Effect of a Carbon Black Surface Treatment on the Microwave Properties of Poly(ethylene terephthalate)/ Carbon Black Composites

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ABSTRACT: A surface treatment was applied to carbon black to improve the electrical and microwave properties of poly(ethylene terephthalate) (PET)-based composites. Three different formamide solutions with 1, 2, and 3 wt % concentrations were prepared to modify the surface chemistry of carbon black. Microwave properties such as the absorption loss, return loss, insertion loss, and dielectric constant were measured in the frequency range of 8–12 GHz (X-band range). Composites containing formamide-treated carbon black exhibited enhancements in the electrical conductivity, electromagnetic interference (EMI) shielding effectiveness, and dielectric constant values when compared to composites with untreated carbon black. In addition, increases in the formamide solution concentration and carbon black content of composites resulted in an increase in the electrical conductivity, EMI shielding effectiveness, and dielectric constant values. The percolation threshold concentration of PET composites shifted from a 3 to 1.5 wt % carbon black composition with the surface treatment. The best EMI shielding effectiveness was around 27 dB, which was obtained with the composite containing 8 wt % carbon black treated with a 3 wt % formamide solution. Moreover, this composition gave the lowest electrical resistivity and the highest dielectric constant among the produced composites. J Appl Polym Sci 109: 152–159, 2008

Key words: composites; conducting polymers; dielectric properties; polyesters

INTRODUCTION

Electromagnetic interference (EMI) is degradation in the performance of equipment by electromagnetic disturbances, which might be due to electromagnetic noise or an undesired signal. The EMI problem has attracted great scientific attention in recent years because various electronic devices, radio and television broadcast stations, communication transmitters, and radar equipment emit electromagnetic energy. This radiation may interfere with the operation of electrical and electronic devices.[1] Devices need to be shielded against the transmission of electromagnetic waves. Electromagnetic shielding is the method used to reduce or prevent the interference of undesired electromagnetic radiation with devices.[1,2]

Two mechanisms dominate in terms of EMI shielding: reflection loss and absorption loss. Some parts of electromagnetic waves are reflected from the shielding surfaces. Mobile charges, which are electrons and holes, are responsible for the reflection mechanism by interacting with electromagnetic waves. In addition, some parts of waves are absorbed in the shielding material. To absorb electromagnetic waves, a shield should possess electric and magnetic dipoles.[1,3] Besides absorption and reflection mechanisms, there are multiple reflections between interfaces of the shields. The multiple reflection mechanism may be taken into account when the absorption mechanism is not effective. As a result, the sum of the reflection loss, absorption loss, and internal reflection loss constitutes the EMI shielding effectiveness:[1]

$$\begin{aligned}\text{Shielding effectiveness (dB)} = {} & \text{Reflection loss (dB)} \\ & + \text{Absorption loss (dB)} \\ & + \text{Internal reflection loss (dB)} \quad (1)\end{aligned}$$

Various studies have been carried out on conductive polymer composites as EMI shielding materials.[4–8] Yang et al.[4] studied the EMI shielding effectiveness of carbon-nanofiber-filled liquid-crystalline polymer composites. With 10 wt % carbon filaments, they exhibited an EMI shielding effectiveness of 16 dB at 1.5 GHz.[4] Some microwave properties of composites filled with carbon black (CB) and carbon fiber were studied in the X-band range.[5,6] Ethylene vinyl acetate based composites with a 20 phr CB loading had

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an effectiveness of nearly 5 dB at 8 GHz,[5] whereas polypropylene-based composites with 25 wt % CB showed an effectiveness of nearly 10 dB at 10 GHz.[6] In addition, the microwave properties, insertion loss, and return loss of composites filled with nickel-coated carbon fiber were studied in the X-band range.[7] With 10 wt % carbon fiber, the insertion loss value of the polypropylene composite reached 10 dB between frequencies of 8 and 12 GHz.[7] Moreover, the effect of processing conditions on the EMI shielding effectiveness of carbon-fiber-filled composites was investigated.[8] As the screw speed increased and process temperature decreased, the average carbon fiber length and shielding effectiveness of composites decreased.

Any attempt to increase the electrical conductivity of composites may improve the EMI shielding effectiveness.[1,4–8] The electrical resistivity of conductive polymer composites especially depends on the electrical resistivity of conductive fillers.[9] CB was exposed to chemical treatments to improve the electrical property of polymer composites.[10,11] In a previous study, Koysuren et al.[12] investigated the effect of a surface treatment on the electrical conductivity of CB-filled low-density polyethylene and nylon 6 based composites. Composites filled with chemically treated CB showed an improvement in electrical conductivity compared to untreated carbon black (UCB) filled composites. Surface modifiers, one of which was formamide, were used to react with CB, resulting in a doping reaction. The amide-type functional group of formamide reacted with the carboxyl group on CB to yield the amide salt of $R—NH_3^+—COO^-$, for which a nitrogen atom was in the quaternary structure. According to electron spectroscopy for chemical analysis (ESCA), the N_{1s} spectrum of CB treated with a 1 wt % formamide solution confirmed this reaction, with the peak for the $—NH_3^+$-type nitrogen at 398.6 eV. The percolation threshold of the composites was shifted from 5 to 3 wt % CB with the surface treatment of the conductive filler.[12]

Additionally, the dielectric property is as important as the EMI shielding effectiveness of electronic and electrical devices because an incident electric field from any source will affect the operational performances of this equipment. This effect of an electric field might be diminished by the use of a shielding material with a high dielectric constant value. Under an external electric field, mobile charge carriers of the shielding material are pushed in opposite directions, and the distorted charge distribution forms its own electric field that opposes the external field, thereby causing a reduction in the net field within the device.[13,14] The dielectric constant is defined as the ratio of the applied field to the field inside the material.[15] The electrical conductivity and dielectric constant values of an insulating polymer can be improved by the incorporation of a conductive filler into its matrix. There have been a few studies on the dielectric properties of conductive polymer composites, and most of these studies have included low-frequency dielectric constant measurements.[16–18] The dielectric constant of a polypropylene/6 wt % CB composite was around 30 between 10^{-1} and 10^6 Hz.[16] Moreover, the dielectric constant of a CB-filled polyethylene composite was around 30 at 1 kHz, whereas the value was almost 80 for a ternary system of high-density polyethylene and ethylene vinyl acetate including 6 wt % CB.[17] Carbon nanotubes at a concentration of 3.6 vol % in a polyethylene matrix exhibited a dielectric constant value of nearly 450 at 1 kHz.[18] However, the frequency dependence of the dielectric constant is high, and the dielectric constant decreases with increasing frequency.[17–20] The dielectric constant of a polyethylene/3.6 vol % carbon nanotube composite decreased to around 25 as the frequency increased to 10^7 Hz.[18] A relatively low dielectric constant was measured for a CB-filled acrylonitrile-butadiene-styrene system in the frequency range of 6.4–6.7 GHz.[21] The composite exceeded a dielectric constant value of 8 with 20 vol % CB.[21]

In this study, microwave properties such as the absorption loss, return loss, insertion loss, and dielectric constant were studied for composites containing CB. A comprehensive characterization of the poly(ethylene terephthalate) (PET)-based composites with formamide-treated CB was performed in terms of the electrical, microwave, and dielectric properties. To reveal the effect of the surface treatment on the electrical and microwave properties, composites with treated CB were compared with composites containing UCB. The microwave properties of the composites were studied in the X-band range from 8 to 12 GHz.

EXPERIMENTAL

Materials

In this study, PET as a matrix material, CB as a conductive filler, and formamide as a chemical treatment agent for CB were used, and their physical properties are given in Table I.

Composite preparation

Before compounding with PET, the surface chemistry of CB was modified with formamide solutions, which were prepared by the dilution of formamide with distilled water to obtain solutions with 1, 2, and 3 wt % formamide concentrations. UCB particles were mixed with diluted surface-modifier solutions, and these mixtures were stirred for 10 min at room

TABLE I
Physical Properties of the Materials

Material	Trade name (supplier)	Specifications
PET	Advansa (Melinar)	Melting temperature = 250°C Electrical resistance = 10^{14} Ω cm Density = 1.4 g/cm^3
CB	ISAF N-220 (Turkish Petroleum Refinery)	Iodine number = 119 mg/g Dibutyl phthalate absorption = 114.2 mL/100 g
Formamide	Formamide (Merck)	Density (20°C) = 1.13 g/cm^3 Flash point = 175°C

temperature. Then, treated CB particles were filtered from solutions, and wet particles were dried at 100°C for 24 h.[12] FA1–CB, FA2–CB, and FA3–CB were the codes used to identify CB treated with 1, 2, and 3 wt % formamide solutions, respectively.

PET pellets were dried in a vacuum oven for 12 h at 80°C before the extrusion process. PET and CB were compounded with a Thermo Prism TSE-16-TC corotating twin-screw extruder (Staffordshire, England) to prepare composites containing 1, 2, 4, 6, and 8 wt % CB. The extrusion process was carried out with the temperature profile of 230–255–260–265–270°C at a screw speed of 80 rpm.

PET/CB test samples were compression-molded at 285°C to characterize their electrical properties. First, pellets were heated for 1.5 min under a gauge pressure of 50 bar, and then they were heated for 1 min under a gauge pressure of 150 bar. Finally, compression-molded samples were quenched to room temperature by water. A DSM Micro 10-cc laboratory-scale injection-molding instrument (Geleen, The Netherlands) was used to prepare test specimens for microwave measurements. During the injection-molding process of PET-based composites, the barrel and mold temperatures were 285 and 30°C, respectively.

Composite characterization techniques

ESCA was used to analyze the surface chemistry of 2 and 3 wt % formamide solution treated CB. A Specs model spectrometer (Sarasota, FL) equipped with aluminum radiation at 1 W was used to obtain ESCA spectra of CB. The high-resolution spectra of nitrogen (N_{1s} peak) were recorded with a pass energy of 48 eV at a vacuum level lower than 10^{-5} Pa. A nonlinear background was removed from the spectra, and XPSPeak 41, which is a curve-fitting program, was used to fit the high-resolution spectra of nitrogen.

The electrical conductivities of FA1–CB, FA2–CB, and FA3–CB in powder form were measured with the four-point probe method according to ASTM F 43. The electrical property of the compression-molded composites was examined with the two-point probe method with a Keithley 2400 constant current source (Cleveland, OH). For better electrical contact in the two-point probe method, copper wires were placed in the compression-molded composites during sample preparation. Conductivity measurements were performed by the probes being placed in contact with these copper wires. All measurements were done at room temperature, and the average of six measurements was taken into account for each composition.

Microwave properties of composites containing 4, 6, and 8 wt % treated CB and UCB were tested with a Hewlett–Packard HP 8720D network analyzer (Englewood, CO) and a wave guide with dimensions of 2.286 cm × 1.016 cm operating in the X-band range (8–12 GHz). To prevent radiation leakage during absorption loss, return loss (reflection loss), and insertion loss measurements, test samples were fitted precisely to the shape of the wave guide. The thickness of the test samples was 4 mm. Before the measurements, the network analyzer was calibrated according to the X-band range. The incident, reflected, and transmitted waves were measured in the absence of samples (which were taken as reference data) and in the presence of the test samples. Absorption loss, return loss, and insertion loss data were obtained in decibels. During absorption loss measurements, a conductor was placed behind the samples. Hence, radiation that was not absorbed into the sample could be returned. Return loss and insertion loss measurements were conducted in an air medium.

The dielectric property of composites including 4, 6, and 8 wt % treated CB and UCB was measured with a setup including an FMI 449x microwave source (Narda Microwave Corp., Folsom, CA), a model 441F voltage standing wave ratio (VSWR) meter (Narda Microwave Corp., Folsom, CA), a slotted line, an isolator, a wavemeter, and a sliding short (Fig. 1). Test samples, used during absorption loss, return loss, and insertion loss measurements, were used to measure dielectric constant values of composites at 8.5 and 10 GHz, respectively. The measurement method involved inserting the dielectric sample at the end of a short-circuited wave guide. To

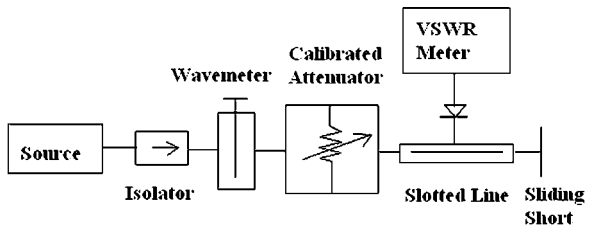

Figure 1 Experiment setup for the dielectric constant measurements.

calculate the dielectric constant, the voltage standing wave pattern without the test sample was measured. When the test sample was inserted, the position of the voltage minimum shifted by a distance of L. By the measurement of L, dielectric constant ε_r was calculated from the following relationships:

$$\lambda_o = \frac{c}{f_o} \tag{2}$$

$$\frac{1}{\lambda_g^2} = \frac{1}{\lambda_o^2} - \frac{1}{(2a)^2} \tag{3}$$

$$\frac{\tan\left(2\pi\frac{d}{\lambda_{g\varepsilon}}\right)}{\left(2\pi\frac{d}{\lambda_{g\varepsilon}}\right)} = \frac{\lambda_g}{2\pi d}\tan\left(2\pi\frac{(L+d)}{\lambda_g}\right) \tag{4}$$

$$\frac{1}{\lambda_{g\varepsilon}^2} = \frac{\varepsilon_r}{\lambda_o^2} - \frac{1}{(2a)^2} \tag{5}$$

where c is the speed of light, f_o is the applied frequency, λ_g is the guided wavelength without the sample, a is equal to 2.286 cm (the larger dimension of the wave guide), d is the thickness of the sample, and $\lambda_{g\varepsilon}$ is the guided wavelength in the sample-filled region.

Fractured surfaces of injection-molded specimens were analyzed with a JEOL JSM-6400 scanning electron microscope (Tokyo, Japan). Morphologies of the composites containing 6 wt % UCB and 6 wt % FA1–CB were studied. To increase CB resolution in scanning electron microscopy (SEM) micrographs, injection-molded specimens were fractured and stained with nitric acid for 3 h at room temperature.

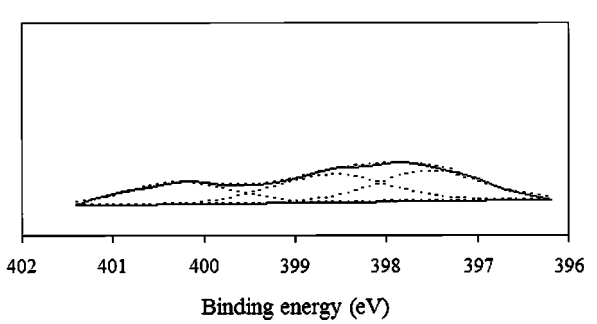

Figure 2 High-resolution spectra of nitrogen (N_{1s}) of FA2–CB.

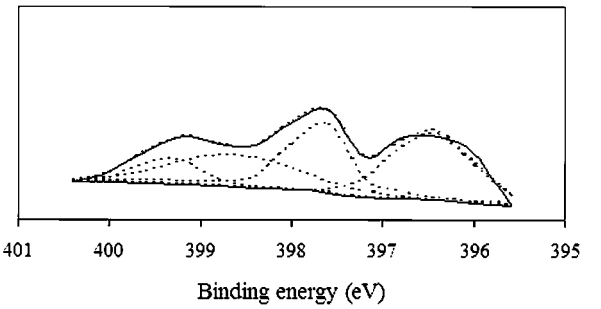

Figure 3 High-resolution spectra of nitrogen (N_{1s}) of FA3–CB.

RESULTS AND DISCUSSION

The N_{1s} spectra of FA2–CB and FA3–CB exhibit a peak for $-NH_3^+$-type nitrogen at 398.6 eV, at which the nitrogen atom is in a quaternary structure (Figs. 2 and 3). The same peak has also been found in the N_{1s} spectrum of FA1–CB, which can be found in our previous study.[12] Quaternary nitrogen, formed during the surface treatment, is crucial in terms of the doping mechanism because nitrogen as an n-type of dopant atom increases the electrical conductivity of CB and its composite by increasing the number of charge carriers.[12] Thus, formamide treatment is efficient in terms of its doping effect on the electrical conductivity of CB. Electrical conductivity values of UCB, FA1–CB, FA2–CB, and FA3–CB were determined to be 3.9, 16.9, 18.4, and 19.2 S/cm, respectively. Figure 4 exhibits the influence of treated CB and UCB on the electrical conductivity results of PET-based composites, confirming the doping effects of 1, 2, and 3 wt % formamide solutions because FA1–CB, FA2–CB, and FA3–CB enhance the electrical conductivity of the PET composites more than UCB does. Formamide treatments

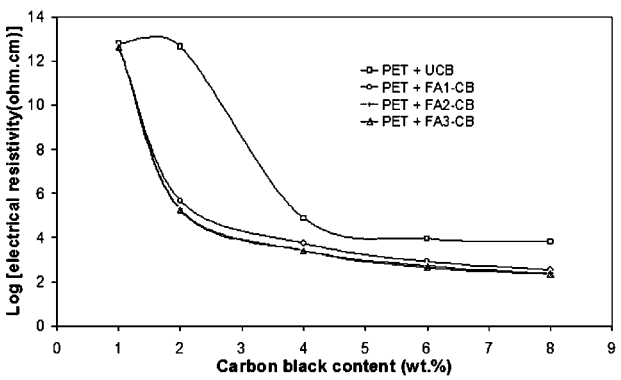

Figure 4 Electrical resistivity values of the PET-based composites.

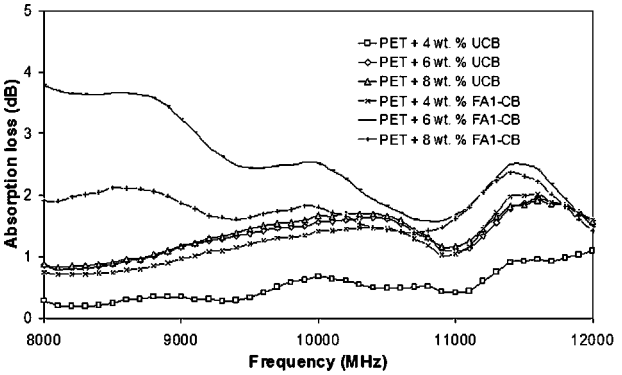

Figure 5 Variation in the absorption loss of the PET/UCB and PET/FA1–CB systems in the X-band range.

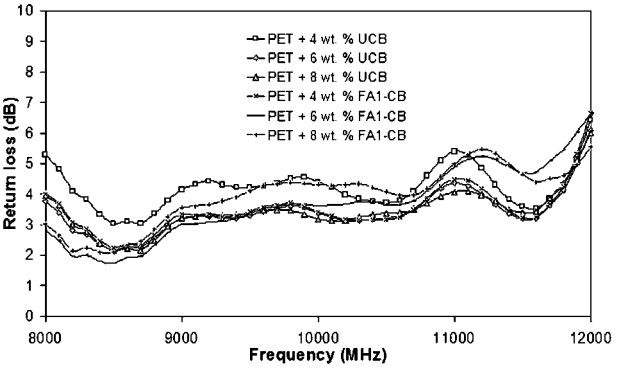

Figure 7 Variation in the return loss of the PET/UCB and PET/FA1–CB systems in the X-band range.

reduce the percolation threshold concentration from 3 to 1.5 wt % CB. In addition, lower electrical resistivity was obtained for each composition of PET composites containing treated CB in comparison with the composites having UCB. The electrical conductivities of FA2–CB/PET and FA3–CB/PET are higher than the electrical conductivity of the FA1–CB/PET system. The change in the concentration of the formamide solution from 2 to 3 wt % leads to a small improvement in the electrical conductivity of the PET composites, which is difficult to figure out from the electrical resistivity graph on a logarithmic scale (Fig. 4). In both FA2–CB- and FA3–CB-filled PET composites, the resistivity levels off around 200 Ω cm for a CB content of 8 wt %.

Microwave properties such as the absorption loss, return loss, and insertion loss of the composites having 4, 6, and 8 wt % UCB, FA1–CB, FA2–CB, and FA3–CB have been measured because these compositions exceed the percolation threshold content among the composites studied (Fig. 4). Figure 5 shows that the absorption loss changes between 0 and 4 dB for the different contents of UCB and FA1–CB particles that are incorporated into PET. The absorption loss behavior shows a trend similar to that of the electrical conductivity of the shielding material, in that the absorption loss of the composites increases with increasing filler content.[1,3] Except for the composite containing 8 wt % FA1–CB, the absorption loss is improved with the CB concentration increasing in the whole X-band range (Fig. 5). In addition, absorption loss values approach each other at a frequency close to 12 GHz. This implies that the filling effect diminishes with an increasing frequency of the incident wave. The absorption loss of the composite including 4 wt % UCB is low compared to those of all other composites. The low conductivity value of this composite may be responsible for this result. Additionally, the dimensions of the test samples are held to be the same because the absorption loss depends on the thickness of the shielding material.[1,3] Composites containing 8 wt % UCB, FA1–CB, FA2–CB, and FA3–CB have high electrical conductivity and may exhibit metallic character. This points out that these composites may function mainly by reflection rather than an absorption mechanism because of the free electrons in them.[3] Therefore, absorption loss values of all PET composites, including 8 wt % CB, do not change significantly, and this means that the surface treatment of CB seems to be

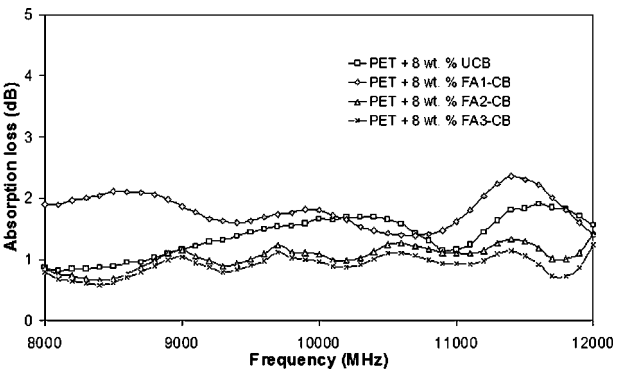

Figure 6 Variation in the absorption loss of composites containing 8 wt % UCB, FA1–CB, FA2–CB, and FA3–CB in the X-band range.

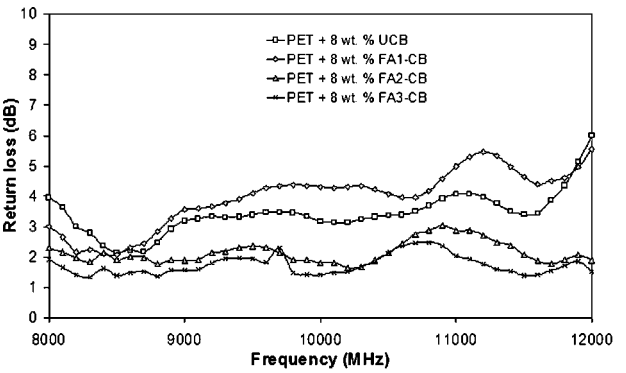

Figure 8 Variation in the return loss of composites containing 8 wt % UCB, FA1–CB, FA2–CB, and FA3–CB in the X-band range.

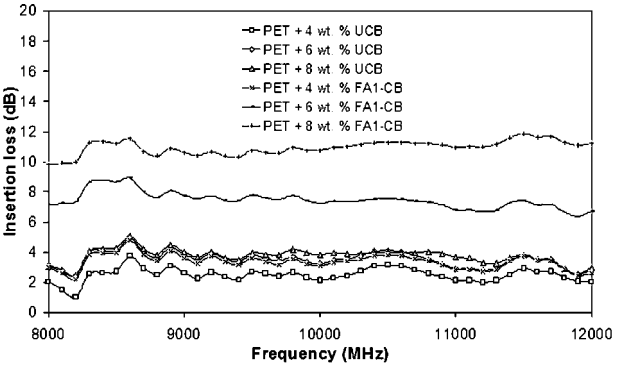

Figure 9 Variation of the insertion loss of the PET/UCB and PET/FA1–CB systems in the X-band range.

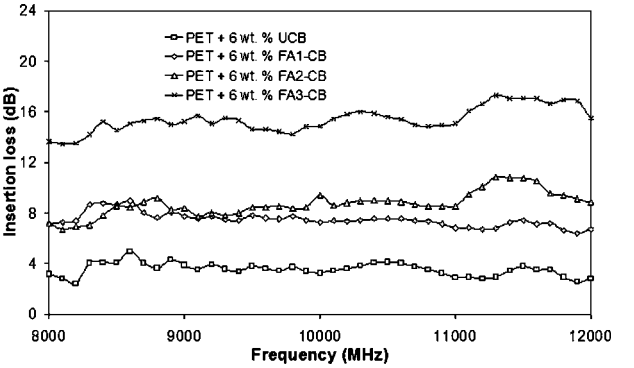

Figure 11 Variation of the insertion loss of composites containing 6 wt % UCB, FA1–CB, FA2–CB, and FA3–CB in the X-band range.

ineffective in terms of its doping effect on the absorption loss of composites (Fig. 6).

The return loss also has a direct relation with the electrical conductivity of the shielding material. As the electrical conductivity of the shielding material increases, the reflection mechanism should be more effective. That is, the return loss should diminish with increasing filler content.[1,3,5,6] Figure 7 exhibits the effects of the return loss for the different compositions of UCB and FA1–CB particles compounded with PET. As expected, the composite filled with 4 wt % UCB has a higher return loss value than the composites having 6 and 8 wt % UCB. There is a small difference between the return loss values of composites containing 6 and 8 wt % UCB as they have similar electrical conductivity values (Figs. 4 and 7). However, this trend cannot be seen by the composites filled with FA1–CB. The return loss depends not only on the conductivity of the composite but also on the polymer viscosity, polarity of the matrix, aspect ratio, and distribution of the conductive filler in the matrix.[5] The uneven variation in the return loss with the frequency for FA1–CB/PET systems may be due to the random distribution of CB in the matrix.[5] Consequently, the relation of the return loss to the CB concentration in composites seems to be complicated. That is, the return loss might not change according to the composite concentration variation.[6,7] Return losses of composites including UCB and FA1–CB vary from 2 to 7 dB (Fig. 7). Meanwhile, FA2–CB/PET and FA3–CB/PET systems exhibit lower electrical resistivity when compared with UCB/PET and FA1–CB/PET systems (Fig. 4). Parallel to the conductivity results, return loss values of PET composites having 8 wt % CB decrease as the formamide solution concentration changes from 1 to 2 and 3 wt % (Fig. 8).

The shielding effectiveness is the ratio of the electromagnetic power incident on the shielding material (P_i) to the electromagnetic power transmitted through the shielding material (P_t):[1,2]

$$\text{Shielding effectiveness (dB)} = 10 \log_{10}(P_i/P_t) \quad (6)$$

A shielding effectiveness of 10 means that 90% of incident radiation is blocked and 10% of this radiation is allowed to transmit through the shielding material. EMI shielding effectiveness is equal to the insertion loss.[2,5,7] The shielding effectiveness of pre-

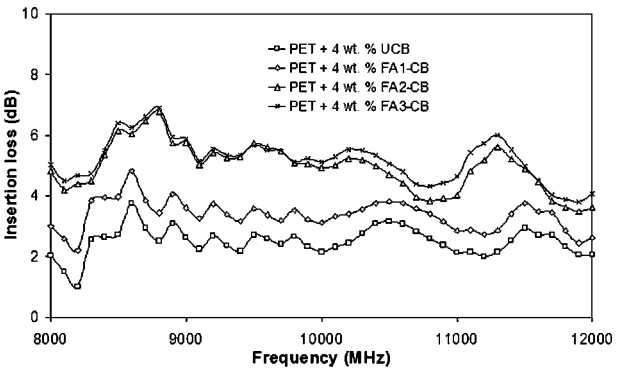

Figure 10 Variation of the insertion loss of composites containing 4 wt % UCB, FA1–CB, FA2–CB, and FA3–CB in the X-band range.

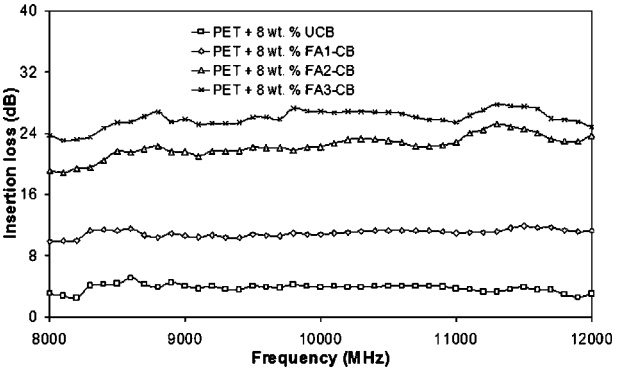

Figure 12 Variation of the insertion loss of composites containing 8 wt % UCB, FA1–CB, FA2–CB, and FA3–CB in the X-band range.

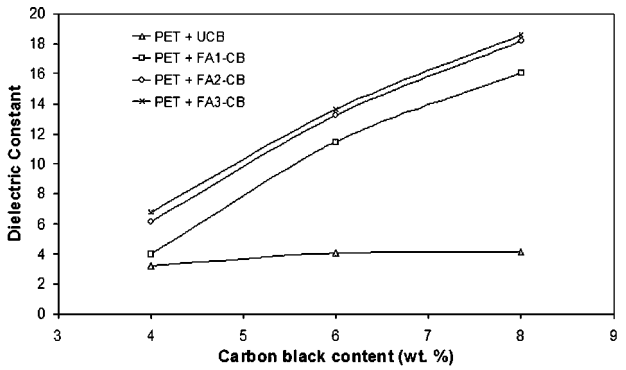

Figure 13 Dielectric constant values of the PET-based composites at 8.5 GHz.

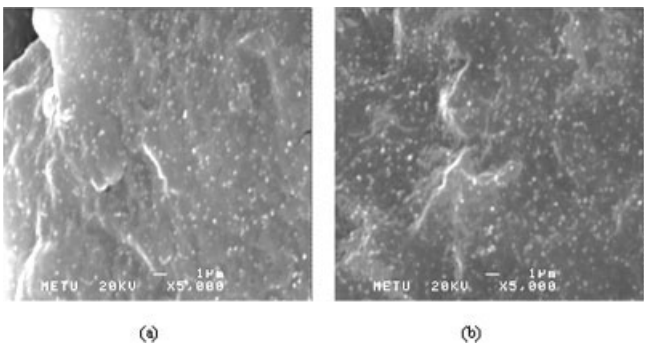

Figure 15 SEM micrographs of the PET-based composites containing (a) 6 wt % UCB (5000×) and (b) 6 wt % FA1–CB (5000×).

pared composites, which is obtained from insertion loss values, increases with increasing CB contents (Fig. 9) and formamide solution concentrations (Figs. 10–12). As expected, the EMI shielding effectiveness of composites improves with a parallel improvement in the electrical conductivity (Figs. 9–12). Higher shielding effectiveness values are obtained at low conductive filler compositions.[5,6] In particular, composites filled with 6 and 8 wt % FA1–CB, FA2–CB, and FA3–CB show higher EMI shielding effectiveness, and 27 dB of EMI shielding effectiveness is obtained when 8 wt % FA3–CB is incorporated into PET (Figs. 11 and 12); this reveals again the success in improving the electrical conductivity of PET-based composites containing formamide-treated CB.

The dielectric constant value depends on the number of mobile charge carriers,[13] and chemical treatments have been applied to CB to improve its electrical conductivity by increasing the number of mobile charge carriers.[12] The changes in the dielectric constant with the addition of both treated CB and UCB particles to PET at different filler concentrations are shown in Figures 13 and 14. As expected, the dielectric constant increases with the filler content increasing from 4 to 8 wt % and with the formamide solution concentration increasing from 1 to 3 wt %. Dielectric constant values of composites containing treated CB are higher than those of composites containing UCB at 8.5 and 10 GHz, and composites containing FA3–CB exhibit the highest dielectric constant values at both frequencies (Figs. 13 and 14). Although the dielectric constant decreases exponentially with increasing frequency,[19] dielectric values obtained at a frequency of 8.5 GHz are not so different from dielectric values measured at 10 GHz. Mobile charges form dipoles against the incident electric field. Meanwhile, at a high frequency, mobile charges do not have sufficient time to form dipoles and to absorb the energy of the incident electric field, which might result in low dielectric values.[18] Despite this negative effect of microwave frequency measurements, higher dielectric constant values have been obtained at low CB concentrations.[21]

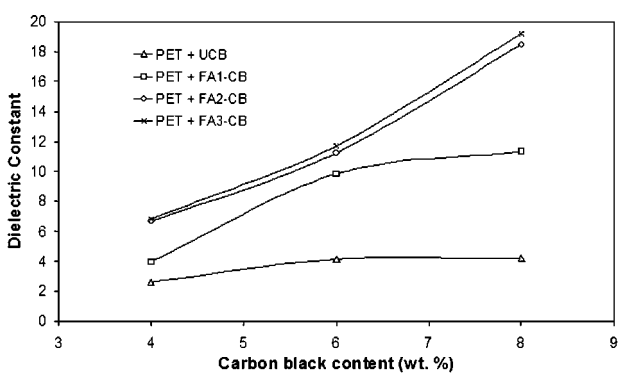

Figure 14 Dielectric constant values of the PET-based composites at 10 GHz.

To increase CB resolution in SEM micrographs, staining was applied to fractured test specimens for microwave measurements with nitric acid. SEM micrographs of the composites containing UCB and FA1–CB are not different from each other. CB agglomerates can be seen in both micrographs (Fig. 15). There is no distinct change at the submicrometer level in the distribution and size between UCB and formamide-treated CB. Hence, the surface treatment may not significantly affect the agglomerate size of CB and the distribution in the composites. On the other hand, PET and CB are compatible, and the surface treatment of CB with formamide makes the filler more compatible with PET because of more reactive groups on the surface of treated CB, as observed in ESCA analyses.[12] Thus, potential interactions between CB and PET upon a formamide treatment can affect the electrical and microwave properties of the composites.

CONCLUSIONS

The electrical resistivity of PET-based composites was successfully lowered more than one order of magnitude with the surface treatment of CB. The

percolation threshold concentration of composites containing the treated conductive filler shifted from 3 to 1.5 wt % CB. An increase in the formamide solution concentration resulted in an increase in the electrical conductivity of the composites. In addition, microwave properties such as the insertion loss and dielectric constant of composites including treated CB were improved when compared with those of composites filled with UCB. The shielding mechanism of the prepared composites was mainly insertion; the absorption and reflection mechanisms seemed to be less effective in the X-band range. The EMI shielding effectiveness and dielectric constant increased with the CB content and formamide solution concentration increasing. The composite containing 8 wt % FA3–CB was shown to exhibit EMI shielding effectiveness up to 27 dB and a dielectric constant value up to 19.

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