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The Investigation on the Potential of Coconut Shell Powder Composite in Term of Carbon Composition, Surface Porosity and Dielectric Properties as a Microwave Absorbing Material

Yew Been Seok[a], Siti Nurbazilah[a], Wee Fwen Hoon[b], Saiful Bahri Bin Mohamed[a], Ahmad Humaizi[c], M. Rizon. M. Juhari[a] and W Noor Fatihah W Mohamad[a]

[a]*Faculty of Innovative Design and Technology, Universiti Sultan Zainal Abidin (UniSZA), Kampus Gong Badak, 21300 Kuala Terengganu, Terengganu, Malaysia.*
[b]*Embedded Computing Research Cluster, School of Computer and Communication Engineering, Universiti Malaysia Perlis (UniMAP), JalanKangar-Alor Star, Taman Pertiwi Indah Seriab, 01000 Kangar, Perlis, Malaysia.*
[c]*School of Manufacturing Engineering, Universiti Malaysia Perlis (UniMAP), Kampus Pauh Putra, 02600 Arau, Perlis, Malaysia.*

Abstract

Agricultural wastes are renewable resources that are potentially useful as microwave absorbing materials. This paper presents the investigation on the carbon composition, surface porosity of the raw coconut shell powder particles and the dielectric properties of coconut shell powder with epoxy resin matrix composites. From CHNS elemental analysis, it was found that the carbon composition of coconut shell powder is 46.700%. Presences of macropores (≈ 2μm) were detected in the SEM analysis of the coconut shell powder particles. Measurement on dielectric properties of the coconut shell powder composites was performed by using open-ended coaxial probe method over microwave frequency range of 1-8 GHz. The overall dielectric constant (ε_r') and dielectric loss factor (ε_r'') of the composite with ratio 50:50 were 3.56 and 0.26, ranging from 3.35-3.76 and 0.21-0.30 respectively; whereas for composite ratio 40:60, the overall dielectric constant (ε_r') and dielectric loss factor (ε_r'') were 2.97 and 0.21, ranging from 2.74-3.17 and 0.16-0.27 respectively. The electrical conductivity calculated based on measured ε_r'' was 0.067 and 0.054 for composite ratio 50:50 and 40:60 respectively. The dielectric properties and electrical conductivity of the coconut shell powder composites were influenced by the greater presence of high dielectric material (coconut shell powder). This experimental investigation on the potential of the coconut shell powder with epoxy resin composites indicates that the ability of the composite to absorb and convert microwave signals is dependent on the carbonaceous materials of the composite. This result offers a great opportunity to diversify the use of coconut shell powder as microwave absorbing material.

Keywords: coconut shell powder; microwave absorbing material; carbon composition; macropores; dielectric properties

1. Introduction

Malaysia is located in a region with fertile agricultural land where it produces high productivity in agricultural industry. It was estimated that more than 2 million tons of agricultural wastes are produced annually (Wan *et al.,* 2010; Abdullah *et al.,* 2011; Sulaiman *et al.,* 2013). This serves a great opportunity for harnessing agricultural waste by-products in an eco-friendly and commercially-viable manner. Utilization of agricultural wastes helps to minimize environmental menace such as emission of methane and leachate from rotten waste agricultural biomass and open burning that generates carbon dioxide. In recent, researchers have focused to identify the agriculture wastes as a new microwave absorbing material (Nornikman *et al.,* 2010; Abdullah *et al.,* 2012; Zahid *et al.,* 2013). The agricultural wastes include rice husk wastes, coconut wastes, oil palm wastes, dried banana leaves and sugarcane bagasse. The results obtained from recent research projects show that the agricultural wastes are potentially useful as a microwave absorbing material.

In this paper, we investigate the potential of coconut shell powder in terms of carbon composition, surface porosity and dielectric properties to be used as microwave absorbing material. In section 2, we present the materials and methods that were used in the experimental investigation. Section 3 presents the result and discussion of the experimental determination on the carbon composition, SEM analysis of the raw coconut shell powder, fabrication and dielectric properties measurement of coconut shell powder composites. A final conclusion is drawn in Section 4.

2. Materials and Methods

2.1. Important features of microwave absorbing material

Microwave absorption is the irreversible conversion of microwave energy into heat energy. Microwave absorbers are materials that attenuate the energy in an electromagnetic wave and are very useful for suppressing electromagnetic interference (EMI) (Tong, 2009). It is also used to recreate a free space environment by eliminating reflections in an anechoic chamber. The microwave absorbers can also be used as radar absorbing materials (RAM) in military and stealth technology. RAM is used to attenuate return radar signal, to disguise structures from radar detection and to minimize the levels of electromagnetic energy emitted by electric and electronic devices. Most radars used for air-traffic control and air-defense systems operate in the microwave band (L, S, C, X and Ku bands), whereas radars operating in the UHF, L, S and C bands are used to monitor the airspace over large distances (Folgueras *et al.,* 2014). In the next subsection, we discuss on the two important characteristics of a material to be used as microwave absorbing material.

2.1.1. The dielectric properties

The main characteristic that enables a material to absorb microwave energy is based on its dielectric properties. The dielectric properties *(ε_r)* of materials are derived from transmission line theoryand are expressed as complex permittivity shown in equation 1 (Datta *et al.,* 2005; Saini and Arora, 2012):

$$\varepsilon_r = \varepsilon_r' + j\varepsilon_r'' \qquad (1)$$

The real part of the permittivity is called the dielectric constant *(ε_r')* whereas the imaginary part of the permittivity is called the dielectric loss factor *(ε_r'')*. The ability of a material to act as a microwave absorber depends on the dielectric constant and dielectric loss factor. The dielectric constant defines the ability of a material to store the microwave energy while the dielectric loss factor defines the ability of a material to convert and dissipate the stored microwave energy to heat. The dielectric loss factor is always greater than zero and is usually much smaller than the dielectric constant. In other words, materials that possess higher thermal conductivity are preferable as microwave absorbing materials as the ability of these materials to dissipate heat increase as the thermal conductivity increases.

2.1.2. Carbon composition

Carbon is the main element to absorb unwanted microwave energy (Menéndez *et al.,* 2010; Malek *et al.,* 2011; Lee et al., 2013; Iqbal *et al*., 2014). Carbon materials are a very good absorbent of microwaves due to its high thermal conductivity and it is easily heated by microwave energy. The carbon based microwave absorbers absorb the microwave energies that propagate through it, covert and dissipate those energies as heat. When microwave energies propagate through a material, electric field will be induced and this electrical energy is transformed into thermal energy (Zahid *et al.,* 2013). The electrical conductivity (σ) of a dielectric material can be evaluated based on the dielectric loss factor *(ε_r'')*. Refer to equation 2 and 3 (Micheli *et al.,* 2011).

The dielectric loss factor is presented as

$$\varepsilon_r'' = \frac{\sigma}{\omega\varepsilon_o} = \frac{\sigma}{2\pi f\varepsilon_o} \qquad (2)$$

Rearrange equation (2)

$$\sigma = 2\pi f\varepsilon_o\varepsilon_r'' \qquad (3)$$

Where σ is the electrical conductivity (S/m),ε_o is the free space permittivity (8.854 x 10^{-12} F/m), f is the frequency (Hz), ε_r'' is the dielectric loss factor. The dielectric properties can be determined through open-ended coaxial probe method by using high temperature dielectric probe connected to network analyzer with Agilent 85070E measurement software.

2.2. Based material-raw coconut shells

Commonly available raw coconut shells in Gong Badak region, Kuala Terengganu, Malaysia were selected as the based material. The raw coconut shells were cleaned and well dried before sanded into fine powder form based material. Fig. 1 shows the preparation of the based material. Fine powder form materials are preferred to enhance the surface area of the material.

2.3. Carbon, hydrogen, nitrogen, sulphur (CHNS) elemental analysis

The elemental composition of the raw coconut shell powder is determined through elemental (ultimate) analysis by using CHNS Elemental Analyzer. The elemental analysis determines the elemental composition of the coconut shell powder including carbon, hydrogen, nitrogen, sulfur. The elemental composition is expressed as a percentage of the total mass of the coconut shell powder sample. When sum of these compositions is subtracted from 100, it gives oxygen composition.

(a) (b) (c)

Figure 1. Preparation of the based material (a) Cleaned and well dried coconut shell, (b) Sanding process and (c) Coconut shell powder

2.4. Scanning electron microscopy (SEM) analysis

Samples for the SEM analysis used in this study was prepared by placing the fine coconut shell powder on an adhesive tape and mounting the sample on specimen holder with the help of the conductive silver adhesive. The specimen surfaces were coated with a thin film of platinum, placed in the sample holder and viewed with the Hitachi TM3000Tabletop microscope with an acceleration voltage of 15kV. Fig.2 presents the preparations for SEM analysis.

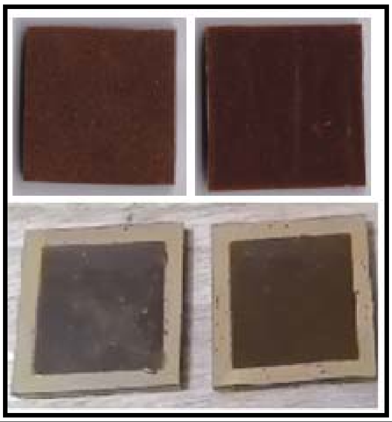
2.5. Fabrication of coconut shell powder composite

The fabrication of the planar shape coconut shell powder composite can be done based on the steps presented in Table 1. The raw coconut shell powder is mixed with the binder agent. Epoxy resin is used as the binder agent to bind the materials whereas Amine hardener agent is used to facilitate the curing process. In this work, the ratio of 40:60 and 50:50 of the raw coconut shell powder to the binder agent were used. The purpose of using two different ratios is to investigate

(a) (b)

Figure 2. (a) Coating of coconut shell powder sample with platinum (JFC 1600 Auto Fine Coater), (b) Placement of the sample on the specimen holder (Hitachi TM3000 Tabletop microscope)

Table 1. Fabrication of coconut shell powder composite

Step 1: Preparation of based material	Step 2: Mixing of based material and binder agent	Step 3: Composite curing
The cleaned and dried raw coconut shell is sanded into fine powder form.	Mixing of coconut shell powder and binder agent (Epoxy resin and hardener)	Composite curing at room temperature for approximately 48 hours
		

the behavior of dielectric properties of the coconut shell powder composite. The mixture of the coconut shell powder and binder agent is then poured into planar shape mold and let the mixture cure at room temperature for approximately 48 hours. Once the composite is completely cured, the measurement of the dielectric properties can be performed.

2.6. Dielectric properties measurement using open-ended coaxial probe method

The apparatus used for the dielectric properties measurement using open-ended coaxial probe method include the Agilent high temperature dielectric probe and 85070E measurement software, Agilent E8362B PNA series network analyzer and coaxial cables. A “three standard” calibration, namely open, short, water is performed at the end of the dielectric probe before the measurement process. Details on the measurement apparatus can be obtained by referring to the Agilent technical and application notes. Fig. 3 presents the measurement apparatus and setup. In this work, the dielectric properties of the fabricated composite were measured over the frequency range of 1-8 GHz, which represents L, S and C bands.

Measurement is made by simply bring the end of the coaxial probe into the contact with the composite surface. The coaxial probe senses the signals that are reflected from the sample and sends those signals back to the network analyzer to compute the dielectric properties. It is crucial to ensure that the composite surface is as flat as the surface of the coaxial probe in order to minimize the fringing effect. Fringing effect is the phenomenon that is caused by the air gap between the coaxial probe and the non-uniform composite surfaces. Air gap between the probe and sample can be a significant source of error that affects the measurement accuracy. The reflected signals from the sample, particularly small wavelength signals tend to escape through the air gaps that are formed from the non-uniform composite surfaces.

3. Results and Discussion

In order to analyze the potential of coconut shell powder to be used as microwave absorbing materials, the carbon composition and surface porosity of raw coconut shell powder were investigated and presented in the next subsections.

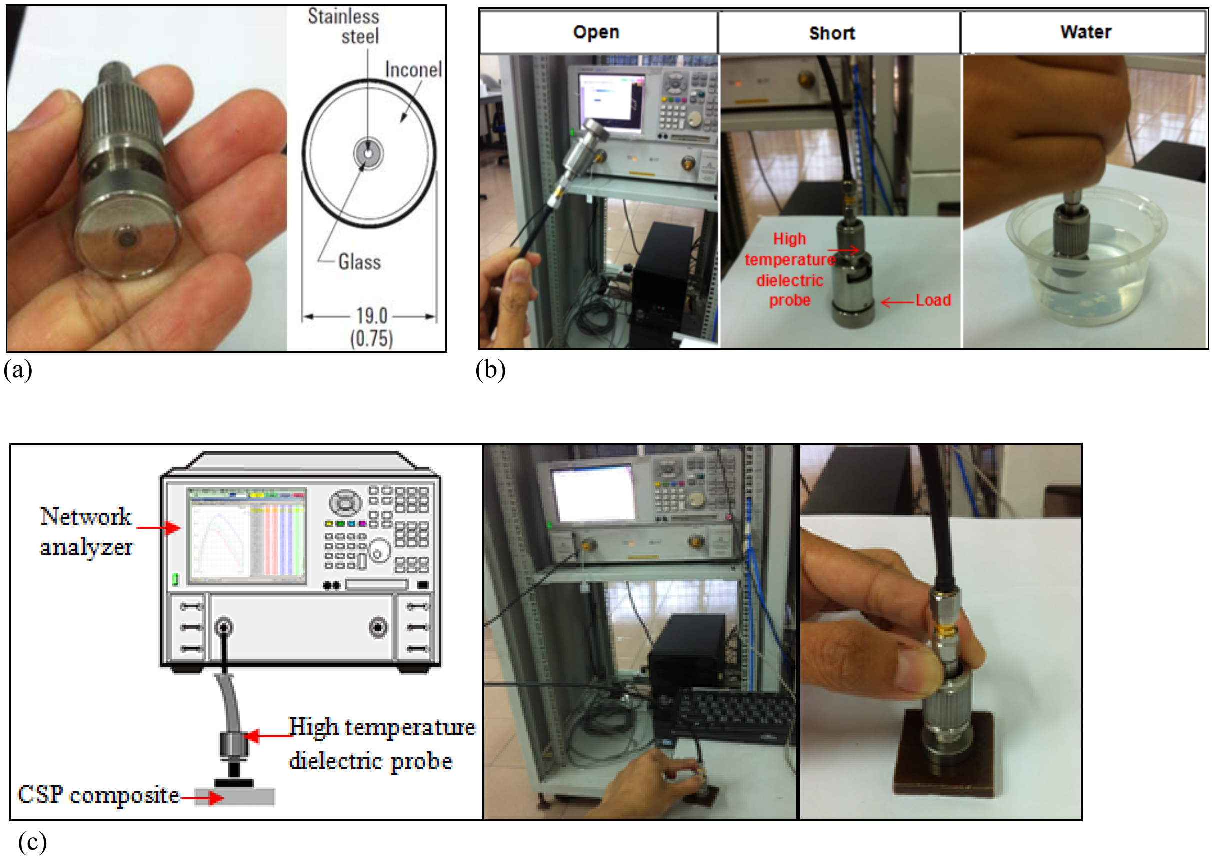


Figure 3. Measurement apparatus (a) High temperature dielectric probe, (b) Three standard calibration and (c) Dielectric properties measurement setup by using open-ended coaxial probe method

3.1. Elemental composition of raw coconut shell powder (CSP)

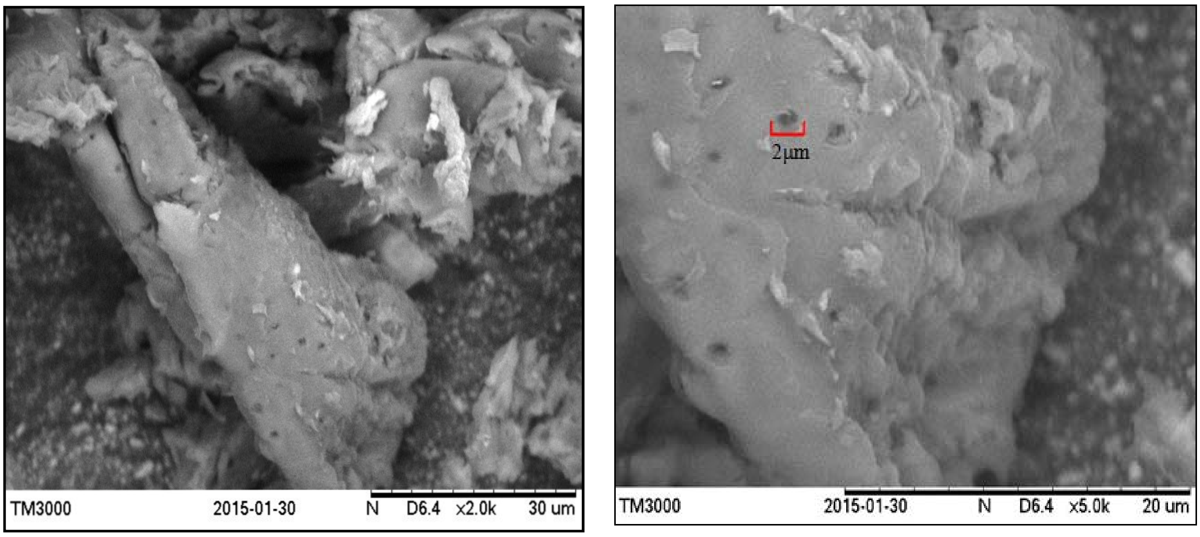
The elemental analysis of raw coconut shell powder is presented in Table 2. From the analysis, it was found that carbon is the most dominant solid element in the raw coconut shell powder, which is 46.700 %. As carbon is the main element in microwave absorbing materials, the elemental analysis proves that the raw coconut shell powder is potentially useful to be used as microwave absorber.

3.2. SEM analysis of raw coconut shell powder (CSP)

Fig.4 (a) and 4 (b) present the SEM analysis of the coconut shell powder with magnification at 2.0kX and 5.0kX respectively. Presences of macropores (approximately 2μm) were detected in coconut shell powder particles. According to IUPAC nomenclature (McCusker *et al.,* 2001; Langner *et al.,* 2011), macropores are those pores with pore widths greater than 50 nm. Materials with minimized pores size are preferable to be used as a microwave absorbing materials they possess larger surface area. Larger surface area enhances the thermal conduction losses during the conversion and dissipation of microwave energies to heat (Liu *et al.,* 2011; Che *et al.,* 2015).

Table 2. Elemental analysis of the raw coconut shell powder

Sample	Elemental Analysis (wt %)				
	Carbon (C)	Hydrogen (H)	Nitrogen (N)	Sulfur (S)	Oxygen (by difference)
Coconut shell powder	46.700	3.174	0.171	1.036	48.919



(a) (b)

Figure 4. (a) SEM image at 2.0 k X magnification, (b) SEM image at 5.0 k X magnification

3.3. Dielectric properties measurement of coconut shell powder composite

The sample of dielectric properties measurement data that is computed by Agilent 85070E measurement software is presented in Fig. 5. The measurement data is viewed in complex permittivity format (ε_r', ε_r"). The dielectric properties of the coconut shell powder composite with respect to the ratio are presented in Table 3.

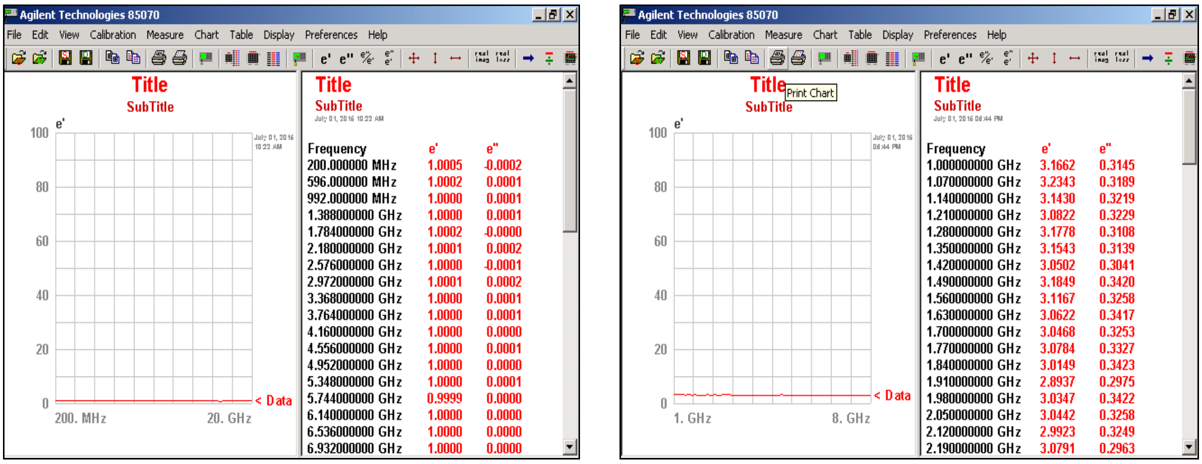
The frequency spectrum of dielectric properties over 1-8 GHz is plotted in Fig.6. It can be observed that the dielectric constant, ε_r' (real part of the permittivity) of both composites are greater that of air (ε'_{air} =1.00). This indicates that the composites are able to be polarized to a greater extent than air. The dielectric properties of the composites fluctuate and decrease as the frequency increases. These fluctuations were due to reflections at the interface of the non-uniform nature of the fabricated samples and the coaxial probe as frequency increases (Iqbal *et al.,* 2014).

The dielectric properties of a material are determined by its molecular structure. As the molecular structure changes, the dielectric properties changes respectively. For composite ratio of 50:50, the overall dielectric constant (ε_r') and dielectric loss factor (ε_r") is 3.56 and 0.26, ranging from 3.35-3.76 and 0.21-0.30 respectively. Whereas for composite ratio of 40:60, the overall dielectric constant (ε_r') and dielectric loss factor (ε_r") is 2.97 and 0.21, ranging from 2.74-3.17 and 0.16-0.27 respectively. The dielectric properties of the coconut shell composite with ratio of 50:50 were greater than that of ratio 40:60. Composite with higher composition of coconut shell powder possess greater dielectric properties due to the presence of higher carbonaceous (dielectric) material.

The frequency spectrum of electric conductivity over 1-8 GHz is plotted in Fig. 7. It can be observed that the electric conductivity of both composites is directly proportional to the frequency. The electric conductivity for the entire frequency range of composite ratio 50:50 is greater than that of composite ratio 40:60. Composite

Table 3. The dielectric properties and conductivity of the coconut shell powder composites

Composite	Range of dielectric properties over 1-8 GHz		Overall dielectric properties over 1-8 GHz		Conductivity (S/m)
Ratio of coconut shell powder to binder agent	Dielectric constant (ε_r')	Dielectric loss factor (ε_r")	Dielectric constant (ε_r')	Dielectric loss factor (ε_r")	
40:60	2.74-3.17	0.16-0.27	2.97	0.21	0.054
50:50	3.35-3.76	0.21-0.30	3.56	0.26	0.067



(a) (b)

Figure 5. Sample of dielectric properties measurement data (a) Permittivity of air,ε_{air} = 1.00, (b) Permittivity of CSP composite at frequency 1-8 GHz.

ratio 50:50 exhibits greater electric conductivity as the presence of carbonaceous materials composition is higher. The average electric conductivity over 1-8 GHz is 0.054 S/m and 0.067 S/m respectively for composite ratio 40:60 and composite ratio 50:50.

4. Conclusions

This study offers an alternative for microwave application seeking for low cost and organic based microwave absorber materials. The results of this experimental investigation led us to conclude that the coconut shell powder composites are potentially useful as microwave absorbing materials. Coconut shells are carbonaceous materials with 46.700% of carbon composition and possess macropores (2μm) particles structure. The dielectric properties of the coconut shell powder composites are greater than that of air, which shows the ability of the composite to be polarized to a greater extent of air. It was found that the dielectric constant *(ε_r')* and dielectric loss factor *(ε_r")* of the coconut shell powder composite with ratio 50:50 was in the range of 3.35-3.76 and 0.21-0.30 respectively, with the overall value of ε_r'=3.56 and ε_r" = 0.26. Whereas for composite ratio 40:60, the dielectric constant *(ε_r')* and dielectric loss factor *(ε_r")* was in the range of 2.74-3.17

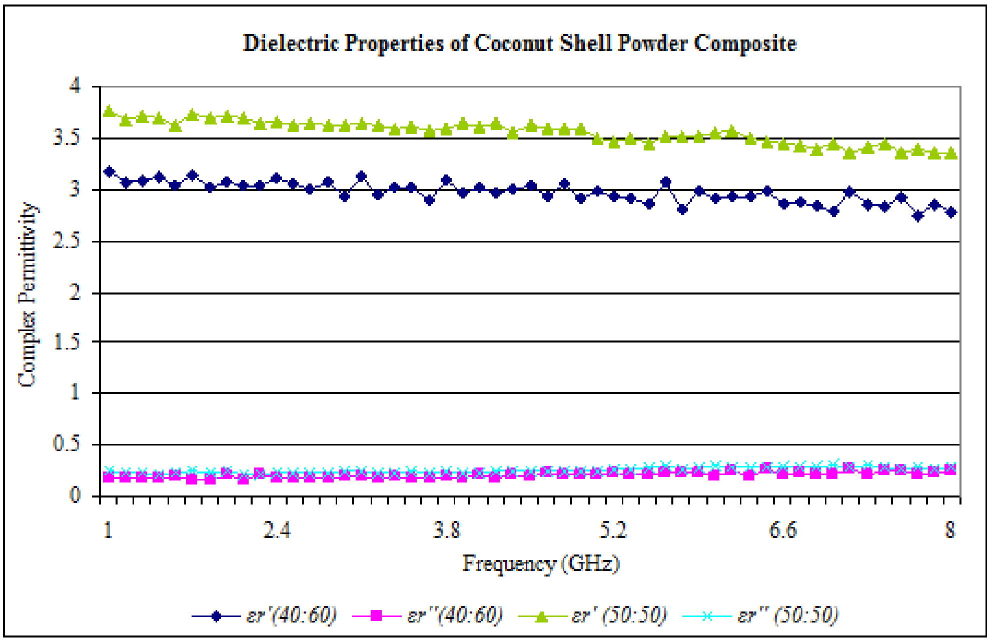


Figure 6. Dielectric properties of coconut shell powder composites

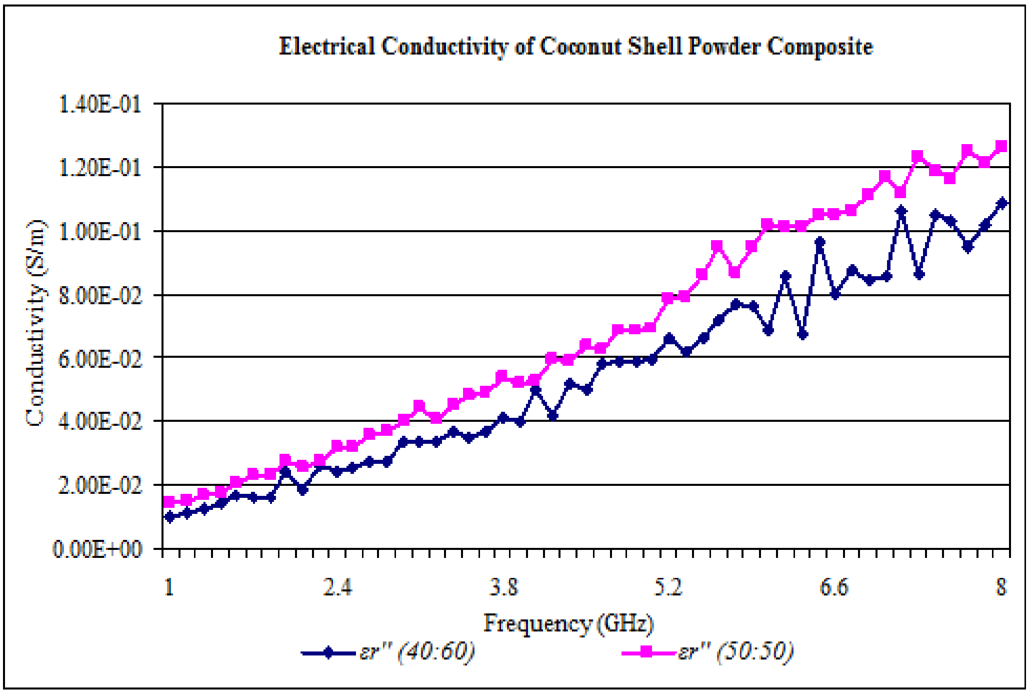


Figure 7. Conductivity of coconut shell powder composites

and 0.16-0.27 respectively, with the overall value of ε_r'=2.97 and ε_r''=0.21. Based on the measured ε_r'', the calculated electrical conductivity of the composite ratio 50:50 and 40:60 was 0.067 S/m and 0.054 S/m respectively. Materials with greater dielectric properties and conductivity are preferable to absorb and convert unwanted microwave signals into heat and this can be achieved by enhancing the amount of carbonaceous materials in the microwave absorber/composite. However, in order to determine the microwave absorption ability of the composite, the coconut shell powder composite must be further fabricated into specific shape and size composite for various microwave absorption application.

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Correspondence to
Dr. Yew Been Seok
Faculty of Innovative Design and Technology,
Universiti Sultan Zainal Abidin (UniSZA),
Kampus Gong Badak,
21300 Kuala Terengganu,
Terengganu,
Malaysia
Tel: + 60 9668 8407
E-mail: bseokyew@unisza.edu.my; bseokyew@gmail.com