Multi-walled Carbon Nanotube Filled Polypropylene Nanocomposites: Electrical, Mechanical, Rheological, Thermal and Morphological Investigations

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Abstract

The present research aims to investigate the mechanical, rheological, thermal, electrical and morphological properties of nanocomposites prepared from polypropylene (PP) and multi-walled carbon nanotube (MWCNT) whose content was varied from 1-6wt%. The electrical resistance of the PP/MWCNT decreased dramatically with the addition of the MWCNT; the PP/MWCNT showed an electrical percolation threshold at the MWCNT content of 1 wt%. The relative viscosity was reduced as the shear rate and temperature increased. The thermal properties of the PP/MWCNT and the crystallization temperature were raised significantly with the MWCNT loading but the degree of crystallinity remained unchanged. The tensile modulus of the PP/MWCNT increased significantly with the presence of the MWCNT while the impact energy increased with the MWCNT loading.

Keywords: Polymer nanocomposites; Carbon nanotube; Electrical properties; Mechanical properties.

1. Introduction

In the last decades, small scaled carbon materials such as carbon nanotube (CNT) have generated much attention from both research institutions and industries due to their outstanding performances especially when they are incorporated into polymer matrices [1]. This was believed to be the results of their extremely high surface area,

modulus and strength [2-3]. Numerous literatures have found that the mechanical properties of polymers, especially modulus and strength can be enhanced by incorporating only a small amount of CNT [4]. Therefore CNT has been utilized as reinforcement in polymers to form nanocomposite materials [5] in engineering applications.

Polypropylene (PP) is one of the most important commodity plastics; it is generally used in the plastic industry to produce bottles, films, sheets or fibers [6]. Unfortunately, the application of PP has restricted by its softness at ambient temperature, its brittleness at low temperatures and its insulation properties [7]. Thus, many attempts have been made to improve the performances of PP or to achieve new properties of the PP composites [4-7]. Composite materials based on PP and CNT have gained much attention in the path of developing new materials. In many observations, the property enhancement was investigated by studying the electrical [8], mechanical [2], thermal [7] and other properties [9]. The flexural modulus of the PP/CNT composites was found to increase from 1.28 to 2.15 GPa when the CNT was add at 5.0wt% [2]. Significant improvement in the crystallization was found when the CNT was added at only 0.5wt% [10]. Moreover, it was reported that the electrical percolation could occur at very low CNT content of 0.05vol% [11].

The present study aims to investigate the effects of multi-walled carbon nanotubes (MWCNT) on the mechanical, thermal, rheological, electrical and

morphological properties of PP/MWCNT composites. Each formulation of the PP/MWCNT was processed by using a twin screw extruder with the MWCNT content varied from 1-6wt% at different melt-blending screw speeds of 200 and 270 rpm.

2. Experimental Works

2.1 Raw Materials

Polypropylene (PP) (Polimaxx 1100RC) with a melt flow index (MFI) of 20g/10 min at 230°C was obtained from IRPC Public Company Limited (Bangkok, Thailand). The masterbatch of MWCNT (Plasticyl-2001), containing 20wt% of MWCNT was provided by Siam Extek Company Limited, Thailand. The MWCNT masterbatch contained the nanotubes with average diameter of 9.5nm and average length of 1.5µm. The purity of the masterbatch was over 90%.

2.2 Preparation of the PP/MWCNT composites

The initial MWCNT masterbatch concentration was diluted to 1, 2, 4 and 6wt% by melt blending with a neat PP. Each formulation of the PP/MWCNT was processed by using a twin screw extruder (HAAKE minilab II) with different melt-blending at the re-circulation residence time of 15 min and at the screw speeds of 200 and 270rpm at 200°C. Both speeds covered the range appropriate for the PP with MFI 20g/10 min selected for the present study and yielded the residence of 15min for melt blending within the extruder. After extrusion, the PP/MWCNT composites were injected by using the injection molding machine (Thermo Scientific HAAKE MiniJet II) to obtain specimens for subsequence tests.

2.3 Testing and characterizations

A dielectric constant (K) is an important parameter indicating the capability of the materials to store the electrical energy. In the current research, the dielectric constant was evaluated by using the Agilent E4980A Precision LCR Meter according to the method described in ASTM D 150 [12]. The test was conducted at 1 volt for 60 seconds. The K value was calculated by using Equation (1).

$$C = K\epsilon_0 \frac{A}{d} \quad (1)$$

where C is the capacitance, K is the dielectric constant, A is the cross sectional area of the test specimen (mm^2), d and ϵ_0 are the thickness of the specimen (mm) and permittivity of vacuum (0.0088542 pF/mm) respectively.

The electrical resistivity was determined in accord with ASTM D 257 by using a high resistance meter (Agilent 4339B) and a source meter (Keithley source meter 2420). Equation (2) was applied in order to calculate the electrical resistivity (ρ , $\Omega\cdot\text{cm}$) of the neat PP and the PP/MWCNTs composites.

$$\rho = R \frac{A}{l} \quad (2)$$

where R is the electrical resistance of the material (Ω), l is the length of the test specimen (cm) and A is the cross-sectional area of the test specimen (cm^2).

The transition temperatures namely glass transition temperature (T_g), crystallization temperature (T_c) and melting temperature (T_m) of the PP/MWCNT composites were evaluated by using a Differential Scanning Calorimeter (DSC, Mettler Toledo). The test was run from -30 to 200°C (cooling from 25 to -30°C, holding for 10 min, heating from -30 to 200°C, holding for 10min, cooling from 200 to -30°C, holding for 10 min and re-heating from -30 to 200°C) at the heating rate of 10°C/min under nitrogen atmosphere.

The degree of crystallinity (X_c) of the PP/MWCNT composites was evaluated from the DSC scans and calculated by using Equation (3) [13].

$$X_c (\%) = 100 \frac{\Delta H_m}{\Delta H_f \times X_{pp}} \quad (3)$$

where ΔH_m , and X_{pp} are the enthalpy of melting, the weight fraction of the PP respectively. ΔH_f is the heat of fusion, defined as the melting enthalpy of 100% crystalline PP homopolymer, which was 190J/g [14].

The tensile test was conducted by using a universal testing machine (INSTRON, 5567) at room temperature according to the procedure described in ASTM D 638. The notched Izod impact strength of the nanocomposites was determined following ASTM D 256 by using an impact tester (Yasuda, 258PC) at room temperature. A three-point bending test with a support span of 48 mm was carried out by using a Universal Testing Machine (INSTRON, 5567) at room temperature and the crosshead speed was set at 12 mm/min. Hardness test was conducted by using Rockwell Hardness tester R Scale with a 0.5in ball penetrator (Identec hardness tester). A minor load of 10kg was first applied on the PP/MWCNT surfaces to obtain the primary imprint. A 50 kg major load was then applied to the test specimen in order to evaluate the Rockwell hardness number.

The shear viscosity of the PP/MWCNT was measured by using a twin screw extruder (HAAKE minilab II). The relative viscosity and the shear rate at 200°C were recorded

simultaneously.

Microscopic observation of the fractured surfaces of the PP/MWCNT composites was conducted by using a field-emission scanning electron microscope (FE-SEM) (Hitachi S-4700) at an accelerating voltage of 5 kV. The study was performed to investigate the fractured topology and the dispersion of the MWCNT in the PP matrix.

3. Results and Discussion

3.1 Effects of MWCNT on the electrical properties of the PP/MWCNT nanocomposites

The influence of concentration of the MWCNT and the screw speed on the electrical resistivity and the dielectric constant of PP/MWCNT composites are shown in Figs. 1 and 2 respectively. It is evident that the electrical resistivity was obviously affected by the MWCNT contents studied, leading to a wide range of resistivity from 10^{15} to $10^4 \Omega \cdot \text{cm}$. The electrical resistivity of the neat PP was $2.16 \times 10^{15} \Omega \cdot \text{cm}$ while the addition of only 1wt% MWCNT drastically reduced the electrical resistivity by approximately 109 times. This implies that the concentration of conductive MWCNT played an essential role in the formation of conducting network in PP/MWCNT nanocomposites. Further increment of the MWCNT content did not lead to any significant reduction of the electrical resistivity due to the formation of MWCNT agglomeration, as illustrated in Fig. 6.

Dielectric constant data of the PP/MWCNT nanocomposites at different concentrations of MWCNT and screw speeds is presented in Fig. 2. The dielectric constant increased with the MWCNT content at all compositions studied. By adding only 1wt% MWCNT, the dielectric constant of the nanocomposites increased around two times while the electrical resistivity decreased as shown in Fig. 1. When the MWCNT content reached 6wt%, the dielectric constant increased significantly by 15 and 16 times for the nanocomposites processed at speed 200 and 270rpm respectively. However, the dielectric constant values were found to be lower than those reported by Tjong *et al.* [17] and Bikiaris [16]. This was probably the consequence of the agglomerations of MWCNT formed in the nanocomposite, as illustrated in the FE-SEM micrograph in Fig. 6.

However, identical resistivity was found irrespective of the screw speed of 200rpm and 270rpm. Similar PP/MWCNT morphology and dispersion characteristic was observed at both speeds of screws during melt blending,

this was illustrated in the FE-SEM micrographs enclosed in Fig. 1. The influences of the MWCNT content on the electrical properties of nanocomposites were observed by many researchers. The resistivity decrement was believed to have caused by the formation of conductive chains in the composites [15]. The percolating network of the MWCNT at all the concentrations tested seemed to have assumed random positions but MWCNTs were observed to have overlapped with other MWCNTs [16] and formed interconnected conductive pathway as enclosed in Fig. 1.

Fig. 1 Electrical resistivity of PP/MWCNT composites at the screw speed of 200 and 270 rpm.

Fig. 2 Dielectric constant of PP/MWCNT composites at the screw speed of 200 and 270 rpm.

3.2 Effects of MWCNT on the mechanical properties of the PP/MWCNT nanocomposites

Tensile properties of the injection molded

nanocomposites are shown in Fig. 3. The Young's modulus of the nanocomposites was found to have increased substantially compared with that of the neat iPP as illustrated in Fig. 3(a). The tensile modulus of PP/MWCNT increased with increasing MWCNT contents. It became stagnate at greater MWCNT content of 4-6 wt%. This was due to the formation of some agglomerates at the MWCNT content of 4-6wt%. The size of the MWCNT agglomerates was found to increase with increasing nanotube content. At low MWCNT contents, the nanotubes act as a reinforcing phase, but at higher MWCNT contents they tended to form agglomerates which adversely acted as mechanical failure concentrators.

Fig. 3 Tensile properties of PP/MWCNT nanocomposites at the screw speed of 200 and 270 rpm (a) tensile modulus and (b) tensile elongation at break.

Thus, PP/MWCNT nanocomposites exhibited their

higher mechanical performance at the concentration of 1-4wt% but further increase of the MWCNT led to a lower tensile strength of the nanocomposites. At low MWCNT content, partial tensile stress could be transferred to the MWCNT embedded in the PP matrix, leading to an increase of the tensile strength. With further addition of the MWCNT, it was difficult to disperse the MWCNT during melt mixing [16-17]. Hence, more agglomerates of MWCNT were formed in PP matrix; they acted like many defects in the PP matrix. In Fig. 3(b), the typical ductile behavior with a very high elongation at break of around 610 and 615% for the PP processed at 200 and 270 rpm respectively changed drastically with the presence of MWCNT. The addition of only 1wt% MWCNT led to dramatic reductions in the tensile elongation at break by 40 and 30times for PP/MWCNT nanocomposites processed at 200 and 270 rpm respectively. It was believed that the numerous MWCNT obstructed the entanglement of the PP chains and lowered the tensile elongation at break.

Fig. 4 Impact strength of PP/MWCNT composites at the screw speed of 200 and 270 rpm.

The impact strength of the notched PP/MWCNT samples is shown in Fig. 4. The impact resistance increased slightly with increasing MWCNT content. At low MWCNT concentration, the impact strength increased steadily reaching the maximum value over the MWCNT ranged 1-4 wt%, depending on the melt mixing speed of the screw, and gradually decrease after that concentration. At 1wt% MWCNT the impact strength was found to increase by 5 and 11% for the PP/MWCNT processed at 200 and 270 rpm respectively. More pronounced decrease was observed at 6 wt% MWCNT content; this was also due to the presence of

nanotube agglomerations in the PP matrix. They became the points of higher stress concentrations, thus providing sites for crack initiation [2]. The lower impact energy at higher nanotube content was due to the presence some masterbatch agglomerates in the composites [18] as shown in Fig. 6.

Fig. 5 Hardness of PP/MWCNT composites melt blended at the screw speed of 200 and 270 rpm.

Changes in the hardness of the PP and PP/MWCNT nanocomposites exhibited a similar trend as the tensile modulus as illustrated in Fig. 5. The hardness of the MWCNT reinforced PP composites was enhanced with increasing MWCNT content simply because the MWCNT was a much harder phase than the PP matrix. The hardness result was in agreement with those found in many nanocomposites having MWCNT as the reinforcing phase although the matrix studied was varied such as poly(methyl methacrylate) (PMMA) [19], epoxy [20], polyacrylonitrile (PAN) [21] and nitrile rubber [22].

The SEM micrographs of the PP/MWCNT composites with various MWCNT contents are illustrated in Fig. 6. The MWCNT agglomerates were found to partially infiltrate by the polymer matrix during melt-mixing. As illustrated in Fig. 6(a) and 6(e), at low MWCNT content, most of the MWCNTs dispersed well with only small clusters in the iPP matrix. At relatively high MWCNT content, more MWCNT agglomerates appeared in the PP matrix, the size of the agglomerates increased with increasing MWCNT content. The cluster sizes depended mainly on the MWCNT concentration. At low MWCNT content of 1 and 2wt%, the agglomerates were smaller in cluster. The mean cluster sizes were in the range of 0.2 up to 1 μ m. As MWCNT content increased, MWCNT formed larger agglomerates

than 1 μ m. This was due to the surface forces of each MWCNT [16].

Fig. 6 SEM micrographs of iPP/MWCNT composites.

Fig. 7(a) and (b) shows the effect of the MWCNT content on the rheological properties of the PP/MWCNT composites processed at 200 and 270rpm respectively. Comparison of the melt viscosities indicated the significant effects of the MWCNT content. Over the shear rate range of 35-500s⁻¹, the molten PP/MWCNT composites showed higher viscosity compared with that of neat PP for both processing speeds of 200 and 270rpm. The PP/MWCNT was formed to exhibit a shear thinning character. The change of viscosity by the addition of MWCNT lied within this range of shear rate. The rise in the viscosity might have been due to the confinement of the PP chains due to the presence of numerous MWCNTs. However, at a higher shear rate of 500-960 s⁻¹, the melt viscosity of PP/MWCNT was close to that of the neat PP.

Table 1 lists the temperatures of glass transition (T_g), crystallization (T_c), melting (T_m) as well as the degree of crystallinity (X_c) of the PP and PP/MWCNT

nanocomposites. Both the incorporation of the MWCNT and the variation of the screw speed exerted insignificant effects on the T_g , T_m and the X_c of the PP. Interestingly, the shift of the T_c to high temperatures was relatively small for the composites with lower MWCNT contents compared to those with higher MWCNT. Further increase of the MWCNT contents in the composites above 2wt% led to a rapid rise of the T_c .

Fig. 7 Relative viscosities of iPP/MWCNT composites at the screw speed of (a) 200 and (b) 270 rpm.

The additions of MWCNT by 2wt% produce a highly heterogeneous nucleation effect in the PP. Park *et al.* also reported that the addition more than 1wt% MWCNT enhanced the nucleation process during PP crystallization [10]. With increasing the MWCNTs content, it has been

found that the PP crystallinity could remain stable [23], decrease slightly [24] or increase [25].

Table 1 Transition temperatures and degree of crystallinity of PP/MWCNT nanocomposites.

Transition temperature	Screw speed (rpm)	MWCNTs content (wt%)				
		PP	1.0	2.0	4.0	6.0
T_g (°C)	200	-17.3	-17.2	-17.4	-16.7	-16.6
	270	-17.1	-17.3	-17.3	-17.4	-17.6
T_c (°C)	200	116.1	128.0	128.8	131.3	131.9
	270	116.0	127.4	129.8	132.2	133.2
T_m (°C)	200	161.2	164.7	164.9	166.0	167.2
	270	161.2	164.4	164.1	165.0	167.4
X_c (%)	200	46.8	47.3	47.7	47.0	45.9
	270	46.8	49.3	46.2	45.1	45.0

4. Conclusions

By adding only 1wt% of MWCNT, the dielectric constant of the PP/MWCNT nanocomposites increased around two times while the electrical resistivity decreased. The tensile modulus and the hardness of the PP/MWCNT increased with increasing MWCNT contents. The modulus of the PP/MWCNT with 6 wt% MWCNT processed at both 200 and 270 rpm were found to decline due to the formation of some MWCNT agglomerates. The molten PP/MWCNT composites show higher viscosity compared with that of the neat PP due to the shear thinning character. At low MWCNT content, the MWCNTs dispersed well within the PP matrix. At relatively high MWCNT contents, more MWCNT agglomerates appeared in the PP matrix. The size of the agglomerates increased with increasing MWCNT content.

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