Effect of multi-walled carbon nanotube dispersion on the electrical and rheological properties of poly(propylene carbonate)/poly(lactic acid)/multi-walled carbon nanotube composites

Dong Hyup Park · Tae Gyu Kan · Yun Kyun Lee · Woo Nyon Kim

Abstract In this study, the morphological, electrical, and rheological properties of the poly(propylene carbonate) (PPC)/poly(lactic acid) (PLA)/multi-walled carbon nanotube (MWCNT) composites were investigated. From the results of transmission electron microscopy of the PPC/PLA/MWCNT composites, the MWCNT preferred to locate more in the PPC phase than in the PLA phase. This maybe due to the lower interfacial tension of the PPC/MWCNT composites compared to that of the PLA/MWCNT composites. The electrical conductivities of the PPC/PLA/MWCNT composites were higher than those of the PPC/MWCNT and the PLA/MWCNT composites, which was likely due to the selective localization of the MWCNT in the PPC phase (continuous phase). From the results of the complex viscosity of the composites, the ratio of increasing the complex viscosity of the PPC/MWCNT composites with the MWCNT content was higher than that of the PLA/MWCNT composites. This is maybe due to the fact that the MWCNT dispersion in the PPC phase was higher than in the PLA phase. The results from the morphology, electrical conductivity, and complex viscosity of the PPC/PLA/MWCNT composites suggest that the selective localization of the MWCNT in the PPC phase can improve the conductive path and increase the electrical conductivity of the PPC/PLA/MWCNT composites.

Introduction

Poly(propylene carbonate) (PPC) is a new thermoplastic polymer derived from carbon dioxide and propylene oxide

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[1]. It is also a bio-degradable polymer and many previous studies have shown that its molecular weight decreases in specific solutions [2]. It also has interesting physical and chemical properties and is an attractive green environmental material for many applications [3–5]. This polymer has been used as medical materials and in food packaging because it utilizes CO_2 and shows good processability, providing transparent films with excellent oxygen barrier performance. However, PPC is amorphous with a low glass transition temperature ($T_g \approx 40$ °C) due to the weak interchain interaction of this copolymer. Thus, this plastic can only be used between the temperatures of 15 and 40 °C, since below 15 °C, it becomes brittle and above 40 °C, it quickly loses strength. Therefore, the mechanical performance of PPC requires improvement.

Poly(lactic acid) (PLA) is aliphatic polyester with high biodegradability and good mechanical properties, such as tensile strength, for industrial applications [6–10]. However, PLA has limited application because of its brittleness and low heat distortion temperature. Polymer blending is an efficient and economic method to tailor new materials through an advantageous combination of end-use properties [10, 11]. Blending PLA with other polymers presents a practical and economic approach to obtain toughened products [12]. Blending PLA with other bio-degradable polymers is a particularly interesting strategy to retain the integrity of biodegradability.

In this study, we blended PPC with PLA to improve the mechanical property of the material and mixed the PPC/PLA blends with multi-walled carbon nanotube (MWCNT) to improve the electrical property. Many academics and industries have recently extensively investigated polymer/CNT composites [13–25]. After the structure of carbon nanotubes was discovered by Iijima [17], CNTs have become a very attractive research source because of their

Table 1 Characteristics of the polymer samples used in this study

Sample	M_n	M_w	T_g (°C)	T_m (°C)
PPC[a]	61,700	142,300	40	–
PLA[b]	82,000	159,700	69	169

[a] Supplied by SK Innovation, South Korea

[b] Supplied by NatureWorks, USA

prominent properties, such as mechanical, electrical, physical, and chemical properties. However, it is difficult to use CNT as a reinforced filler in a polymer composite, because CNTs generally agglomerate in the polymer matrix, like a bundle. So dispersion of CNTs in the polymer matrix is very important.

Localization of the conductive filler depends on many factors [26], such as the affinity of the conductive filler to each polymer, interfacial tension, mixing sequence, mixing time, and viscosity of the two polymers and can be predicted thermodynamically by interfacial tension using Sumita's model [27]. For example, Gubbels et al. [28] found that the electrical resistivity of carbon black (CB)-filled polyethylene/polystyrene blends changed as a function of mixing time, corresponding to different localizations of CB, where the lowest electrical resistivity was observed when CB was localized at the interface.

In this study, we investigated the effect of MWCNT localization on the electrical, morphological, and rheological properties of PPC/PLA/MWCNT composites. The electrical properties of the PPC/PLA/MWCNT composites were measured using the four-probe method and the morphological and rheological properties of the PPC/PLA/MWCNT composites were measured using scanning electron microscopy, transmission electron microscopy and an advanced rheometric expansion system (ARES).

Experimental

Materials

The materials used in this study were obtained from commercial sources. The PPC was supplied by SK Innovation Ltd (Korea). The PLA was supplied by NatureWorks Co. (Blair, NE, USA). The number and weight
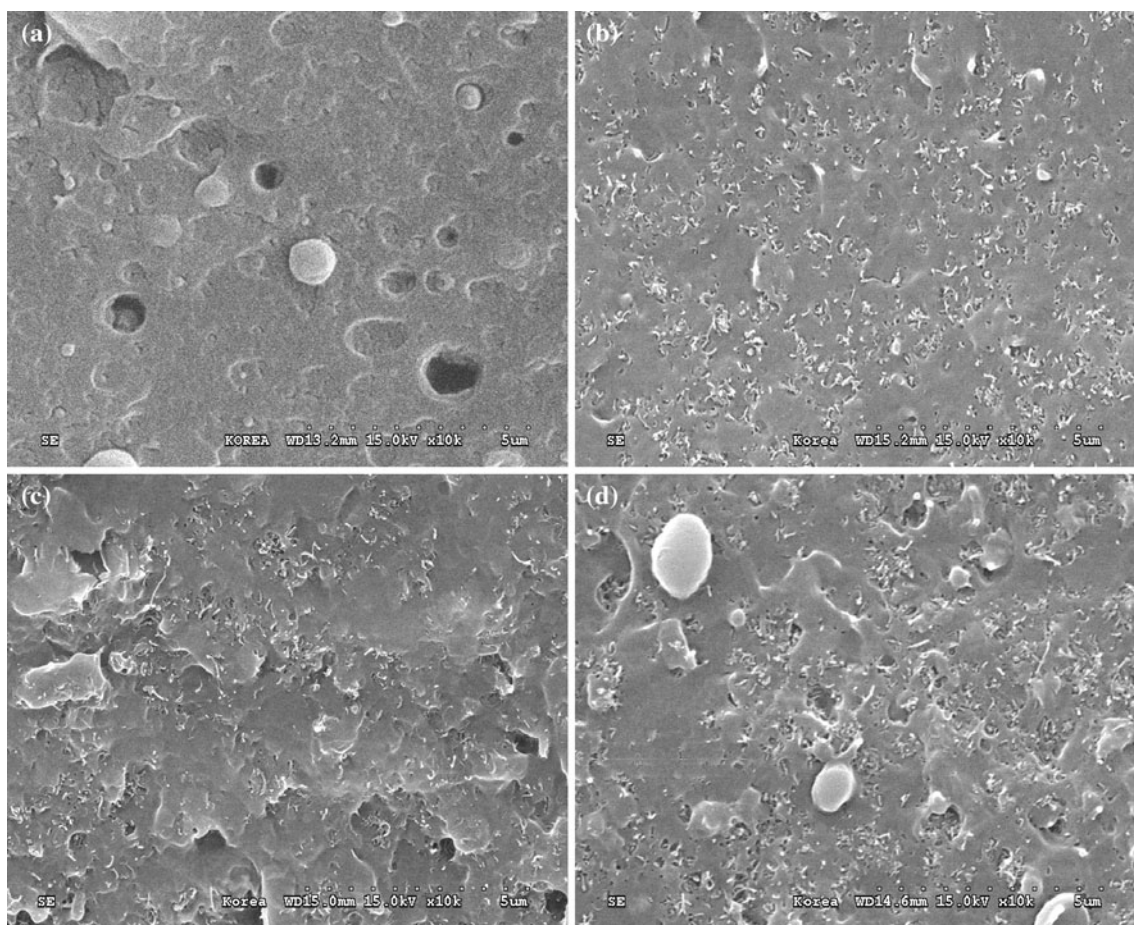
Fig. 1 Scanning electron micrographs of **a** poly(propylene carbonate) (PPC) and poly(lactic acid) (PLA) (70/30), **b** poly(lactic acid) (PLA)/MWCNT (3 phr), **c** poly(propylene carbonate) (PPC)/MWCNT (3 phr), and **d** PPC/PLA (70/30) with MWCNT (3 phr) composites
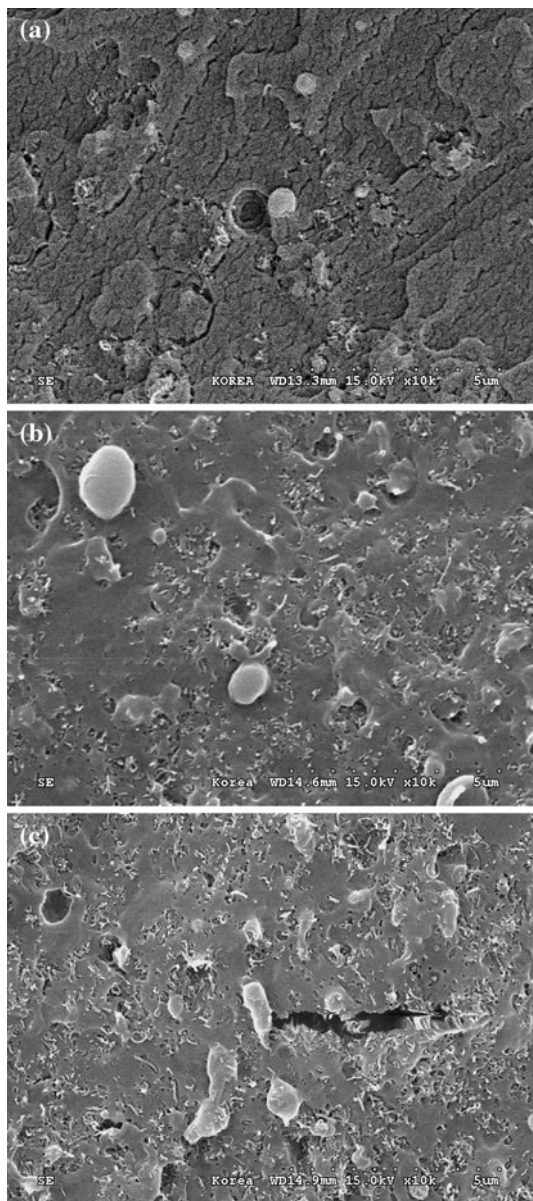
Fig. 2 Scanning electron micrographs of poly(propylene carbonate) (PPC) and poly(lactic acid) (PLA) (70/30) with MWCNT composites: **a** 1 phr, **b** 3 phr, and **c** 5 phr

average molecular weights of the PPC and PLA were 61,700 and 142,300, and 82,000 and 159,700, respectively. MWCNTs were synthesized by the chemical vapor-grown method, supplied by Jeio Ltd., and had diameters ranging from 9 to 12 nm and lengths ranging from 10 to 15 μm. The characteristics and sources of the PPC and PLA are shown in Table 1.

Composite preparation

Composites of PPC and PLA (70/30, wt%) with the MWCNT in the compositions of 1, 3, and 5 phr (parts per

hundred PPA and PLA by weight) were melt mixed using a bench mixer (PBV-03, Inie Shokai Co.). The barrel temperature was 180 °C, and the rotation speed was 30 rpm for 10 min. Before melt mixing the PLA, PPC, and MWCNT were dried in a vacuum oven at 50 °C for 24 h. The blends were pressed at 180 °C in a hot press to obtain sheets.

Morphology

The morphology of the PPC/PLA/MWCNT composites was examined using a S-4300SE field emission (SEM) (Hitachi, Tokyo, Japan). Samples were cryogenically fractured and coated with gold before scanning. The accelerating voltage was 25 kV. A transmission electron microscope (FEI Tecnai 20, Eindhoven, The Netherlands) was used to examine the MWCNT dispersion in the PPC/PLA blends. The sectioning thickness was around 100 nm, and the samples were prepared using the ultramicrotome technique (Power-Tome PC Ultramicrotome, Boeckeler Instruments, Inc.) at the appropriate temperature for TEM observation.

Electrical properties

Electrical conductivity of the PPC/PLA/MWCNT composites was measured by the four-probe method using a digital multimeter (Keithley, Model 2000 multimeter) to eliminate contact resistance. Samples for the electrical conductivity measurements were prepared using hot press molding, and the samples were cut to a dimension of $15 \times 10 \times 0.2$ mm^3. Four thin, gold wires (0.05-mm thick and 99 % gold) were attached in parallel to the samples with conductive graphite paint [29].

Mechanical properties

The tensile strength of the PPC/PLA/MWCNT composites was studied at room temperature using a universal testing machine (Instron 4467) according to the ASTM D-638. The crosshead speed was 5.0 mm/min. In the tensile strength test, five specimens were measured and averaged.

Rheological properties

Dynamic measurements of the rheological properties were carried out using an ARES. Frequency sweeps from 0.1 to 100 rad/s were performed at 180 °C under dry nitrogen conditions. For all measurements, the PPC/PLA/MWCNT samples were tested within the linear viscoelastic strain range.
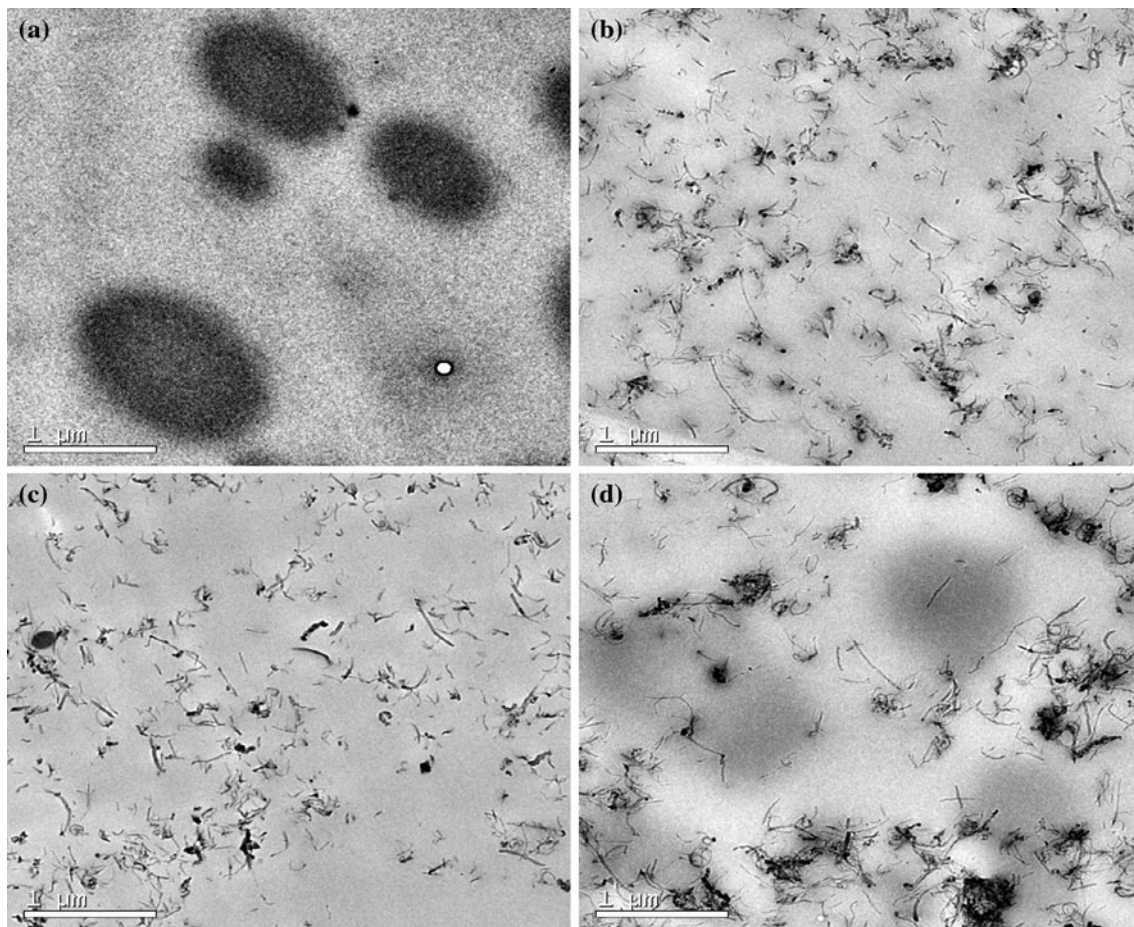
Fig. 3 Transmission electron micrographs of **a** poly(propylene carbonate) (PPC) and poly(lactic acid) (PLA) (70/30), **b** poly(lactic acid) (PLA)/ MWCNT (3 phr), **c** poly(propylene carbonate) (PPC)/MWCNT (3 phr), and **d** PPC/PLA (70/30) with MWCNT (3 phr) composites

Results and discussion

Morphology of the PPC/PLA/MWCNT composites

Figure 1a, b, c, and d shows the scanning electron micrographs of the PPC/PLA (70/30, wt%) blends with MWCNT (3 phr). As shown in Fig. 1a, the droplet size of the PLA was observed to be 0.89 μm. After adding the MWCNT (3 phr) in the PPC/PLA blends shown in Fig. 1d, the droplet size did not seem to change significantly. Figure 1b and c shows the SEM images of the PPC/MWCNT and PLA/ MWCNT composites, respectively. Figure 2a–c shows the scanning electron micrographs of the PPC/PLA (70/30, wt%) blends with the MWCNT of 1, 3, and 5 phr, respectively. The MWCNT seems to disperse evenly in the PPC and PLA phases; however, the localization of MWCNT in the polymer blends could not be analyzed based on the SEM images. Therefore, we used a TEM to assess the localization of the MWCNT in the PPC/PLA blends.

Figure 3 shows the transmission electron micrographs of the PPC/PLA (70/30, wt%) blends with MWCNT (3 phr).

In Fig. 3a, the droplet (dark circle) shows the PLA phase in the PPC/PLA (70/30) blends and the droplet size was about 0.85 μm. Figure 3b, c shows the TEM images of the PLA/ MWCNT and PPC/MWCNT composites, respectively, and the MWCNT seems to be dispersed evenly throughout the PPC and PLA phases. For the PPC/PLA/MWCNT composites shown in Fig. 3d, the MWCNT appears to be located more in the PPC phase, which is the continuous phase, than in the PLA phase, which is the domain phase (dark circle). However, agglomeration of the MWCNTs is found in Fig. 3d compared to Fig. 3c, and this is maybe due to that the MWCNTs can be agglomerated when they tends to avoid locating in a certain phase. Conductive fillers tend to prefer to locate in one phase that has a lower interfacial tension between the polymer and conductive filler [27]. Figure 4a, b, and c shows the transmission electron micrographs of the PPC/PLA (70/30, wt%) blends with the MWCNT of 1, 3, and 5 phr, respectively. The MWCNT appears to be located more in the PPC phase than in the PLA phase (dark circle); however, some of the MWCNTs are found in the PLA phase. The above result of
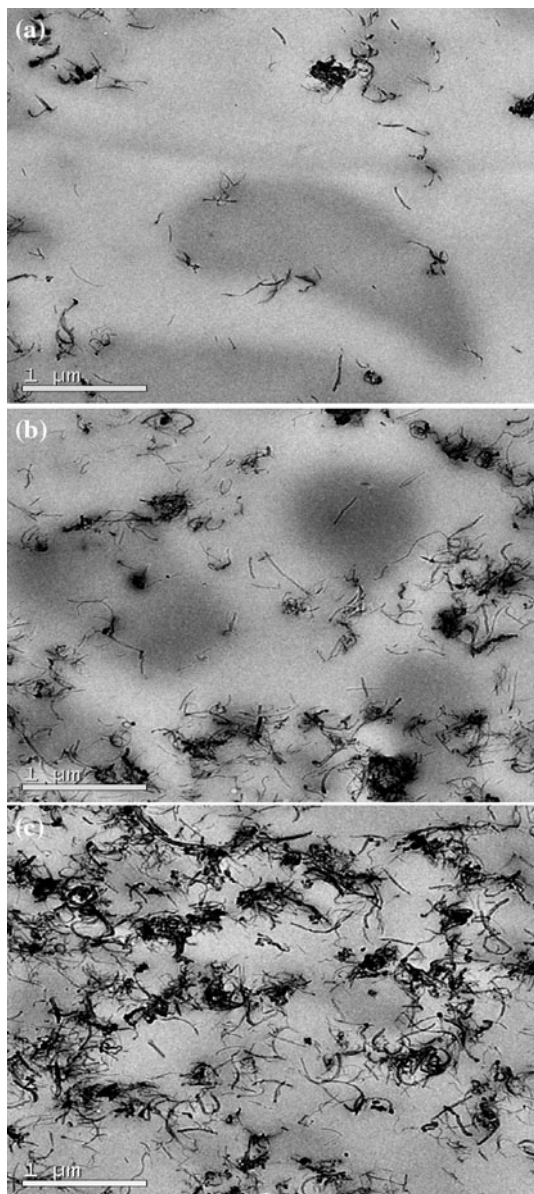
Fig. 4 Transmission electron micrographs of poly(propylene carbonate) (PPC) and poly(lactic acid) (PLA) (70/30) with MWCNT composites: **a** 1 phr, **b** 3 phr, and **c** 5 phr

Table 2 Surface tensions(γ) of dispersive part (γ^d) and polar part (γ^p) for poly(propylene carbonate), poly(lactic acid), MWCNT and water

Materials	γ (mJ/m^2)	γ^d (mJ/m^2)	γ^p (mJ/m^2)
PPC	37.0	12.8	24.2
PLA	35.5	18.1	17.4
MWCNT	45.3	18.4	26.9
Water	72.8	21.8	51.0

the preferred localization of MWCNT in one phase can be explained by measuring the interfacial tension between MWCNT and PPC and PLA.

Interfacial tension of MWCNT and polymers

The localization of conductive filler in the PPC/PLA blend was further examined by measuring the interfacial tension between the MWCNT and polymers. Sumita et al. [27] measured the localization of MWCNT in polymer blends using the wetting coefficient, which can be obtained from the interfacial tension of the components. The interfacial tension can be calculated from the harmonic mean equation [30] which is a general method for calculating the interfacial tension (γ_{12}) between the polymer pairs, as follows:

$$\gamma_{12} = \gamma_1 + \gamma_2 - 4\left[\frac{\gamma_1^d \gamma_2^d}{\gamma_1^d + \gamma_2^d} + \frac{\gamma_1^p \gamma_2^p}{\gamma_1^p + \gamma_2^p}\right], \qquad (1)$$

where γ_1 and γ_2 are the surface tensions of the components 1 and 2, respectively. γ^d is the dispersive part of the surface tension, and γ^p is the polar part of the surface tension. The surface tensions of the PPC, PLA, MWCNT, and water were 37.0, 35.5, 45.3, and 72.8, respectively (Table 2). The surface tensions of the PPC and PLA were obtained from the contact angle measurements shown in Fig. 5 (PPC = 64.9° and PLA = 68.7°). The surface tensions of the water and MWCNT were obtained from Nuriel et al. [31] and the surface tensions for the γ^d and γ^p were calculated from Owens and Wendt [32] and Van Krevelen and Te Nijenhuis [33]. Based on the surface tension shown in Table 2, the interfacial tensions of the PPC and PLA pair and between the MWCNT and polymers were calculated using Eq. (1), and the results are shown in Table 3.

As shown in Table 3, the interfacial tension of the PPC and MWCNT was determined to be 1.15 mJ/m^2. Also the interfacial tension of the PLA and MWCNT was calculated to be 2.04 mJ/m^2, which was higher than that of the PPC and MWCNT. Based on the TEM analysis of the PPC/PLA/MWCNT composites shown in Fig. 2d, MWCNT appear to be more localized in the PPC phase than in the PLA phase. Because the interfacial tension of the PPC/MWCNT composite was lower than that of the PLA/MWCNT composite, MWCNT would prefer to locate more in the PPC phase. The melt viscosity of the PPC and PLA are similar; therefore, the localization of the MWCNT is dependent on a combination of thermodynamic and kinetic parameters. Since selective localization of MWCNT can result in the formation of a conductive path, the localization of MWCNT may affect the electrical conductivity of the polymer/MWCNT composites.
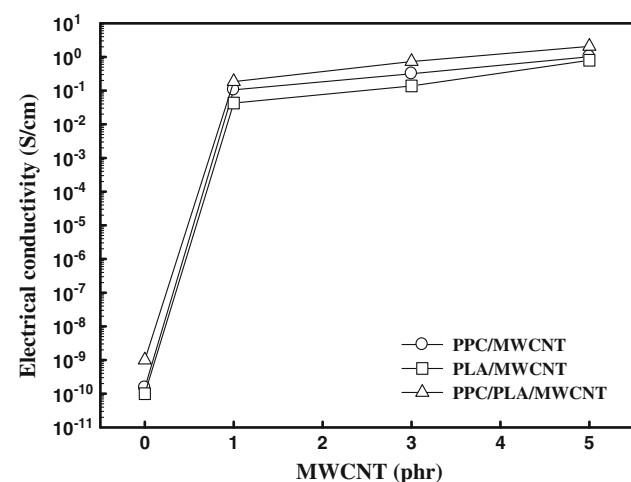
Electrical conductivity of the PPC/PLA/MWCNT composites

Figure 6 shows the electrical conductivities (σ) of the PPC/PLA/MWCNT composites at different MWCNT concentrations.
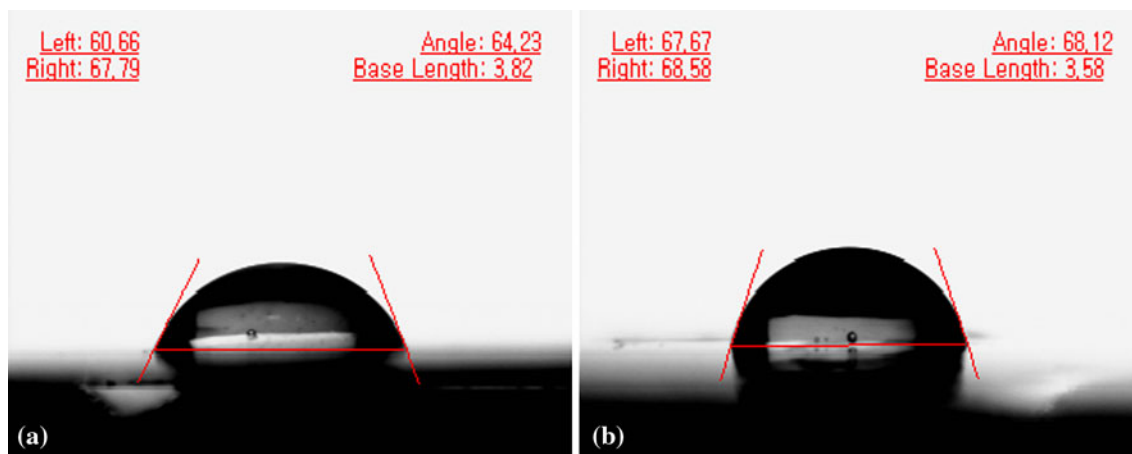
Fig. 5 Phase angle measurements of **a** poly(propylene carbonate) and **b** poly(lactic acid)

Table 3 Interfacial tensions of poly(lactic acid), poly(propylene carbonate), and multi-walled carbon nanotube

Materials	Interfacial tension (mJ/m^2)
PPC/PLA	2.02
PPC/MWCNT	1.15
PLA/MWCNT	2.04

Fig. 6 Electrical conductivity of poly(propylene carbonate) (PPC)/ MWCNT (*open circle*), poly(lactic acid) (PLA)/MWCNT (*open square*), and PPC/PLA (70/30) (*open triangle*) with MWCNT composites

As shown in Fig. 6, the electrical conductivities of the PPC/ PLA/MWCNT composites increased from 1.00×10^{-9} to 2.062 S/cm when the MWCN content was increased from 0 to 5 phr. For the PPC/MWCNT and PLA/MWCNT composites, the electrical conductivities increased from 1.5×10^{-10} to 1.012 S/cm and 1.0×10^{-10} to 0.797 S/cm, respectively. Based on the results shown in Fig. 6, the values of electrical conductivities of the PPC/PLA/MWCNT composites showed slightly higher than those of the PPC/MWCNT and PLA/MWCNT composites. This maybe explained by the fact that selective localization of MWCNT in the PPC phase can form more conductive paths, as was observed in Fig. 4; however, some of the MWCNTs were still located in the PLA phase (dark circle). The results presented in Fig. 6 suggested that limited increase in the electrical conductivities was observed because of the MWCNTs remained in the PLA phase of the PPC/PLA/MWCNT composites.

Mechanical property of the PPC/PLA/MWCNT composites

Figure 7 shows the tensile strength of the PPC/PLA/ MWCNT composites as a function of MWCNT concentration. As shown in Fig. 7, the tensile strength of the PPC/ MWCNT and PLA/MWCNT composites increased from 29.5 to 43.2 MPa and from 60.1 to 78.3 MPa which were 46.4 and 30.3 % increase from their initial values, respectively, when the MWCNT content was increased from 0 to 5 phr. The increase in tensile strength is less significant for the PLA/MWCNT composite (30.0 %) than the PPC/MWCNT composite (46.4 %). This result is maybe due to the higher interfacial tension of the PLA/ MWCNT composite than that of the PPC/MWCNT composite. More homogeneous dispersion of the MWCNT in the PPC phase may increase the tensile strength of the PPC/ MWCNT composites. For the PLA/MWCNT (5 phr) composites, the increase in tensile strength is insignificant when we also compare with the tensile strength reported by other researchers. In the studies of MWCNT dispersed phenolic composites, Tai et al. [34] reported that tensile

strength was increased from 42.5 to 61.0 MPa when the MWCNT content was increased from 0 to 3 phr, which showed 51.9 % increase in tensile strength. For the PPC/PLA/MWCNT composites, tensile strength values were in between those of the PPC/MWCNT and PLA/MWCNT composites.

Rheological property of the PPC/PLA/MWCNT composites

Figure 8 show the complex viscosity of the PPC/MWCNT and PLA/MWCNT composites at MWCNT concentrations of 1, 3, and 5 phr. As shown in Fig. 8, the complex viscosity of the PPC/MWCNT and PLA/MWCNT composites increased with an increase in the MWCNT content. At a frequency of 10 rad/s, the values of the complex viscosity of the PPC/MWCNT composites were 214.8, 1331.4, and 8745.7 Pa s at MWCNT concentrations of 1, 3, and 5 phr, respectively. For the PLA/MWCNT composites at a frequency of 10 rad/s, the values of the complex viscosity were 863.5, 2820.4, and 10865.3 Pa s at MWCNT concentrations of 1, 3, and 5 phr, respectively. For the PPC/MWCNT composites, the ratio of increasing the complex viscosity with the MWCNT was 6.20 and 6.57 at MWCNT concentrations ranging from 1 to 3 phr and 3 to 5 phr, respectively. For the PLA/MWCNT composites, the ratio of increasing the complex viscosity when the MWCNT concentrations ranged from 1 to 3 phr and 3 to 5 phr was 3.26 and 3.85, respectively. Figure 8 show that the change in ratio of increasing the complex viscosity at higher MWCNT concentrations was more significant for the PPC/MWCNT composites than PLA/MWCNT composites.

In the studies of single-walled carbon nanotube/poly (methyl methacrylate) composites, Du et al. [35] reported that the higher values of the storage modulus were associated with better nanodispersion of CNT, when all other factors were constant. Therefore, based on results presented in Fig. 8, the ratio of increasing the complex viscosity of the PPC/MWCNT may have resulted from the higher MWCNT dispersion in the PPC phase than PLA phase. This result is consistent with the results of TEM and electrical conductivities of the PPC/PLA/MWCNT composites, where the selective localization of the MWCNT in the PPC phase resulted in improved conductive paths, and therefore increased the electrical conductivities of the PPC/PLA/MWCNT composites.

Conclusions

In this study, the morphological, electrical, and rheological properties of the PPC/PLA (70/30, wt%) with MWCNT
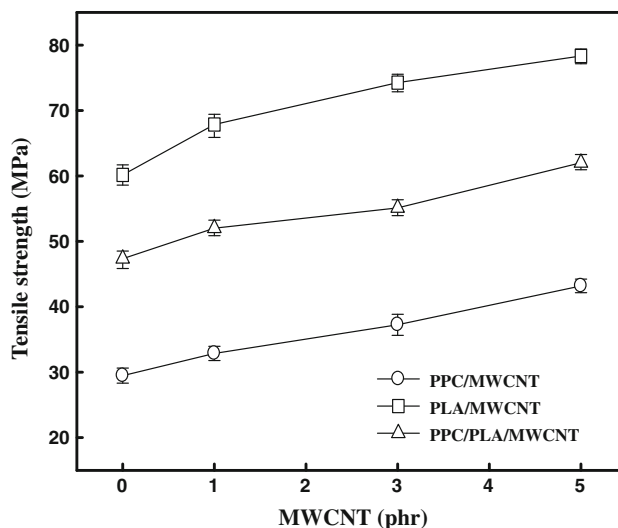

Fig. 7 Tensile strength of poly(propylene carbonate) (PPC)/ MWCNT (*open circle*), poly(lactic acid) (PLA)/MWCNT (*open square*), and PPC/PLA (70/30) (*open triangle*) with MWCNT composites
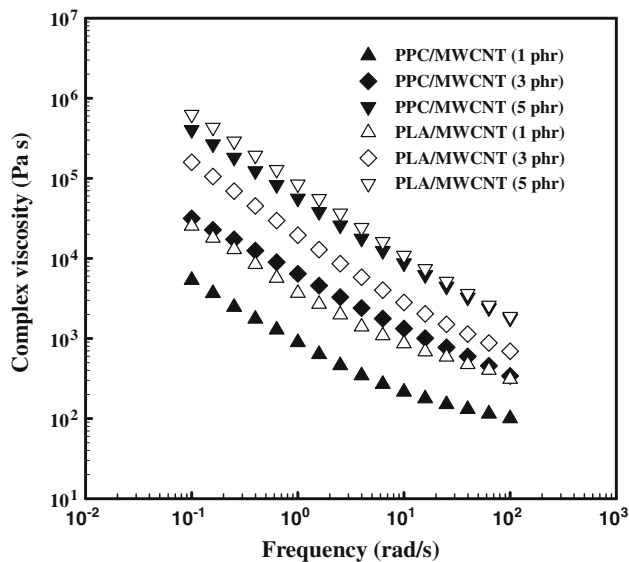

Fig. 8 Complex viscosity of poly(propylene carbonate) (PPC)/ MWCNT and poly(lactic acid) (PLA)/MWCNT composites: PPC/ MWCNT (1 phr) (*filled triangle*), PPC/MWCNT (3 phr) (*filled diamond*), PPC/MWCNT (*filled inverted triangle*) (5 phr), PLA/ MWCNT (1 phr) (*open triangle*), PLA/MWCNT (3 phr) (*open diamond*), and PLA/MWCNT (5 phr) (*open inverted triangle*)

composites were investigated. Based on the morphology of the PPC/PLA/MWCNT composites, the MWCNT tended to locate more in the PPC phase (continuous phase) than in the PLA phase (domain). From the measurements of interfacial tension of the composites, the interfacial tension of the PPC/MWCNT composites was lower than that of the

PLA/MWCNT composites. Based on this result, it was suggested that the lower value of interfacial tension of the PPC/MWCNT composites affected the preferred localization of the MWCNT in the PPC phase.

The electrical conductivities of the PPC/PLA/MWCNT composites was higher than those of the PPC/MWCNT and PLA/MWCNT composites. This is maybe due to the selective localization of the MWCNT in the PPC phase (continuous phase); however, limited increased in the electrical conductivities were observed due to the MWC NTs remained in the PLA phase (domain).

Based on the complex viscosity analysis of the composites, the ratio of increasing the complex viscosity of the PPC/MWCNT was higher than that of the PLA/MWCNT composites. This is maybe due to the higher MWCNT dispersion in the PPC phase than in the PLA phase. The above result is consistent with the results of TEM and electrical conductivities of the PPC/PLA/MWCNT composites, where selective localization of the MWCNT in the PPC phase was shown to improve the conductive paths, and therefore increased the electrical conductivities of the PPC/PLA/MWCNT composites.

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