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# Polyaniline/Silver Nanoparticle-Doped Multiwalled Carbon Nanotube Composites

Ali Grinou, Hyeonseong Bak, Young Soo Yun, and Hyoung-Joon Jin

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A simple method was used to synthesize the hybrid nanocomposites consisting of the functionalized multiwalled carbon nanotube composites (MWCNTs) with the polyaniline incorporated silver nanoparticles (a-MWCNT/PANI-Ag) through an emulsion polymerization at room temperature in order to enhance the electrical conductivity of polyaniline. The electrical conductivity of the composite with the incorporated Ag nanoparticles was 5% higher than the same weight percent for the composite without Ag nanoparticles, and the thermal stability was dramatically increased from 54% for the composite (a-MWCNT/PANI) to 69% through the incorporation of the Ag nanoparticles at 830°C. Additionally, the advantages of the Ag nanoparticles, including the improved electrical and thermal properties without damage to the polyaniline structure, were confirmed using FTIR and Raman spectroscopy.

**Keywords** Conductivity, emulsion polymerization, multiwalled carbon nanotubes, polyaniline, silver nanoparticles

## 1. INTRODUCTION

Carbon nanotubes (CNTs), which were discovered by Iijima,<sup>[1]</sup> have attracted considerable interest with respect to their applications in the fabrication of new classes of advanced materials because of their unique structural, thermal, and mechanical properties in terms of their stiffness, high Young's modulus, flexibility, and high electrical conductivity. These properties can be attributed to the high degree of organization and the high aspect ratio of the carbon nanotubes. However, pure CNTs cannot be processed and are difficult to dissolve or disperse in common organic solvents or polymeric matrices because of their chemical inertness and strong  $\pi$ - $\pi$  interactions. Therefore, the side walls of CNTs must be chemically modified in order to improve their dispersion or solubility in solvents or polymers.<sup>[2,3]</sup> Since the first report on the preparation of carbon nanotube/polymer composites by Ajayan et al.,<sup>[4]</sup> many efforts have been made to design and fabricate nanotube based composites with superior properties, such as high mechanical strength, flexibility, and electric conductivity.<sup>[5]</sup> Recently, many attempts have been conducted in order to deposit various metal particles onto the surface of the

CNTs for catalysis. Some metals and their compounds, such as Pt, Pd, Ag, Au, Ni, Fe, have successfully been deposited on the CNTs.<sup>[6]</sup> Among them, Ag nanoparticles (Ag-NPs) that are attached onto the CNTs (Ag-CNTs) have gained significant attention because their potential applications as catalysts,<sup>[7]</sup> optical limiters,<sup>[8]</sup> and advanced materials.<sup>[9]</sup> On the other hand, many studies have shown that conducting polymers with a porous structure and a high-surface area can usually be used as the matrix to incorporate noble metal catalysts.<sup>[10]</sup>

Conductive polymers have been intensively researched in different electrochemical devices, such as electrical conducting adhesives, sensors and actuators, antistatic coatings and films, electromagnetic interference shielding materials for electronic devices, thermal interface materials, etc.<sup>[11,12]</sup> Among the various conducting polymers, polyaniline (PANI) could potentially be used to synthesize polymer/CNT composites because of its environmental stability, good processability and reversible control of the conductivity both through protonation and charge-transfer doping.<sup>[13]</sup> PANI can be exploited in many applications, such as microwave absorbing and shielding materials, microporous electrically conducting materials, supporting materials for catalysts, etc.<sup>[14–16]</sup> The combination of PANI with the CNTs could produce an attractive composite support material for electrocatalysts that would enhance the activity and stability based on morphological modification or electronic interaction between these two components PANI and multiwalled carbon nanotube composites (MWCNTs).<sup>[17]</sup>

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In this study, the silver nanoparticles were incorporated into the a-MWCNT/PANI composites in order to increase the conductivity and thermal stability of the hybrid nanocomposites. A simple strategy was used to synthesize the hybrid nanocomposites consisting of the functionalized MWCNT with polyaniline that was reinforced with the silver nanoparticles through the emulsion polymerization at room temperature. Sodium dodecyl sulfate (SDS) was used as the anionic surfactant and polyaniline was doped with hydrochloric acid (HCl) in order to obtain a good dispersion of the CNTs in polyaniline and to improve the interfacial interactions between these components (a-MWCNT/PANI). Additionally, the silver nitrate solution was incorporated into the composite (a-MWCNT/PANI) after the polymerization using a dropwise method in order to avoid any agglomeration and to obtain a uniform dispersion on the entire polyaniline surface. Several characterization tools were employed in order to investigate the morphology and the structure of the composite (a-MWCNT/PANI-Ag).

## 2. EXPERIMENTAL

### 2.1. Materials

The MWCNTs (purity of 95% and a diameter of 10~15 nm, supplied by Hanwha Nanotech, Korea) were produced through thermal chemical vapor deposition (CVD) and used in this study. The aniline monomer was purchased from DC Chemical Co. (Korea), and ammonium persulfate (APS, 98%) was purchased from DAEJUNG Chemicals and Materials Co. (Korea). Sodium dodecyl sulfate (SDS) and all of the other organic solvents were used without any further purification.

### 2.2. Acid Treatment of MWCNT (a-MWCNT)

Typically, 1.0 g of raw MWCNTs was added to 200 mL of H<sub>2</sub>SO<sub>4</sub>/HNO<sub>3</sub> (3:1, v/v) and concentrated through ultra-sonication for 15 minutes. Then the solution was transferred into a 500 mL three neck flask, where it was magnetically stirred and heated at 65°C for 6 hours. After cooling to room temperature, the mixture was filtered and washed several times with distilled water until a pH of 7.0 was reached. The filtered solid was dried under vacuum for 6 hours at 60°C. This treatment provided carboxylic acid groups at the defect sites of the carbon nanotubes (MWCNT-COOH), which improved the solubility of the MWCNTs in organic solvents and water.

### 2.3. Synthesis of a-MWCNT/PANI-Ag Composites

The conducting a-MWCNT/PANI-Ag nanocomposites were synthesized through emulsion polymerization. In a typical synthesis experiment, various amounts of the acid treated MWCNTs were dispersed in 50 mL of the 0.2 M SDS solution and sonicated for 1 hour. The aniline

monomer (1.0 ml) was sonicated for 1 hour in 50 ml of a 1.0 M HCl solution. Then the mixture of the a-MWCNTs in the SDS solution were transferred into a double-walled glass reactor that was equipped with a mechanical stirrer at a reaction temperature of 60°C. Aniline doped HCl was added dropwise to the a-MWCNT solution in order to prevent the aggregation of the carbon nanotubes. Aniline was polymerized through the dropwise addition of 50 mL of 1.0 M aqueous HCl of APS (2.0 g). After polymerization for 2 hours, the silver nitrate solution was added to the mixture dropwise to prevent agglomeration and to obtain a uniform dispersion over the entire polyaniline surface. Then the reaction mixture was stirred for an additional 6 hours at 60°C to ensure that the nanocomposite was homogeneous. The product was filtered and rinsed several times with distilled water. The black powder was dried under a vacuum at room temperature for 24 hours.

### 2.4. Characterization

After pre-coating the samples with a homogeneous Pt layer through ion sputtering (E-1030, Hitachi, Japan), field-emission scanning electron microscopy (FESEM, S-4300SE, Hitachi, Japan) at an accelerating voltage of 15 kV was used to observe the morphology of the a-MWNT/PANI-Ag composites. Transmission electron microscopy (TEM, CM200, Philips, the Netherlands) was used to confirm the presence of the Ag nanoparticles in the a-MWCNT/PANI composite. FT-Raman, UV-visible, and FTIR spectroscopy were used to characterize the chemical structures of PANI, the a-MWCNTs, and the a-MWCNT/PANI and a-MWCNT/PANI-Ag composites. The thermogravimetric analysis (TGA, TA instruments, Q50, UK) was used to measure the thermal stability of PANI, the a-MWCNTs, and the a-MWCNT/PANI and a-MWCNT/PANI-Ag composites. The TGA was carried out at a heating rate of 10°C/min from 20 to 850°C under a dynamic nitrogen flow of 10 cm<sup>3</sup>/min. The electrical conductivities of the a-MWCNTs, and the a-MWCNT/PANI and a-MWCNT/PANI-Ag composites were measured using a four-probe method with an electrical conductivity meter (Hiresta-UP MCP-HT450, Mitsubishi Chemical, Japan). X-ray powder diffraction (XRD) was used to determine the crystallinity of PANI, a-MWCNT/PANI, and a-MWCNT/PANI-Ag nanocomposites using an x-ray diffractometer.

## 3. RESULTS AND DISCUSSION

Scanning electron microscopy (SEM) images of the composites (a-MWCNT/PANI) before and after incorporation Ag nanoparticles are given in Figure 1. The a-MWCNT/PANI composite exhibited a typical core-shell structure, where the a-MWCNTs served as the core and were individually dispersed into the PANI matrix. The aniline monomer was uniformly polymerized on the surface

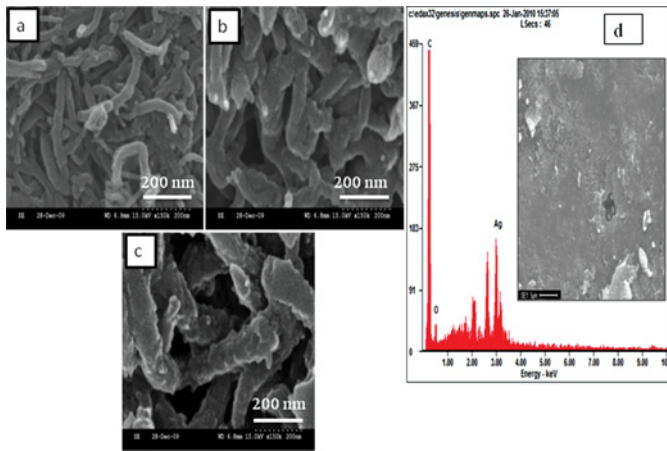


FIG. 1. SEM and EDAX analysis of the a-MWCNT/PANI composites: (a) a-MWCNT/PANI, (b) a-MWCNT/PANI-Ag 1 wt%, (c) a-MWCNT/PANI-Ag 2 wt%, and (d) a-MWCNT/PANI-Ag 2 wt%. (Figure available in color online.)

of the MWCNTs and formed the tubular shell of the a-MWCNT/PANI composites (Figure 1a). Compared to the average diameter in the composite (a-MWCNT/PANI), the diameters in the tubular composite after adding Ag nanoparticles are increased from 32.5 to 55 and 75 nm for the composites a-MWCNT/PANI-Ag 1 wt% and a-MWCNT/PANI-Ag 2 wt%, respectively, as shown in the (Figures 1b and 1c). These results confirm the dispersion of Ag nanoparticles in the composite (a-MWCNT/PANI). Furthermore, the tubes are almost uniform in diameter in the range of 70 to 75 nm after adding 2 wt% of Ag nanoparticles, confirm the uniformly polymerization of aniline monomer on the surface of a-MWCNT and the silver nanoparticles well dispersed in the composite (a-MWCNT/PANI). The presence of Ag nanoparticles in the composite (a-MWCNT/PANI) was analyzed using SEM/energy-dispersive x-ray (EDX) spectroscopy. As shown in Figure 1d, the EDX spectrum strongly revealed the presence of the Ag nanoparticles in the composite, which was also confirmed using XRD. Therefore, a good crystallinity for the composite (a-MWCNT/PANI-Ag) was obtained through the emulsion polymerization.

Figure 2 presents the TEM images of the a-MWCNTs, a-MWCNT/PANI and a-MWCNT/PANI-Ag composites. In Figure 2a, the formation of the carboxylic acid groups on the sidewalls of the carbon nanotubes was confirmed by FTIR spectroscopy. After adding the aniline monomer to the acid treated carbon nanotubes, the interaction corresponded to the  $\pi$ - $\pi^*$  electron interaction between the a-MWCNTs and the aniline monomer as well as the hydrogen bond interaction between the carboxyl groups of the a-MWCNTs and the amino groups of aniline monomers, which serves as the core and the self-assembly template, respectively, during the formation of the tubular

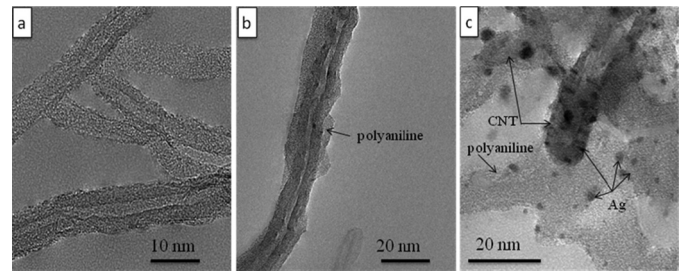


FIG. 2. TEM images of (a) a-MWCNTs, (b) a-MWCNT/PANI, and (c) a-MWCNT/PANI-Ag composites.

nanostructure (Figure 2b). The incorporation of the silver nanoparticles into the composite (a-MWCNT/PANI) is shown in (Figure 2c). The Ag nanoparticles were distributed with a spherical shape on the surface of the composites without agglomeration; the presence of these Ag nanoparticles was confirmed by X-ray diffraction (XRD).

The FTIR spectra were used to confirm the presence of the polymer (polyaniline) in the nanocomposites before and after the addition of the Ag nanoparticles. Figure 3 shows the FTIR spectra of the a-MWCNTs, pure PANI, a-MWCNT/PANI and a-MWCNT/PANI-Ag nanocomposites, respectively. For the acid treatment of MWCNT (Figure 3a), the absorption bands at 1717–1633, 1464–1384, and 1160–1022  $\text{cm}^{-1}$  corresponds to the carboxylic acid group C=O stretching, O-H bending and C-O stretching, respectively. For the pure PANI spectrum (Figure 3b), the bands at 1634 and 1582  $\text{cm}^{-1}$  were characteristic of the C=C stretching of the quinoid and benzenoid rings vibrations, respectively, indicating the oxidation state of polyaniline (emeraldine salt). The intensity of the quinonoid band was higher than the benzenoid band, revealing that PANI was richer in quinonoid units in these composites. The band at 1458  $\text{cm}^{-1}$  was assigned to the stretching of the C-N bonds of the aromatic amines. The strong band at 1118  $\text{cm}^{-1}$  was characteristic of the so-called

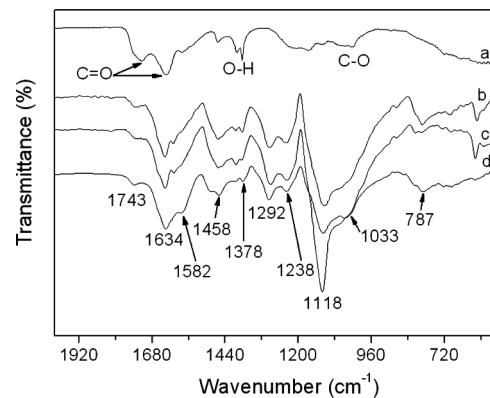


FIG. 3. FTIR spectroscopy of: (a) a-MWCNTs, (b) PANI, (c) a-MWCNT/PANI, and (d) a-MWCNT/PANI-Ag composites.

electronic-like band that was described by MacDiarmid et al.,<sup>[19]</sup> which was a measure of the degree of delocalization of the electrons and thus a characteristic peak of the PANI conductivity. The spectra of a-MWCNT/PANI and a-MWCNT/PANI-Ag nanocomposites (Figure 3c and d) were almost identical to pure PANI, indicating the successful polymerization on the surface of the MWCNTs.<sup>[20]</sup> For the composite a-MWCNT/PANI-Ag, a band absorption appear at  $1033\text{ cm}^{-1}$  corresponding to the interaction between polyaniline and the silver nanoparticles, and the other bands were nearly identical to pure polyaniline, indicating that presence of the silver nanoparticles on the polyaniline surface did not damage the structure of the composite that was obtained.

Figure 4 shows the Raman spectra of a-MWCNTs, PANI, a-MWCNT/PANI, and a-MWCNT/PANI-Ag, respectively. For comparison purposes, this figure included the spectrum of the a-MWNTs, which contained two strong peaks at  $1601$  and  $1285\text{ cm}^{-1}$ . For the a-MWCNT/PANI and a-MWCNT/PANI-Ag composites, C-H bending of the quinoid ring at  $1173\text{ cm}^{-1}$ , C-H bending of the benzenoid ring at  $1268\text{ cm}^{-1}$ , C-N<sup>+</sup> stretching at  $1372\text{ cm}^{-1}$ , and C-C stretching of the benzene ring at  $1507$  and  $1595\text{ cm}^{-1}$  were observed, revealing the presence of the doped PANI structure.<sup>[18]</sup> These Raman spectra were almost identical to pure PANI, indicating that the a-MWCNTs served as the core in the formation of the tubular shell of the a-MWCNT/PANI composites, therefore, the polymerization was successful on the surface of the MWCNTs, and the incorporation of the silver nanoparticles did not damage the polyaniline structure. The intensity of the bands changed, and their wave number slightly shifted in the spectra of the nanocomposites compared to pure polyaniline, indicating a good interaction between polyaniline and the a-MWCNTs.

The UV-visible (UV-vis) absorption spectra of a-MWCNTs, pure PANI, a-MWCNT/PANI and a-MWCNT/PANI-Ag nanocomposites are shown in

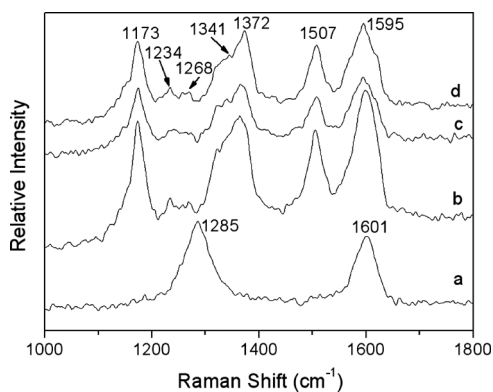


FIG. 4. Raman spectroscopy of (a) a-MWCNTs, (b) PANI, (c) a-MWCNT/PANI, and (d) a-MWCNT/PANI-Ag.

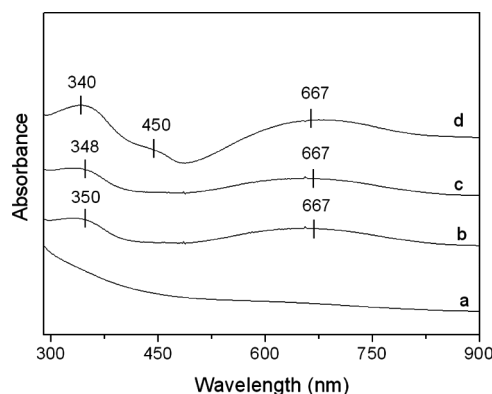


FIG. 5. UV-vis spectra of (a) a-MWCNTs, (b) PANI, (c) a-MWCNT/PANI, and (d) a-MWCNT/PANI-Ag.

Figure 5, respectively. No absorption bands were observed for the a-MWCNTs (Figure 5a). The absorption spectrum of the a-MWCNT/PANI-Ag system displayed three absorption bands at 340, 450 and 667 nm (Figure 5d), whereas the spectra of PANI and the a-MWCNT/PANI nanocomposite (Figure 5b and c) exhibited two absorption peaks at 350 and 667 nm. The absorption peak at around 350 nm (350 nm for PANI and 348 nm for the a-MWCNT/PANI composite) was assigned to the  $\pi$ - $\pi^*$  transition of doped polyaniline. This polaron- $\pi$  transition on the polyemeraldine chain was slightly shifted to a smaller wavelength for the a-MWCNT/PANI composite, revealing the interaction between the quinoid rings of PANI and the a-MWNTs.<sup>[21]</sup> Additionally, the broad band at 667 nm corresponded to a bipolaronic transition of PANI.<sup>[22]</sup> For the a-MWCNT/PANI-Ag composite, another peak appeared at 450 nm, corresponding to the surface plasmon resonance of the silver nanoparticles that were embedded in the polymer matrix.<sup>[23]</sup> These results confirmed the interaction between polyaniline and the silver nanoparticles.

Figure 6 shows the TGA results for the a-MWCNTs, pure PANI, a-MWCNT/PANI and a-MWCNT/PANI-Ag nanocomposites. The typical three-step weight-loss behavior was observed. In the first step, from approximately  $10$ – $200^\circ\text{C}$ , water molecules were lost from the composite structure. The second weight loss in the range from  $200$ – $460^\circ\text{C}$  was attributed to the loss of the oligomers and the dopants. The third weight loss above  $460^\circ\text{C}$  was caused by the degradation and the decomposition of the composites. According to the TGA analysis results, the weight percent of polyaniline and the a-MWCNTs in the composite (a-MWCNT/PANI) was calculated using residues at  $800^\circ\text{C}$ . The composite contained 44 wt% of the carbon nanotubes and 56 wt% of PANI. The TGA measurements of pure PANI, a-MWCNTs, a-MWCNT/PANI, and a-MWCNT/PANI-Ag nanocomposites are displayed in

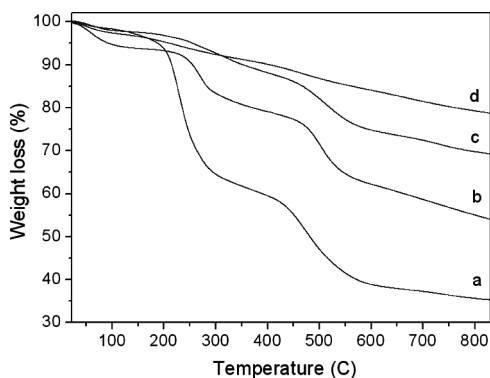


FIG. 6. TGA curves of the (a) PANI, (b) a-MWCNT/PANI, (c) a-MWCNT/PANI-Ag, and (d) the a-MWCNTs.

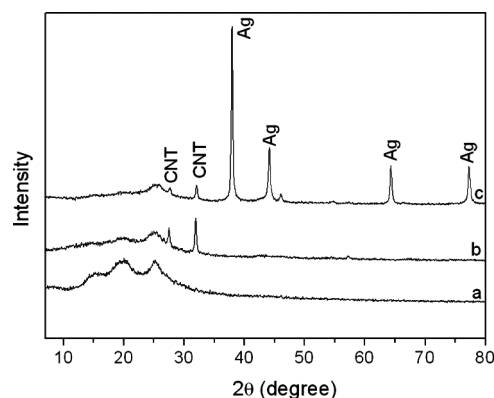


FIG. 7. XRD patterns of (a) pure PANI, (b) a-MWCNT/PANI, and (c) a-MWCNT/PANI-Ag nanocomposites.

Table 1. The thermal stability of the nanocomposite was improved through the incorporation of the a-MWCNTs and the Ag nanoparticles in the polyaniline matrix. The thermal stability dramatically increased from 36% for pure polyaniline to 54.14% for the composite (a-MWCNT/PANI) and 69.47% after the incorporation of the Ag nanoparticles at 830°C, because of the reduced mobility of the PANI chains in the nanocomposite. The chain transfer reaction was suppressed because of the reduce chain mobility. Consequently, the degradation process was slowed and the decomposition took place at higher temperatures.<sup>[24]</sup> The improved thermal stability of the nanocomposite was attributed to the physical interaction between the Ag nanoparticles and the PANI chains.

Figure 7 shows the XRD patterns of pure polyaniline (PANI), a-MWCNT/PANI and a-MWCNT/PANI-Ag nanocomposites. For pure PANI, the crystalline peaks appeared at  $2\theta = 15.16^\circ$ ,  $19.50^\circ$ , and  $25.16^\circ$ , corresponding to the (011), (020), and (200) crystal planes of PANI in its

emeraldine salt form, respectively.<sup>[25]</sup> For the composite (a-MWCNT/PANI) shown in (Figure 7b), the x-ray pattern exhibited both the characteristic peaks of PANI and the MWCNTs, indicating that the composite (a-MWCNT/PANI) was successfully synthesized through the interaction between the aniline monomer and the carboxylic acid on the side walls of the carbon nanotubes. For the composite with the Ag nanoparticles (a-MWCNT/PANI-Ag) shown in (Figure 7c), many crystalline peaks corresponding to the composite (a-MWCNT/PANI) and the silver nanoparticles were observed, confirming the successfully incorporation of the silver nanoparticles into the a-MWCNT/PANI composite. The crystalline peaks for silver nanoparticles appeared at  $2\theta = 37.87^\circ$ ,  $44.23^\circ$ ,  $64.39^\circ$ , and  $77.42^\circ$  (Figure 7c), which corresponded to the face-centered cubic (fcc) phase of silver (200), (111), (220), and (311), respectively.<sup>[26]</sup> The sharp diffraction patterns clearly indicated the existence of the silver nanoparticles in the composites (a-MWCNT/PANI) and their crystalline nature.

The electrical conductivity (data measured using four-point probe technique) values of the a-MWCNT/PANI and a-MWCNT/PANI-Ag nanocomposites are summarized in Table 2. The incorporation of the high conductivity Ag nanoparticles into the a-MWCNT/PANI composite increased the conductivity value of the composite. In Table 2, the electrical conductivity of the composite (2.0 wt% a-MWCNT/PANI) was  $3.48 \times 10^{-1}$  S/cm, and after the incorporation of the Ag nanoparticles at the same weight percent of the composite (a-MWCNT/PANI), the electrical conductivity dramatically increased to 1.685 S/cm. The incorporation of the silver nanoparticles on the surface of the carbon nanotube coated polyaniline greatly affected the electrical conductivity of the composite a-MWCNT/PANI because of the high electrical conductivity of the silver nanoparticles ( $\sigma_{Ag} = 6.30 \times 10^5$  S/cm) and the crystalline structure of the composite after the

TABLE 1  
Weight-loss of the a-MWCNTs, PANI, a-MWCNT/PANI, and a-MWCNT/PANI-Ag nanocomposites at different temperatures

Samples	TGA		
	First weight loss [10–200°C]	Second weight loss [200–460°C]	Third weight loss [above 460°C]
a-MWCNT	5	11	20
Pure PANI	6	46	64
a-MWCNT/PANI	7	23	46
a-MWCNT/PANI-Ag	3	15	31

TABLE 2

Data that was measured according to the standard four-point probe technique

Samples	Conductivity (S cm <sup>-1</sup> )
0.5 wt% a-MWCNT/PANI	$8.25 \times 10^{-2}$
1.0 wt% a-MWCNT/PANI	$2.70 \times 10^{-1}$
2.0 wt% a-MWCNT/PANI	$3.48 \times 10^{-1}$
5.0 wt% a-MWCNT/PANI	$4.37 \times 10^{-1}$
10.0 wt% a-MWCNT/PANI	$4.53 \times 10^{-1}$
2.0 wt% a-MWCNT/PANI-Ag	1.685

addition of the silver nanoparticles through the interaction between the Ag nanoparticles and PANI. Based on these results, the composite (a-MWCNT/PANI) was successfully synthesized and the Ag nanoparticles were incorporated into this composite, increasing the conductivity compared to the original composite (a-MWCNT/PANI).

#### 4. CONCLUSIONS

The polyaniline incorporated silver nanoparticles/functionalized carbon nanotubes composites were successfully synthesized using the emulsion polymerization technique. The covalent functionalization of the MWCNT with PANI ensured the compatibility of the carbon nanotubes in the polyaniline matrix, and consequently, the composite (a-MWCNT/PANI) exhibited a higher conductivity than pure PANI. The incorporation of the Ag-nanoparticles into the composite a-MWCNT/PANI significantly changed the thermal and electrical properties. For example, the thermal stability dramatically increased from 54% for the composite (a-MWCNT/PANI) to 69% after the incorporation of the Ag nanoparticles at 830°C. The four-point probe investigations revealed that the electrical conductivity of the composite with the Ag nanoparticles (a-MWCNT/PANI-Ag) increased 5% compared to the same weight percent of the composite (a-MWCNT/PANI) without the Ag nanoparticles. The FTIR and Raman spectroscopy studies confirmed that the polymerization was successful on the surface of the MWCNT, and the incorporation of the silver nanoparticles did not damage the polyaniline structure. The formation of the Ag nanoparticles and their existence in the prepared nanocomposites were confirmed using UV-vis spectroscopy, the EDX and XRD analysis. Additionally, the spectral characterization using UV-vis exhibited the surface plasmon resonance of the silver nanoparticles that were embedded in the polymer matrix at 450 nm, confirming the interaction between polyaniline and the silver nanoparticles.

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