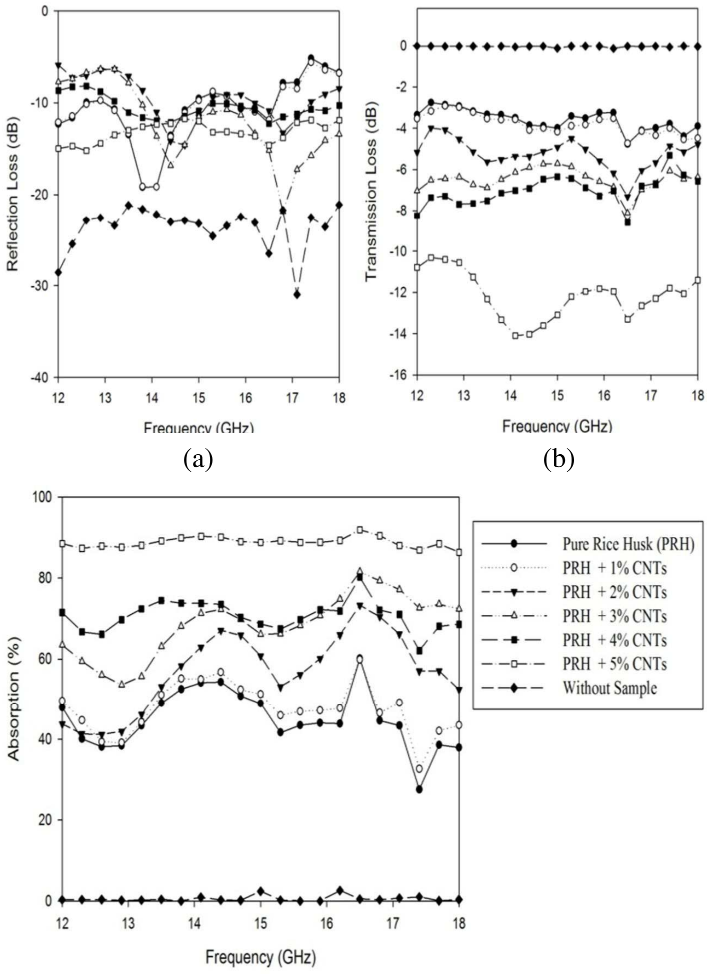


(a) (b)

(c)

Figure 9. (a) Reflection loss, (b) transmission loss, (c) absorption ability of different thicknesses of PRH microwave absorbers.

Figures 10(a) and (b) show the reflection loss of the RHCNT microwave absorber's performance by increasing the quantity of CNTs in the RHCNT microwave absorbers. When the quantity of CNTs increase, lesser signals were received by the receiver (transmission loss, S_{21}). If the receiver receives a lesser signal, it might due to either large amount of reflected signal from absorber or transmitted



(a) (b)

(c)

Figure 10. (a) Reflection loss, (b) transmission loss, (c) absorption ability of different different percentages of CNTs in RHCNTs.

signal through absorber occurred during the interaction of signal with absorber Equation (2) was used to calculate the absorptive capability of the RHCNT microwave absorbers. In Table 4 shows the average absorption percentages of different RHCNT microwave absorbers.

Figure 10(c) shows the ability of the RHCNT microwave absorbers to absorb microwave signals. Without a sample (as reference), the absorption was essentially 0%. The RHCNT microwave absorber with 5% CNTs had the average absorption 88.9%, while the PRH microwave

Table 4. Average absorption percentages of different RHCNT microwave absorbers.

Material	Average absorption, %
PRH	45.2
PRH + 1% CNTs	47.2
PRH + 2% CNTs	57.2
PRH + 3% CNTs	68.4
PRH + 4% CNTs	70.6
PRH + 5% CNTs	88.9

absorber had the average absorption 45.2% over the frequency range of 12 to 18 GHz. When only 1% of CNTs was added to the rice husks, the ability absorption was essentially the same as the PRH absorber. However, as the quantity of CNTs that was added to the rice husks was increased, the absorption increased, as shown in Figure 10(c). When the quantity of CNTs added was increased, the dielectric properties also increased, as shown in Figures 8(a) and (b). When the dielectric constant and loss factor of the absorbing material increase, the ability of the RHCNT microwave absorbers to store and dissipate energy increased too. In other word, the RHCNT microwave absorbers could absorb more microwave signal for absorber that contain higher percentage of CNTs.

5. CONCLUSIONS

Rice husks were mixed with different percentages of CNTs to fabricate RHCNT composite materials that were investigated to determine their dielectric properties and absorbability if microwaves. The waveguide-measurement technique was used to measure the reflected, transmitted, and absorbed fractions of the microwaves by the RHCNT absorbers. In addition, PRH microwave absorbers with different thicknesses were investigated to determine their absorption performances. The results of this study proved that the RHCNT composite microwave absorbers have greater potential than PRH microwave absorber. Normally, agricultural waste is burned, which will pollute the environment. This paper propose an alternative to reuse agricultural waste through technological advancements, in order to reduce environmental pollution, especially air pollution. Generally, carbon is main constituent that contributes to the absorption of electromagnetic waves. Therefore, CNTs (which are high in carbon content) were chosen to mix with RH, in order to increase the quantity of carbon. The experimental results indicated that the dielectric properties of RHCNT absorbers were greater than PRH absorbers in the frequency range of 12.4 to 18 GHz. Thicker PRH samples had a higher ability to absorb microwaves than thinner samples. It was also shown that the absorption of RHCNT absorbers with 5% CNTs in the RH exhibit the best absorbability 88.9% of absorption from 12.4 to 18 GHz. In the future, this work could be extended by mixing the carbon nanotubes with another material with little carbon to see how much the carbon nano tubes are the main source of the absorption versus the rice husks.

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