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The effect of multiwalled carbon nanotube (MWCNT) ratio on electrical properties and electromagnetic shielding effectiveness of PA 6/MWCNT-coated cotton fabrics

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Polyamide 6 (PA 6)/multiwalled carbon nanotube (MWCNT)-coated cotton fabrics having varying amounts of MWCNTs (10, 12, 14 and 16 wt.%) in PA 6 (10, 15 and 20 wt.%) were fabricated with dip coating method to investigate the electromagnetic shielding properties in the frequency range of 15–3000 MHz. The effects of MWCNT loading on electrical resistivity, electromagnetic interference shielding effectiveness and also shielding mechanism have been studied. The highest electromagnetic shielding effectiveness value was obtained at 20 wt.% MWCNT loading in 10 wt.% PA 6 and the PA 6/MWCNT-coated composites showed the absoprtion-dominated shielding mechanism.

Keywords: multiwalled carbon nanotube; polyamide 6; electrical resistivity; electromagnetic shielding effectiveness (EMSE)

Introduction

Electromagnetic interference (EMI) is one of the most unwanted by-products of rapid improvement of electronic products and telecommunications (Gupta & Choudhary, 2011). With the increasing use of electronic products and telecommunication equipment, EMI has become a major problem (Ortlek, Alpyildiz, & Kilic, 2012), because it reduces the lifetime and the efficiency of the instruments (Kim et al., 2003). Electromagnetic waves generated by computer circuits, radio transmitters, fluorescent lamps, electric motors, overhead power lines, lightning, etc. (Heiser, King, Konell, & Sutter, 2004; Roh, Chi, Tae Jin, & Nam, 2008), can cause serious problems, such as malfunction of medical equipment and industrial robots, and can even affect the human body (Im, Kim, Lee, & Lee, 2010) such as leukemia, miscarriages and breast cancer (Kim, Yun, Kim, & Lee, 2012). Therefore, efforts were made to reduce its effect using EMI shielding materials (Das, Chaki, Khastgır, & Chakraborty, 2001; Yildiz, Usta, & Gungor, 2012). Polymers are insulators electrically and do not prevent the passage of electromagnetic waves (Huang, Mo, & Roan, 2004). Electrically conducting polymer composites has received much attention recently compared to conventional metal-based EMI shielding materials (Huang et al., 2007), because of their light weight, resistance to corrosion, flexibility and processing advantages.

EMI shielding describes the reflection and/or absorption of electromagnetic radiation by a material, which thereby acts as a shield against the penetration of the radiation through it (Basuli, Chattopadhyay, Nah, & Chaki, 2012; Krueger & King, 2003). The shield should be in high conductance, thus metals, such as steel, copper, aluminum, etc., are the most common materials used for EMI shielding (Zhang, Ni, Fu, & Kurashiki, 2007). The metal-based materials have been widely used to reduce EMI. However, they have shortcomings due to the disadvantages of heavy weight and easy corrosion which restricted the wide use of these materials and they reflected the electromagnetic wave resulting in the secondary interference. As a result, the materials which can absorb the electromagnetic wave effectively are needed for EMI shielding efficiency (Yun, Im, Lee, & Kim, 2010). Among the various conductive fillers, CNTs have been shown to be an excellent candidate for EMI shielding due to small diameter, high aspect ratio, large surface area, high conductivity and high mechanical strength (Liu et al., 2007).

CNTs possess higher electrical conductivity than copper due to their low resistance and very few defects along their structure. The electrical resistivity of CNTs was found to be as low as 10^{-6} Ω-m and often can be altered by modifying the structure of the nanotube lattice (Dervishi et al., 2009).

The aim of this study is to investigate the effect of MWCNT amount and PA 6 concentration to the electromagnetic shielding effectiveness (EMSE) of cotton fabrics. Therefore, PA 6/MWCNT-coated cotton fabrics were fabricated with the dip coating method. The EMI shielding properties of PA 6/MWCNT coated fabrics

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were measured in the frequency range of 15–3000 MHz. Also, shielding mechanism of coated composites was studied.

Materials and methods

Materials

PA 6 pellets (Akulon K125, DSM, Holland, M_w ~ 35.000) and formic acid (98%, Merck, Germany) were used to a fabricate polymer-based coating solution. Multiwalled carbon nanotubes (MWCNT) were purchased from MK Nano, Canada. External diameters and length of MWCNTs are 10–20 and 10–30 μm, respectively. The purity was higher than 95%. MWCNTs were used without further modification. Figure 1 shows the SEM image and elemental analysis of as-received MWCNTs. From the elemental analysis, it is clearly observed that more than 99% of MWCNT contains carbon. The plain woven cotton fabrics were used in this study as matrix material. Warp density and weft density of cotton fabric are 26 warp/cm and 16 weft/cm, respectively. The basis weight of cotton fabric is 118.7 g/m^2.

Preparation of polymer-based coating solution

In this study, three different PA 6 solution concentrations (10, 15 and 20 wt.%) were determined. PA 6/MWCNT composite solutions (MWCNT content 10, 12, 14 and 16 wt.%) were prepared for each total concentration. The mixture formulas for each type of specimens are listed in Table 1. First, PA 6 pellets were dissolved in formic acid (98%) at 70°C for 2 h with a magnetic stirrer. Then the solution was cooled to room temperature and a known amount of MWCNT is added to the solution, ultrasonication (Branson Sonifier 250) was applied to the solution to create uniform dispersion with a frequency of 20 kHz for 2 h in an ice bath.

Table 1. Constituent materials and their mixture proportions.

	Weight composition[a] (wt.%)	
Denotations	PA 6	MWCNT
10-MWCNT10		10
10-MWCNT12		12
10-MWCNT14	10	14
10-MWCNT16		16
15-MWCNT10		10
15-MWCNT12		12
15-MWCNT14	15	14
15-MWCNT16		16
20-MWCNT10		10
20-MWCNT12		12
20-MWCNT14	20	14
20-MWCNT16		16

[a]Mass ratios of polyamide 6 weight.

Fabrication of PA 6/MWCNT-coated cotton fabrics

Dip coating, a convenient coating method, was used to apply PA 6/MWCNTs onto cotton fabrics. Cotton fabrics with the dimension of 25 × 25 cm were immersed in PA 6/MWCNT dispersion for 60 min, after that they were air-dried in fume hood overnight, then further dried in an oven at 60°C for 4 h to allow solvent evaporation.

Characterization

Surface resistivity measurements of PA 6/MWCNT-coated fabrics were tested based on ASTM D257-07 (2007) standard, using a two-probe method with Keithley 8009 Resistivity Test Fixture. The resistivity measurements of coated structures were tested with two different voltages (1 and 5 V). Surface resistivity is measured by applying a voltage potential across the surface of the specimen, measuring the resultant current and then performing the following calculation:

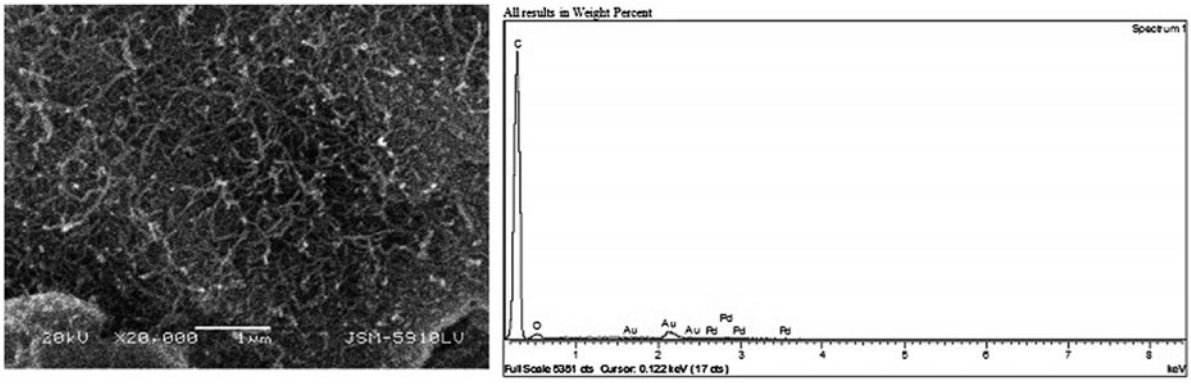


Figure 1. SEM micrographs and elemental analysis of MWCNTs.

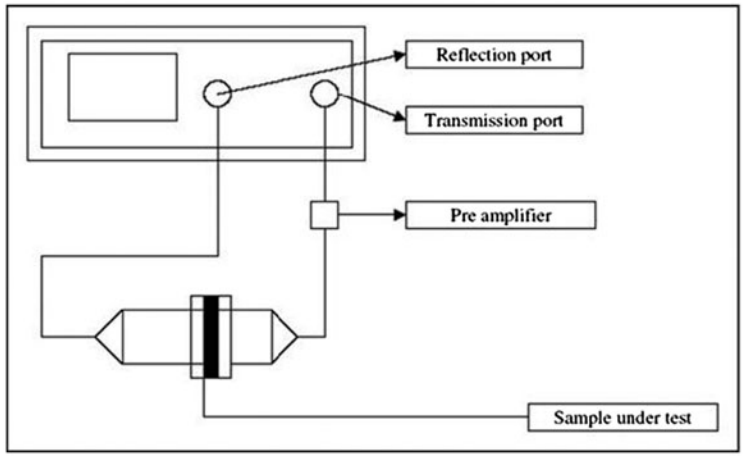


Figure 2. A typical coaxial transmitter for EMSE testing.

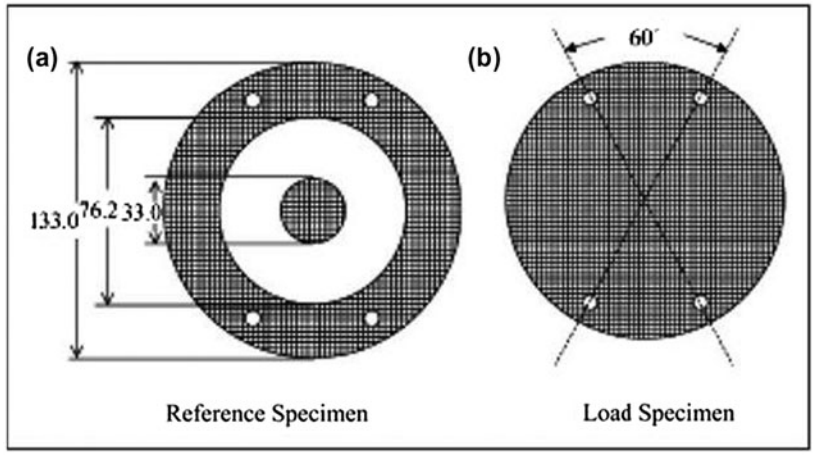


Figure 3. Specimen dimension for reference (left) and load test (right) ASTM D4935 (unit: mm).

$$\rho_s = \frac{53.4V}{I} \text{ ohm } (\Omega), \quad (1)$$

where ρ_s is the surface resistivity of the specimen, V is the applied voltage and I is the current reading from the instrument.

Electromagnetic shielding properties of coated fabrics were tested based on ASTM D4935-10 (2010) the coaxial transmission line method for planar materials standard (Figure 2). A shielding effectiveness test fixture (Electro-Metrics, Inc., model EM-2107A) was used to hold the sample with a network analyser generating and receiving the EM signals. This standard determined the shielding effectiveness of the textile structures using the insertion-loss method. The technique involved irradiating a flat, thin sample of the base material with an EM wave over the frequency range of interest, utilizing a coaxial and a flanged outer conductor. The frequencies used in the SE testing were between 15 and 3000 MHz.

At first, a gold-filmed reference specimen was used for the calibration. This calibration specimen shows certain SE values at certain frequencies, for instance it shows 32 dB SE value at 200 MHz. After calibration process, specimens are prepared with the standard test size of various thicknesses as shown in Figure 3. SE is determined by first taking a measurement of the received power with a reference specimen (Figure 3(a)), which is in the diameter of 133 mm of the outer ring in the test fixture. The load specimen (Figure 3(b)) is then fastened into the fixture and another received power measurement is taken. The SE in decibels is determined by using the following equation:

$$\mathrm{EMSE} = 10 \log \frac{P_1}{P_2}, \quad (2)$$

where P_1 is the received power with the load specimen and P_2 is the received power with the reference specimen present.

EMI shielding material can be classified on the basis of its dominant shielding mechanism, understanding of which led to its optimum use in terms of its effectiveness and cost. With any kind of EMI, there are three mechanisms contributing to the effectiveness of a shield. Part of the incident radiation is reflected from the front surface of the shield, part is absorbed within the shield material and part is reflected from the shield's rear surface to the front where it can aid or hinder the effectiveness of the shield depending on its phase relationship with the incident wave. Therefore, the total shielding effectiveness of a shielding material (SE) equals the sum of the absorption factor (A), the reflection factor (R) and the correction factor to account for multiple reflections (B) in thin shields:

$$\mathrm{SE} = R + A + B. \quad (3)$$

All the terms in Equation (3) are expressed in dB (Geetha, Satheesh Kumar, Rao, Vijayan, & Trivedi, 2009). Reflection depends on number of charge carriers or conductivity of the material; absorption depends on the presence of electrical or magnetic dipoles (Duan, Shunhua, & Hongtao, 2005), whereas multiple reflections are the total internal reflections within the material (Brzeziński et al., 2009).

Results and discussion

Surface morphology of the MWCNTs

Figure 4 shows the SEM micrographs of the MWCNTs before and after the ultrasonication process with formic acid. As seen from Figure 4(a), the raw MWCNTs are in the form of agglomerated bundles and have more fiber-like structure. These bundles are broken down into separate tubes and became shorter in length due to violent sonication during acid processing (Figure 4(b)). Treatment of MWCNTs with strong acids usually shortens the length of the MWCNTs (Singh, Singh, Mathur, & Dhami, 2008). Some researchs have shown that CNTs can be easily and seriously damaged, when a probe sonicator is used, if the sonication treatment is aggressive and long. Deformations and damages occured at the CNT surface affect the electrical properties of the CNT/polymer composites negatively (Garg et al., 2009; Lu et al., 1996; Ma, Siddiqui, Marom, & Kim, 2010).

Surface resistivity measurements

The resistivities of the PA 6/MWCNT-coated fabrics were controlled by changing the concentration of the MWCNT dispersions (Table 2). Figure 5 shows the effect of the PA 6/MWCNT dispersion concentration on the resistance of the coated fabrics at 1 and 5 V. The coated fabrics' electrical resistivity generally shows linear trend except from 15 wt.% PA 6. Figure 5(a) shows the surface resistivities of 10 wt.% PA 6-coated fabrics as a function of MWCNT loading at 1 and 5 V.

Table 2. Surface resistivity values of coated fabrics.

	Surface resistivity (Ω/sq)		Standard deviation	
Sample	1 V	5 V	1 V	5 V
10-MWCNT10	8.17E+07	3.99E+08	5.32E+05	2.10E+06
10-MWCNT12	4.68E+06	8.16E+05	1.45E+04	1.75E+04
10-MWCNT14	4.88E+06	1.13E+06	1.84E+05	7.72E+04
10-MWCNT16	1.89E+07	1.90E+06	2.39E+05	2.37E+05
15-MWCNT10	8.76E+05	3.23E+05	6.38E+04	8.11E+03
15-MWCNT12	8.22E+07	2.19E+08	1.94E+06	8.10E+06
15-MWCNT14	8.86E+05	2.13E+05	1.44E+04	7.00E+02
15-MWCNT16	3.58E+07	1.43E+07	1.28E+06	1.52E+06
20-MWCNT10	2.74E+07	2.23E+08	3.63E+07	1.02E+07
20-MWCNT12	6.14E+07	7.26E+07	8.98E+05	7.61E+06
20-MWCNT14	7.74E+07	1.09E+08	6.98E+05	4.70E+07
20-MWCNT16	8.44E+07	3.74E+08	3.46E+05	6.73E+06

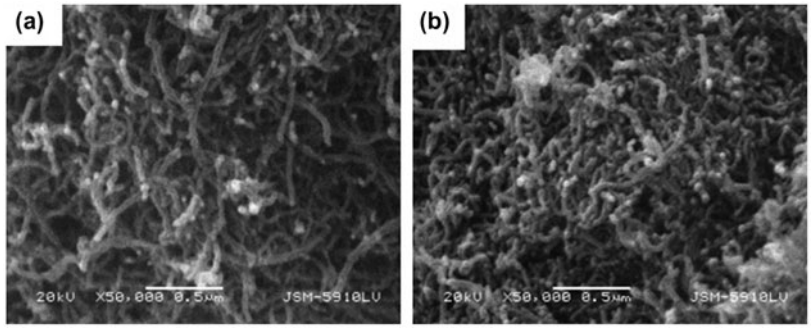


Figure 4. SEM images of MWCNTs (a) before sonication and (b) after 2 h sonication.

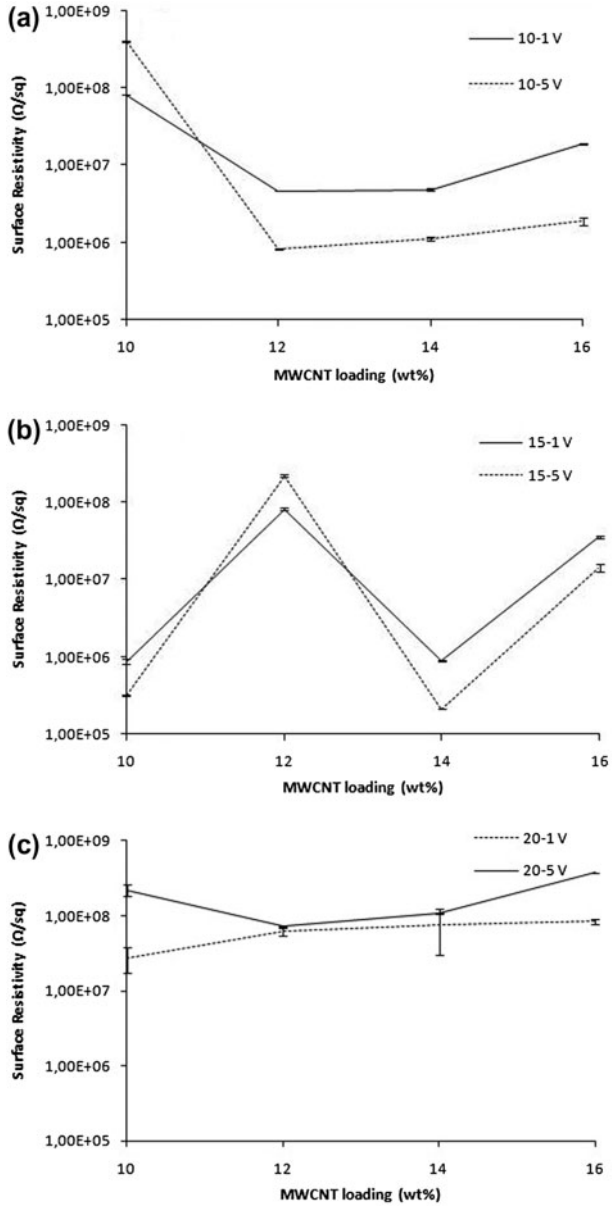


Figure 5. Surface resistivities of PA 6/MWCNT-coated fabrics at 1 and 5 V as a function of MWCNT loading (a) in 10 wt.% PA 6, (b) in 15 wt.% PA 6 and (c) in 20 wt.% PA 6.

It is clear that the surface resistivities of coated fabrics at 5 V decreased more than those of at 1 V with increasing of MWCNT loading. This decrease in electrical resistivity is due to increase in number of MWCNT networks. The electrical resistivity of fabrics starts to increase with further increasing of MWCNT content to 14 wt.%. Resistivity continued to rise at the maximum loading of MWCNT. When all the network connections of MWCNTs are formed, excessive loading of MWCNTs can increase the electrical resistivities of coated fabrics. From Figure 5(b), we can say that the resistivities of 15 wt.% PA 6/MWCNT-coated fabrics show unexpected nonlinear trend with an increase in MWCNT loading both at 1 and 5 V. The reason of this nonlinear trend is related to dispersion state of MWCNTs in polymer solution and the structural deformations occurred from the sonication process. Solution viscosity increased with increasing polymer concentration and the addition of MWCNTs also increased the viscosity. As a result of high viscosity of solution, it became difficult to disperse the MWCNTs homogeneously. Furthermore, the lowest electrical resistivity (2.13E+05) was obtained at 14 wt.% MWCNT in 15 wt.% PA 6 at 5 V. In Figure 5(c), the surface resistivities of 20 wt.% PA 6/MWCNT coated fabrics are given. It is observed that the resistivities of coated fabrics increased with an increase of MWCNT content and thus, the electrical conductivity decreased. The decrease in electrical conductivity is due to the increasing of insulating PA 6 network.

Surface resistivities of 10 and 15 wt.% PA 6/MWCNT-coated fabrics have generally low values at 5 V, on the other hand, surface resistivities of 20 wt.% PA 6/MWCNT-coated fabrics have higher values at 5 than 1 V. It can be said that when the amount of insulating material, PA 6, is increased, the resistivity of coated fabrics increases. Because the excessive amount of PA 6 blocks the MWCNT network at high voltages.

Electromagnetic shielding effectiveness of coated fabrics

Figure 6 shows the EMSE of PA 6/MWCNT-coated fabrics as a function of MWCNT loading in 10 wt.% PA 6 and the effect of frequency on EMSE of PA 6/MWCNT coated fabrics. The EMSE of coated fabrics is independent of frequency and increased linearly with increasing of MWCNT loading.

Figure 7 shows the EMSE of PA 6/MWCNT-coated fabrics as a function of MWCNT loading in 15 wt.% PA 6 and the effect of frequency on EMSE of PA 6/

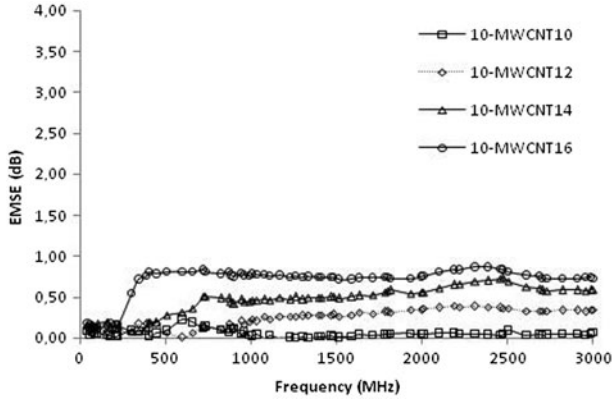


Figure 6. EMSE of PA 6/MWCNT-coated fabrics as a function of MWCNT content in 10 wt.% PA 6 at 15–3000 MHz.

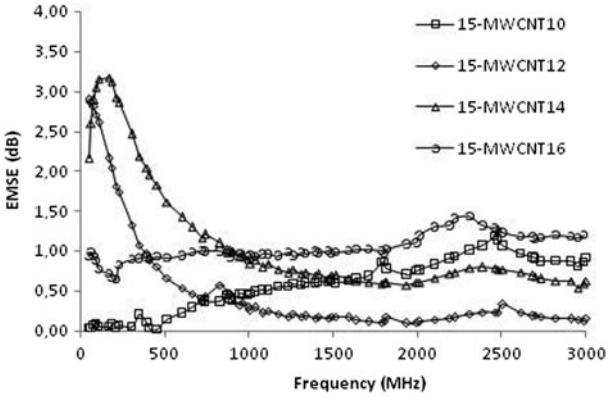


Figure 7. EMSE of PA 6/MWCNT-coated fabrics as a function of MWCNT content in 15 wt.% PA 6 at 15–3000 MHz.

MWCNT coated fabrics. The EMSE increased with increasing of frequency between 15–195 MHz, then decreased with increasing frequency to 300 MHz. At 14 wt.% MWCNT loading, the highest EMSE (3.19 dB) was obtained at low frequencies and the EMSE is dependent on frequency. At 12 wt.% MWCNT loading, the EMSE decreased much more than the other MWCNT loadings with increasing frequency. However, at a low frequency range, the EMSE of coated fabric is higher than that of 10 and 16 wt.% MWCNT loaded fabrics. The EMSE of 10 wt.% MWCNT loaded fabrics increased with increasing of frequency while the EMSE of 12 and 14 wt.% MWCNT loaded fabric is decreasing with the frequency. The EMSE of all coated fabrics do not show any significant change with frequency. The EMSE of 16 wt.% MWCNT loaded fabric has the highest value than the others at higher frequencies.

Figure 8 shows the EMSE of PA 6/MWCNT-coated fabrics as a function of MWCNT loading in 20 wt.% PA 6 and the effect of frequency on EMSE of PA 6/MWCNT-coated fabrics. The EMSE of all coated fabrics increased with increasing requency from 15 to 300 MHz. The EMSE of coated fabric is independent of MWCNT

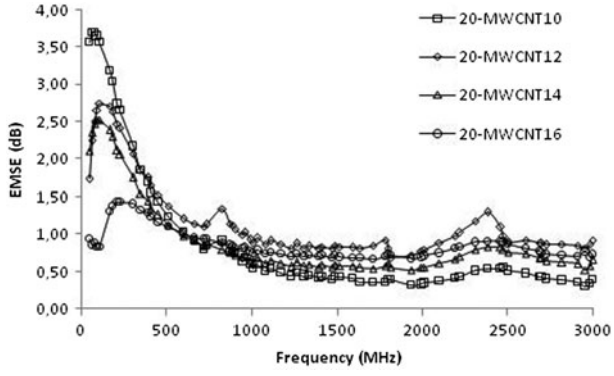


Figure 8. EMSE of PA 6/MWCNT-coated fabrics as a function of MWCNT content in 20 wt.% PA 6 at 15–3000 MHz.

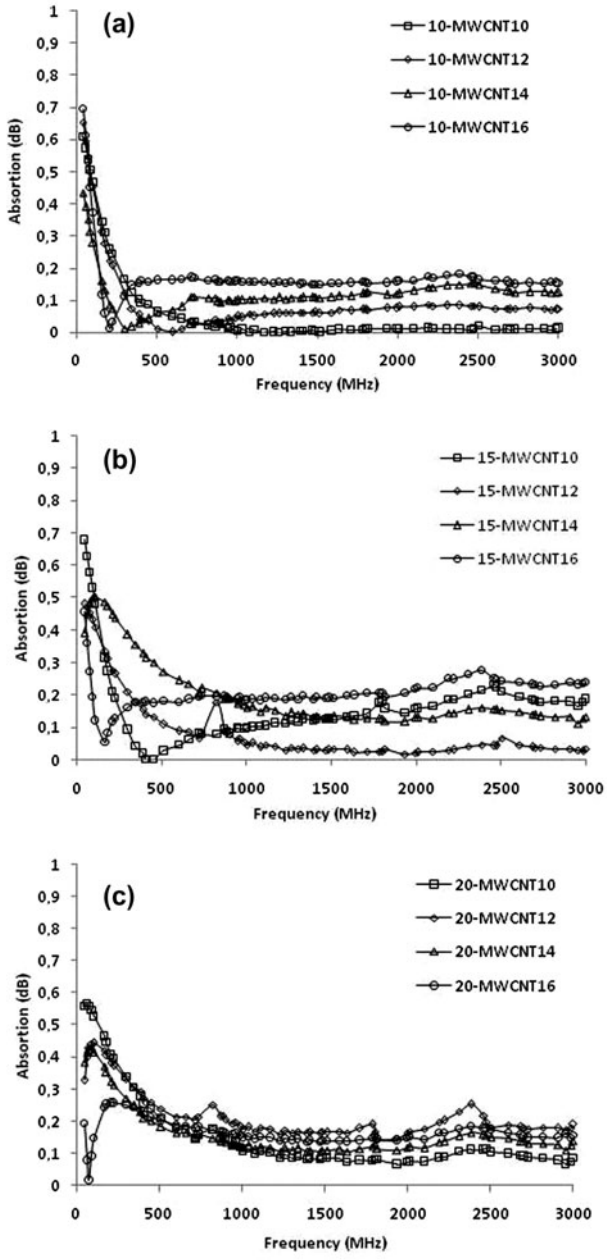


Figure 9. Absorptivities of PA 6/MWCNT-coated fabrics as a function of MWCNT loading (a) in 10 wt.% PA 6, (b) in 15 wt.% PA 6 and (c) in 20 wt.% PA 6.

loading at higher frequencies than 300 MHz. The highest EMSE (3.70 dB) was obtained at 10 wt.% MWCNT loading. At low frequencies, we can say that the EMSE of coated fabrics decreased with increasing MWCNT loading. After the highest shielding effectiveness values of 10, 12 and 14 wt.%, MWCNT-loaded fabrics were obtained, the SE decreased slightly with increasing frequency. However, the EMSE of 16 wt.% MWCNT-loaded fabric increased with increasing frequency, which has the lowest EMSE value of all coated fabrics. The

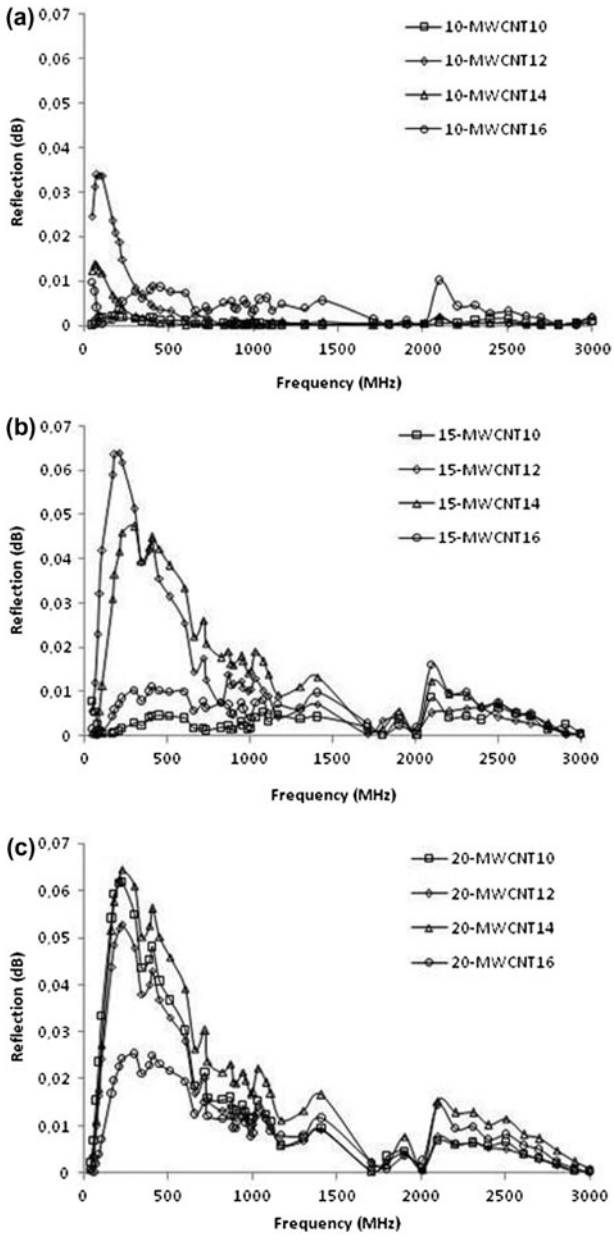


Figure 10. Reflectivities of PA 6/MWCNT-coated fabrics as a function of MWCNT loading (a) in 10 wt.% PA 6, (b) in 15 wt.% PA 6 and (c) in 20 wt.% PA 6.

EMSE of coated fabrics decreased slightly with increasing frequency. As is seen, the EMSE values of coated fabrics do not show significant change with frequency and MWCNT content. However, the highest SE (1.35 dB) was obtained at 12 wt.% MWCNT loading at high frequencies.

Absorption and reflection contributions of coated fabrics

Absorption and reflection mechanisms of coated fabrics were investigated as a function of MWCNT content between 15 and 3000 MHz frequency. Figure 9 shows the absorption mechanisms of PA 6/MWCNT-coated fabrics. All of three plots show that the highest absorption values were obtained at low frequencies and with increasing frequency, the absorption losses of coated fabrics exhibit noticeable decrease. In Figure 9(a), there is a relationship between increasing of absorption and MWCNT content. The absorption losses of coated fabrics increased linearly with increasing of MWCNT content and the highest absorption loss was obtained at 16 wt.% MWCNT loading. In Figure 9(b), at higher frequencies than 300 MHz, the absorption loss of 16 wt.%

MWCNT-loaded fabric has the highest value. Increasing of PA 6 and MWCNT content caused a decrease in absorption loss. However, at 12 wt.% MWCNT loading, relatively higher absorption loss values were obtained between the frequencies of 400 and 3000 MHz (Figure 9(c)).

Figure 10 shows the reflection mechanisms of PA 6/MWCNT-coated fabrics. As is seen in Figure 10(a), the reflections of coated fabrics are close at zero point and with the increase of PA 6 content, the reflection contribution increases slightly (Figures 10(b) and (c)). Relatively high values were obtained at lower frequencies, and with the increase of frequency, the reflections of coated fabrics decreased (Figures 10(b) and (c)).

With increasing MWCNT loading, both absorption and reflection increased, but rate of increase of absorption is much higher than reflection. Absorptions of coated fabrics are related to MWCNT content. As MWCNT content increases, the absorptions of coated fabrics increase. We can say that the electromagnetic shielding mechanism of coated fabrics is absorption dominated.

Conclusions

In this study, the electrical resistivity and EMSE of PA 6/MWCNT coated fabrics were investigated. There is an inverse ratio between the electrical resistivity and the electrical conductivity. The electrical resistivity measurements were done at two different voltages and 14 wt.% MWCNT loading in 15 wt.% PA 6 has the lowest electrical resistivity at 5 V. It can be clearly seen that the electrical resistivity values of coated fabrics are high at 5 V measurement. Electromagnetic shielding effectiveness of PA 6/MWCNT coated fabrics was studied between 15 and 3000 MHz. The highest EMSE was obtained (3.70 dB) at 10 wt.% MWCNT loading in 20 wt.% PA 6. We think that there are several reasons why the electromagnetic shielding properties of PA 6/MWCNT-coated fabrics are low. Electromagnetic shielding properties of coated fabrics can be affected by; (i) structural deformations resulting from the ultrasonication process on MWCNT surface, (ii) the non-uniform dispersion of MWCNTs with polymer solution, (iii) processing MWCNTs without further modification and functionalization. The electromagnetic shielding mechanism of PA 6/MWCNT-coated fabrics is absorption dominated.

As a result of the present study, it was suggested that the proposed MWCNT coatings were suitable electromagnetic shielding materials with the advantages of light weight, simple manufacturing process and low cost. Future works will focus on improving the electrical conductivity and electromagnetic shielding properties of CNT-incorparated materials.

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