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THE EFFECT OF SURFACE TEXTURE AND CARBONACEOUS MATERIAL COMPOSITION ON THE DIELECTRIC PROPERTIES MEASUREMENT OF COCONUT SHELL-POLYMER (CSP) COMPOSITES

Yew Been Seok[1], Siti Nurbazilah[1], Wee Fwen Hoon[2], Saiful Bahri[1], Ahmad Humaizi[3] and WN Fatihah[1]
[1]Faculty of Innovation Design and Technology, Universiti Sultan Zainal Abidin, Kampus Gong Badak, Kuala Terengganu, Terengganu, Malaysia
[2]Embedded Computing Research Cluster, School of Computer and Communication Engineering, Universiti Malaysia Perlis, Jalan Kangar-Alor Star, Taman Pertiwi Indah Seriab, Kangar, Perlis, Malaysia
[3]School of Manufacturing Engineering, Kampus Pauh Putra, Arau, Perlis, Malaysia
E-Mail: bseokyew@unisza.edu.my

ABSTRACT

The dielectric properties of a microwave absorbing material represent the ability of the material to absorb microwave signals and dissipated those signals as heat. Carbonaceous materials are preferable to be used as microwave absorbing material due to their excellent dielectric properties. In this paper, coconut shell in powder form was used as the carbonaceous material and the composite samples were prepared in epoxy resin matrix. Five different ratios of coconut shell: epoxy resin (30:70, 40:60, 50:50, 60:40, 70:30) were prepared in order to investigate the effect of carbonaceous material composition on the dielectric properties measurement. Composites with smooth and rough surface textures were fabricated in order to investigate the effect of surface texture on the dielectric properties measurement. Carbon, hydrogen, nitrogen and sulphur (CHNS) elemental analysis was performed to determine the carbon composition in coconut shell powder. It was evaluated that the coconut shell powder possesses 48.37% of carbon composition. The structural characteristic of the coconut shell powder particles and surface texture were examined using scanning electron microscope (SEM). Presence of irregular shape particles with macropores range (1 µm) porosities was detected in the coconut shell powder. Presence of uneven surface with air gap of approximately 60 µm in diameter was detected on composite with rough surface. Experimental measurement on the dielectric properties of coconut shell-polymer (CSP) composites was performed by using open-ended coaxial probe method over microwave frequency range of 1-8 GHz. It was found that the surface texture of the composites influenced the measurement accuracy of the dielectric properties. From the experimental results, composites with smooth surface texture exhibit statistically significant accuracy of dielectric properties measurement (real part) with error bars that are less than 5% ($\varepsilon_r' = \varepsilon_r' \pm 0.05|\varepsilon_r^*|$), compared to rough composites surface where the error bars exceeded 5 %. The measured dielectric properties for composites were directly proportional to the composition of coconut shell powder. The optimum range of dielectric properties at ε_r' (3.599-3.966), ε_r'' (0.381-0.572) and tan δ (0.101-0.152) was measured for composite with 70 wt% coconut shell powder composition. The electrical conductivity of the composites increased accordingly as the composition of coconut shell powder increases over frequency of 1-8 GHz. The prepared coconut shell-polymer composites can be utilized for electromagnetic suppression (EMI) application.

Keywords: surface texture, carbonaceous, dielectric properties.

INTRODUCTION

In recent years, researchers have focused to diversify the organic carbonaceous sources from agricultural wastes such as rice husk (Nornikman *et al.*, 2010), coconut shell (Salleh *et al*., 2011), banana leave (Farhany *et al*., 2012) and sugarcane bagasse (Zahid *et al.*, 2013) as the dielectric materials for microwave absorber. Carbonaceous materials are potentially useful as microwave absorbing materials due to its excellent dielectric properties and electrical conductivity (Menéndez *et al*., 2010), (Lee *et al*., 2013). Microwave absorbers are materials that attenuate the energy in electromagnetic waves (EM) and are used to suppress electromagnetic interference (EMI). The carbon based microwave absorbers absorb the EM energies that propagates through it, converts and dissipates those energies as heat. Microwave absorbing materials can be manufactured by a number of dielectric materials in powder form, loaded with various kind of polymeric binder (Abbas, Chandra, Verma, Chatterjee, and Goel, 2006).

In this paper, experimental investigation on the dielectric properties measurement of the coconut shell powder prepared with epoxy resin matrix composite with respect to the surface texture and the carbonaceous material composition over microwave frequency of 1 GHz to 8 GHz was performed. The dielectric properties of a material is represented by relative complex permittivity (Mohsenin, 1984), (Folgueras, Alves, and Rezende, 2010), (Nelson, 2015). The relative complex permittivity consists of a real part (dielectric constant) and imaginary part

ARPN Journal of Engineering and Applied Sciences

(dielectric loss factor). The real part represents the ability of the material to absorb microwave energy and the imaginary part represents the ability of the material to convert and dissipate the microwave energy to heat. The imaginary part of permittivity is always greater than zero and is usually much smaller than the real part of permittivity (Agilent, 2014). The relative complex permittivity is presented by equation (1). The dissipation losses of the medium can be determined through the dielectric loss tangent or tangent delta (tan δ). Tangent delta is defined as the ratio of the imaginary part of the permittivity to its real part. It is known that the greater the dielectric loss tangent of the material, the greater the attenuation as the EM waves travel through the material (Dixon, 2012). Equation (2) presents the tangent delta.

$$\varepsilon_r^* = \varepsilon_r' - j\varepsilon_r'' \qquad (1)$$

ε_r^* = relative complex permittivity,

ε_r' = real part or dielectric constant,

ε_r'' = imaginary part or dielectric loss factor.

$$\tan\delta = \frac{\text{Energy lost}}{\text{Energy stored}} = \frac{\varepsilon_r''}{\varepsilon_r'} \qquad (2)$$

EXPERIMENTAL

Elemental composition determination

The elemental composition of coconut shell powder was determined through CHNS elemental analysis by using Vario MICRO Cube CHNS Analyser at temperature of 23±3°C and relative humidity of 50±5%. The aim of performing elemental analysis is to evaluate the carbon composition in coconut shell powder. Carbon is an important element in microwave absorbing materials as it is suitable to convert microwave energy to thermal energy (Lam and Chase, 2012). For comparison purpose, the elemental composition on rice husk and sugarcane bagasse was also performed.

Composite preparation

The coconut shell powder was mixed in epoxy resin matrix. Amine hardener was used to facilitate the curing process. IKA RW 20 Digital Stirrer was used to uniformly stir the mixture at 100-140 moderate revolution per minute (rpm) for approximately an hour. The mixture was then poured into planar shape mould and left to be completely cured at room temperature for approximately 48 hours. Two concerns were considered during the composite preparation. First concern is on the excessive loading of coconut shell powder in the epoxy resin. Heavy loading of particles in the epoxy resin causes cracks in the composite samples as the resin molecules are not sufficient to bind the particles together. The second concern is on the excessive loading of epoxy resin with respect to dielectric materials. Excessive loading of epoxy resin reduces the dielectric behaviour of the composite samples. The composites were prepared into five different ratios, as shown in Table-1.

Table-1. Mixture ratio of coconut shell/epoxy resin.

Composite	Coconut shell (wt %)	Epoxy resin (wt %)
Sample 1	30	70
Sample 2	40	60
Sample 3	50	50
Sample 4	60	40
Sample 5	70	30

For open-ended coaxial probe dielectric properties measurement purpose, the sample or material under test (MUT) must be infinite in size. The minimum requirement for sample thickness is very important to minimise the fringing field effect (Iqbal, Malek, Lee, Zahid, and Mezan, 2014). The sample thickness requirement is based on equation (3) with sample diameter that is greater than 20mm (Agilent, 2012a). In this work, the composite samples were fabricated in 30 mm x 30 mm planar mould with sample thickness of 5 mm.

$$\text{Sample Thickness} > \frac{20}{\left(\sqrt{|\varepsilon_r^*|}\right)} \qquad (3)$$

$|\varepsilon_r^*|$ = modulus relative complex permittivity

The modulus relative complex permittivity is equal to the absolute value of the complex number, as presented in equation (4) (Bronshteĭn and Semendyayev, 1972), (Kelley, 2012).

$$\left|\varepsilon_r^*\right| = \sqrt{\left(\varepsilon_r'\right)^2 + \left(\varepsilon_r''\right)^2} \qquad (4)$$

The surface condition of the composites can vary depending on the fabricating process. For composites with smooth surface texture, the mixture was fabricated into the mould that was placed on flat surface. On the other hand, the mould was placed on uneven surface in order to form composites with rough surface texture. Refer to Figure-1 for the example of smooth and rough composite surface textures.

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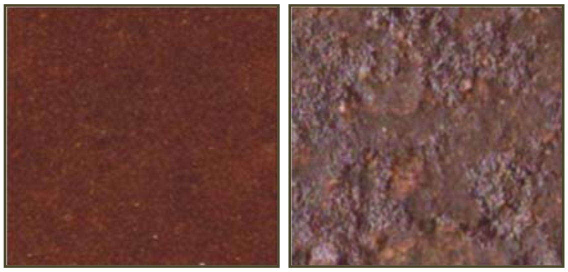


(a) (b)

Figure-1. Coconut shell-polymer composites surface texture (a) smooth, (b) rough.

Material/ composite characterisation

The particle size of the coconut shell powder and surface morphology of coconut shell-polymer composites were examined using Hitachi TM 3000 tabletop and JEOL JSM-6360LA scanning electron microscope (SEM) operated in high magnification with accelerating voltage of 5kV-15 kV.

Dielectric properties measurement

The dielectric properties measurement was performed by using open-ended coaxial probe method. The open-ended coaxial probe method represents open-structure measurement technique and has been used for permittivity measurements (Venkatesh and Raghavan, 2005), (Moukanda, Ndagijimana, Chilo, and Saguet, 2006), (Filali, Boone, Rhazi, and Ballivy, 2008), (Tereshchenko, Buesink, and Leferink, 2011), (Iqbal *et al*., 2014), (Meaney, Gregory, Epstein, and Paulsen, 2014).

The apparatus used for the dielectric properties measurement include the Agilent 85070 High Temperature Dielectric Probe Kit and software, Agilent PNA E8362B Series Network Analyser and coaxial cables. Refer to Figure-2. The high temperature dielectric probe withstands a wide –40 to +200°C temperature range, which allows measurement versus frequency and temperature. The large flange allows measurements of flat surfaced solid materials, in addition to liquids and semi-solids. A “three standard” calibration (open, short, water) was performed at the end of the dielectric probe before the dielectric measurement process (Agilent, 2012a). The coaxial probe, load for “short” and “short” calibration arrangement are shown in Figure-3. Measurement was made by contacting the coaxial probe to the surface of the composite.

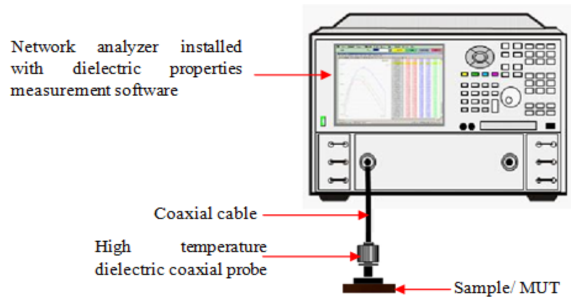
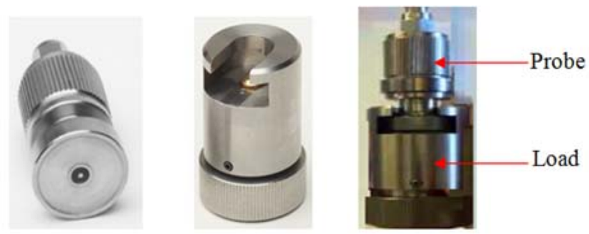


Figure-2. Open-ended dielectric properties measurement method.



(a) (b) (c)

Figure-3. (a) Coaxial probe, (b) load for “short” calibration purpose (c) “short” calibration arrangement.

The high temperature dielectric probe model accuracy for complex permittivity measurement are based on equation (5) and (6) (Agilent, 2012a).

$$\varepsilon_r' = \varepsilon_r' \pm 0.05|\varepsilon_r^*| \quad (5)$$

$$\varepsilon_r'' = \varepsilon_r'' \pm 0.05|\varepsilon_r^*| \quad (6)$$

RESULT AND DISCUSSIONS

CHNS elemental analysis

Table-2 presents the elemental composition of coconut shell powder, rice husk and sugarcane bagasse. From the elemental analysis, it was found that carbon is the most dominant solid element in those specimens. It was evaluated that the coconut shell powder possess greater percentage (48.37%) of carbon composition compared to rice husk (37.76%) and sugarcane bagasse (43.69%) (CTRM, 2015). This result prove that coconut shell powder is preferable useful as microwave absorbing material due to higher composition of carbon compared to rice husk and sugarcane bagasse.

ARPN Journal of Engineering and Applied Sciences

Table-2. Elemental composition of organic carbonaceous sources.

Result (wt **%**)	**Coconut shell**	**Rice husk**	**Sugarcane bagasse**
Carbon	48.370	37.760	43.690
Hydrogen	6.313	6.117	6.355
Nitrogen	1.620	0.970	1.830
Sulphur	0.306	0.380	0.448
Oxygen (by difference)	43.391	54.773	47.677

Structural characteristic

Figure-4 shows the scanning electron micrograph observation of the coconut shell particles and coconut shell-polymer composite. Figure-4(a) and Figure-4(b) reveal the particle's distribution morphology and size of particle porosity. It was observed that the particles of coconut shell powder are of irregular shaped particle. Presence of macropores (≈ 1 µm) was detected in particle of coconut shell powder. By referring to IUPAC, macropores are those pores with pore with greater than 50 nm (McCusker, Liebau, and Engelhardt, 2001), (Radovic, 2004). Materials with smaller pore size resulted in larger surface area. This is to enhance the conduction losses and the capability of microwave absorption (Liu, Zhang, and Wu, 2011), (Che *et al.*, 2015). Figure-4(c) and Figure-4(d) reveal the surface morphology of the smooth and rough surface textures of coconut shell-polymer composite. As seen, for composite with rough surface texture, the uneven surface (air gap) is approximately 60 µm in diameter.

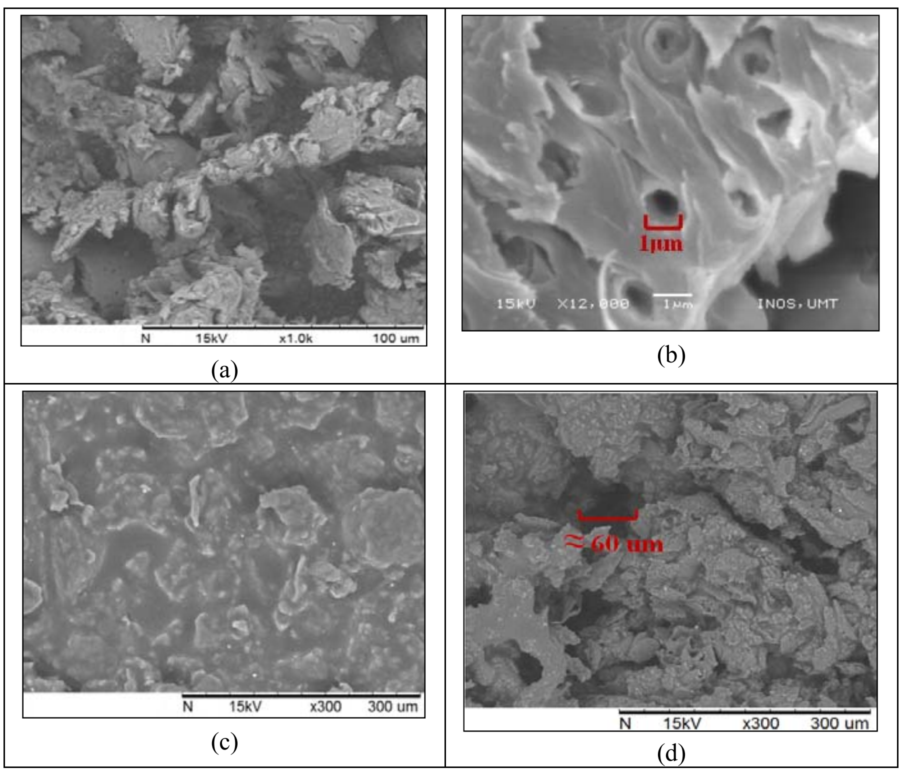


Figure-4. SEM observation (a) coconut shell particles, (b) size of particle porosity, (b) surface morphology of composite with smooth surface texture, (c) surface morphology of composite with rough surface texture.

Dielectric properties measurement

Figure-5 and Figure-6 show the frequency spectrum of the complex permittivity (real and imaginary parts) for the CSP composites with respect to the composition ratio and surface texture. It can be observed that the measured value of the dielectric constant of the composite samples are greater than the air (ε'_{air} = 1.00). This indicates the ability of the materials to be polarized at greater extent than air (Iqbal *et al.*, 2014).

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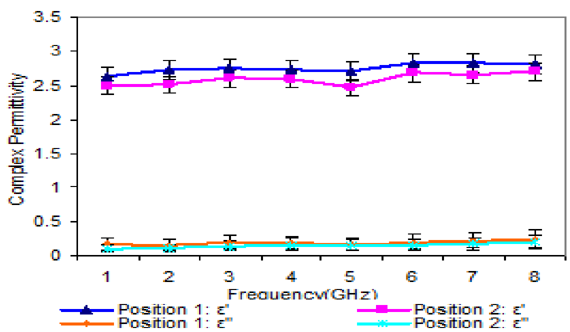
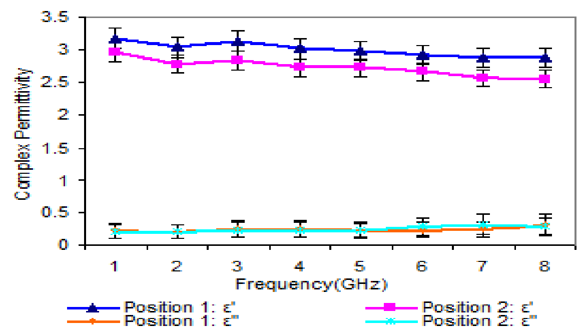
Effect of surface texture on the measurement accuracy of complex permittivity

Measurement of dielectric properties was performed on two difference random positions. The complex permittivity were plot with accuracy error bars for the real and imaginary part of permittivity ($\varepsilon_r' = \varepsilon_r' \pm 0.05|\varepsilon_r^*|$ and $\varepsilon_r'' = \varepsilon_r'' \pm 0.05|\varepsilon_r^*|$). Accuracy error bars were used to visually compare two quantities and to determine whether the differences are statistically significant (Crawley, 2014). It can be observed that for the composites with smooth surface texture (Figure-5), the accuracy error bars of the real part ($\varepsilon_r' = \varepsilon_r' \pm 0.05|\varepsilon_r^*|$) measured values on position 1 and position 2 overlapped over the frequency range of 1-8 GHz. This indicates that the real part measured values of the composites with smooth texture were statistically significant and the error bars are less than 5%. For composites with rough surface texture (Figure-6), the accuracy error bars of the real part does not overlapped for the measured values on position 1 and position 2. The real part measured values of the composite with rough surface texture were not statistically significant and exceeded the 5% error bars. While for the measued values on imaginary part, for both smooth and rough composite surface textures, the accuracy error bar ($\varepsilon_r'' = \varepsilon_r'' \pm 0.05|\varepsilon_r^*|$) on position 1 and position 2 overlapped. As imaginary part of permittivity is usually much smaller than the real part of permittivity, the accuracy error bars show insignificant difference.

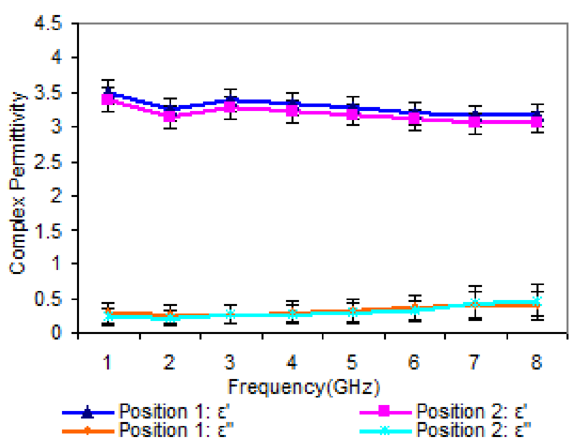
There are three main sources of errors that affect the dielectric measurement by using open-ended coaxial probe method, namely cable stability (system drift errors), sample thickness and air gap. The cable instability can be virtually eliminates by using the automated Electronic Calibration Refresh feature and allow enough time for the cable to stabilise before making any measurement. The errors due to sample thickness can be minimised by fabricating the material under test (MUT) based on equation (3). For composite solid materials, air gap between the coaxial probe and the uneven composite surfaces can be a significant source of errors (Agilent, 2014). For composite with rough surface texture, the dimension of the air gaps that exist on the composite surfaces is greater that than of composite with smooth surface texture. It is easier for small wavelength signals to leak from the air gaps that are formed from rough composite surfaces (Iqbal *et al.*, 2014).



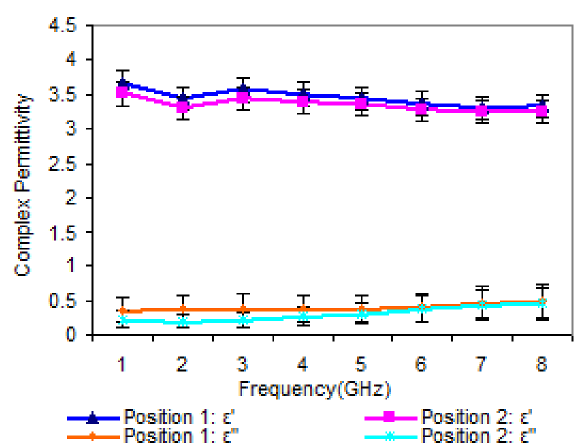
(a) Sample 1 (ratio 30:70)



(b) Sample 2 (ratio 40:60)



(c) Sample 3 (ratio 50:50)



(d) Sample 4 (ratio 60:40)

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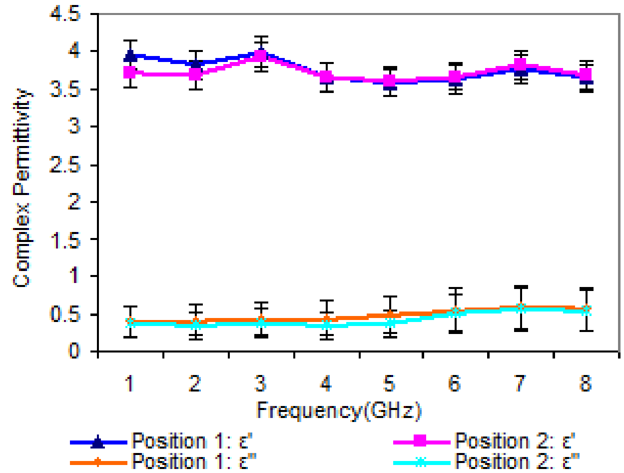
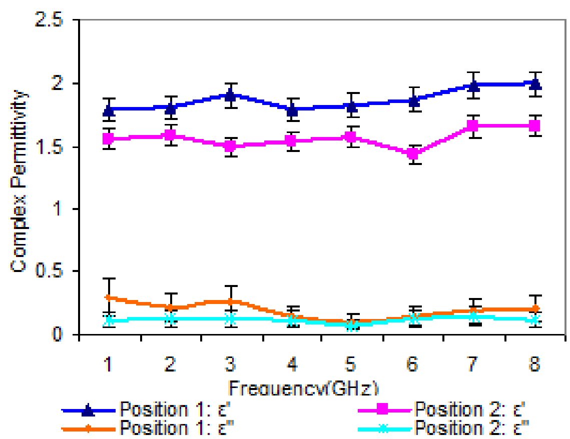
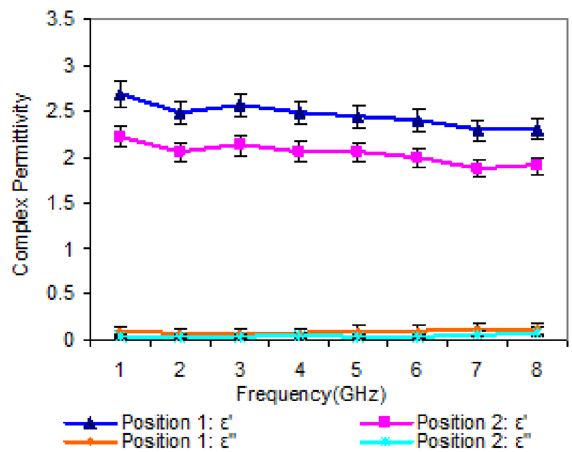


(e) Sample 5 (ratio 70:30)

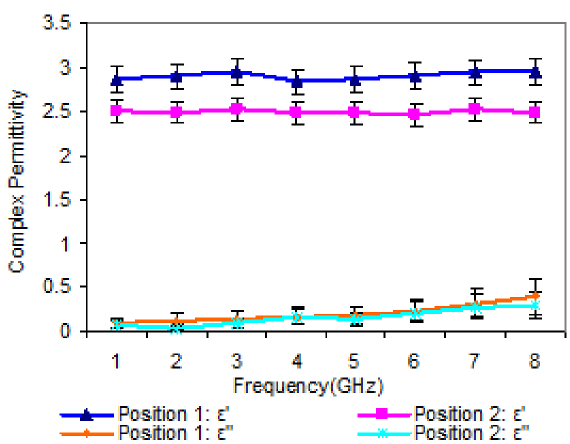
Figure-5. Complex permittivity of composites with smooth surface texture.



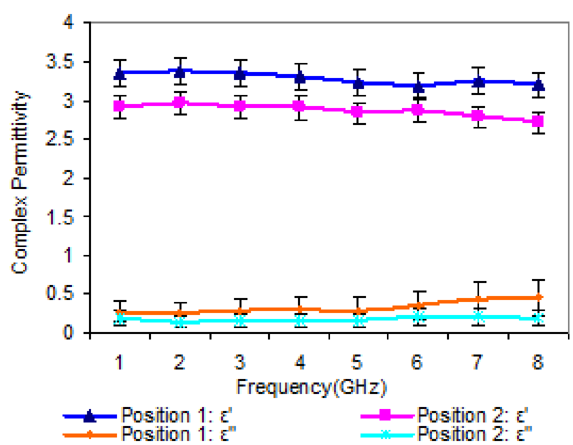
(a) Sample 1 (ratio 30:70)



(b) Sample 2 (ratio 40:60)



(c) Sample 3 (ratio 50:50)



(d) Sample 4 (ratio 60:40)

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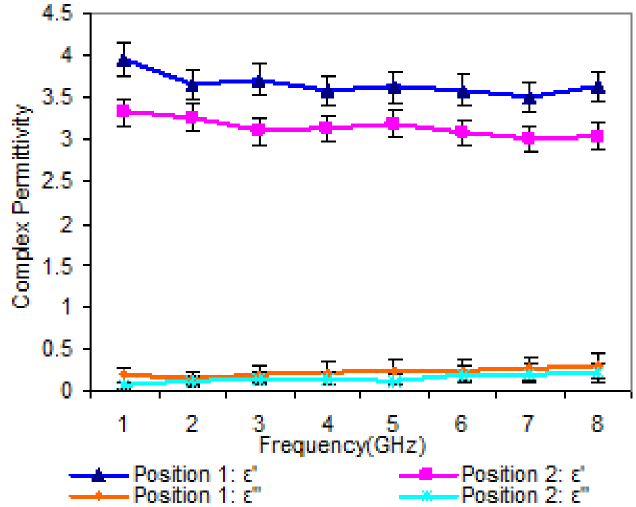

(e) Sample 5 ratio 70:30)

Figure-6. Complex permittivity of composites with rough surface texture.

Effect of the carbonaceous material composition on the measurement of complex permittivity

The composites with smooth surface texture (statistically significant accuracy) were considered in order to analyse the effect of carbonaceous materials composition on the complex permittivity measurement. The average complex permittivity measured on two difference positions over frequency range of 1-8 GHz was considered.

Figure-7 shows the frequency spectrum of composite with respect to the composite composition. It was found that the dielectric properties of the sample increases accordingly as the coconut shell powder composition in the sample increases. Sample 5 with 70 wt% of coconut shell powder was in the highest range of ε_r' (3.599-3.966) and ε_r''(0.381-0.572), whereas sample 1 with 30 wt% of coconut shell powder was in the lowest range of ε_r' (2.567-2.678) and ε_r''(0.133-0.222). Samples with higher ratio of coconut shell powder possess greater dielectric properties due to the presence of higher carbonaceous (dielectric) material. The dissipation losses of the samples were determined through tangent delta *(*tan *δ)*. Figure-8 shows the frequency spectrum of tangent delta with respect to the composite composition. It was observed that the overall pattern of tangent delta increases as the coconut shell powder composition in the sample increases from 30 wt%, 40 wt%, 50 wt%, 60 wt% to 70 wt%. The result shows that sample 5 with 70 wt% of coconut shell powder possess the greatest attenuation as the EM waves propagate through the composite. Table-3 presents the comparison of the dielectric properties and tangent delta for sample 1 to sample 5.

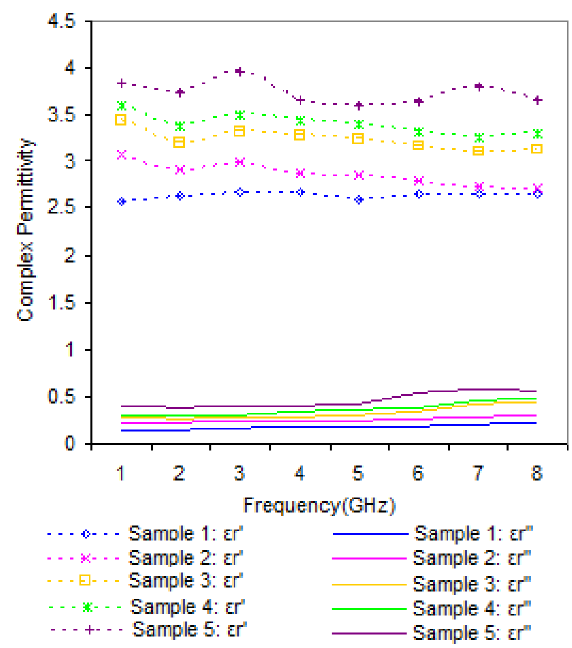

Figure-7. Complex permittivity of the CSP composites.

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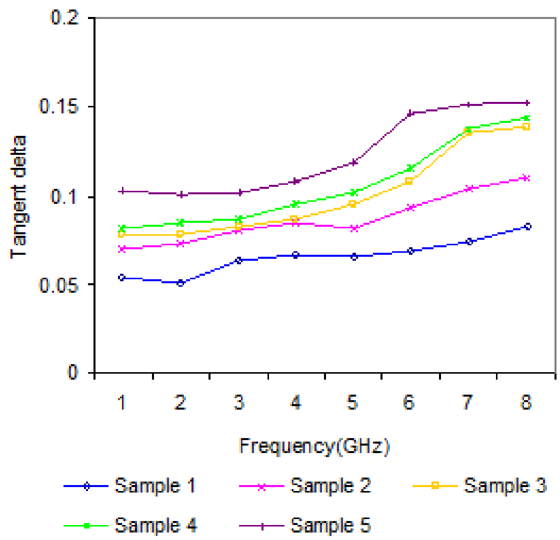

Figure-8. Tangent delta of the CSP composites.

Table-3. Measured dielectric properties.

Composite	Dielectric properties	Range over 1-8 GHz
Sample 1	ε_r'	2.567-2.678
	ε_r''	0.133-0.222
	tan δ	0.051-0.083
Sample 2	ε_r'	2.716-3.068
	ε_r''	0.211-0.299
	tan δ	0.070-0.110
Sample 3	ε_r'	3.110-3.452
	ε_r''	0.249-0.433
	tan δ	0.078-0.139
Sample 4	ε_r'	3.272-3.596
	ε_r''	0.290-0.474
	tan δ	0.081-0.144
Sample 5	ε_r'	3.599-3.966
	ε_r''	0.381-0.572
	tan δ	0.101-0.152

Composite with greater amount of carbon element not only affect the dielectric properties of the material, but also affect the electrical conductivity of the material. As the microwave energy passing through the microwave materials, electrical field is produced and the electrical energy is converted into thermal energy and dissipated as heat(Zahid *et al.*, 2013). The electrical conductivity (σ) is determined based on the measured value of imaginary part of permittivity. The electrical conductivity is represented in equation (7) (Micheli *et al.*, 2011), (Agilent, 2012b). Figure-9 shows the plot of electrical conductivity with respect to the frequency. It can be observed that the electrical conductivity is proportional to the frequency and the measured value of dielectric loss factor (ε_r'').

$$\sigma = \omega \varepsilon_o \varepsilon_r'' \quad (7)$$

σ = electrical conductivity (S/m)
ω = angular velocity (rad/s)
ε_o = free space permittivity (8.854 x 10^{-12} F/m)
ε_r'' = Imaginary part or dielectric loss factor.

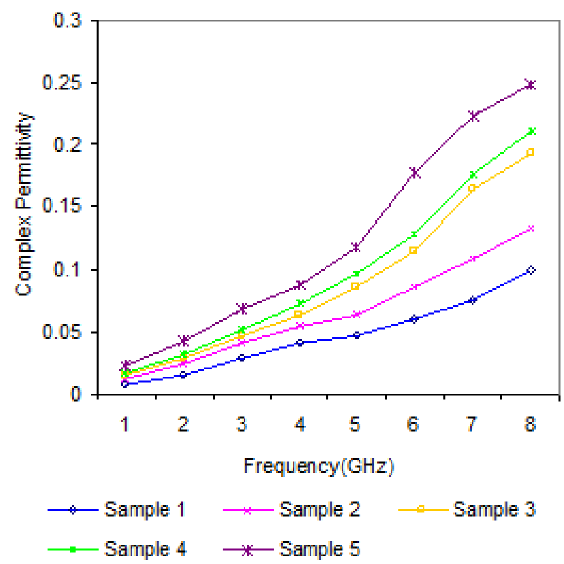

Figure-9. Electrical conductivity of the CSP composite samples.

CONCLUSIONS

The coconut shell-polymer composites with different ratios and surface textures have been prepared in epoxy resin matrix.

The structural characteristic of the coconut shell powder and coconut shell-polymer composites were investigated in term of surface morphology and particles porosities. As seen from SEM observation, irregular shape particles with macropores range porosities were detected in the coconut shell powder. For composites with rough surface texture, the presence of uneven surfaces with air gap of approximately 60 μm in diameter was detected. The dielectric properties of CSP composites were investigated in term of the effect of surface texture and carbonaceous material composition. It was found that the surface texture barely influenced the dielectric properties of the CSP composites as the molecular structure of the composites used for this experimental purpose was the same. However, the composites with rough surface textures influenced the measurement accuracy of the dielectric properties by using the high temperature dielectric probe.

ARPN Journal of Engineering and Applied Sciences

In order to increase the accuracy of the open-ended dielectric properties measurement, the surface of the MUT must be machined to be at least as flat as the probe face.

The measured dielectric properties for composites with higher ratio of coconut shell powder were greater due to the presence of higher composition of dielectric carbonaceous materials. Composites with high composition of carbonaceous materials are desirable to be used as microwave absorbing materials as they possess greater dissipation losses and electrical conductivity.

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