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MECHANICAL AND THERMAL PROPERTIES OF POLYPROPYLENE/CARBON NANOTUBES (CNT) NANOCOMPOSITES: EFFECT OF 1-OCTADECANOL SURFACE MODIFICATIONS

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Abstract
Multiwall carbon nanotubes (CNT) functionalized with 1-octadecanol (C_{18}) and characterized by using Fourier Transform Infrared Spectroscopy (FTIR) before being incorporated into isotactic polypropylene (iPP) matrix. The blends of iPP/CNT composites for both unfunctionalized and functionalized carbon nanotubes (CNTs) were prepared by melt blending techniques in a mini blender. The tensile properties of the composite were observed to increase with nanotube loadings (0.1, 0.25, 1.00 and 5.00 wt %). The DSC analysis showed the nucleating effect of the CNTs on the crystallization properties of iPP matrix. TEM images of the composites confirmed better dispersion of CNT-C_{18} in the iPP matrix in comparison to unfunctionalized CNTs.

Keywords: Multiwall carbon nanotube, functionalization, isotactic polypropylene, crystallization, mechanical properties

Introduction

Polypropylene (PP) is the most important polyolefin, with a wide range of grades available for various applications. Low price, excellent chemical resistance, acceptable range of tensile strength and modulus, good impact strength, and processability make it an attractive candidate for many applications [1]. Various reinforcements like mineral fillers, nanoclay, nanotubes and recently nanotubes with modification including surface modification and grafting [2 - 4], are widely used in the polypropylene matrix on account of increasing stiffness of the matrix, increasing dimensional stability and reducing the production cost. The incorporation of filler may also adversely affect the ductility, strength and processability of the composites [5].

Since the discovery of carbon nanotube, research work on carbon nanotubes and their composites has been reported [3–4]. Studies have shown that iPP has emerged as a widely used matrix for carbon nanotube composites. Both single-wall and multiwall nanotubes have been used to study crystallization behavior, morphology, mechanical and thermal properties of iPP composites [5]. Results have been mixed, for both mechanical and thermal properties, while Moore *et al.* showed no significant improvement in mechanical properties, and Bhattacharya *et al.* showed moderate improvements in tensile strength with decreased toughness for the iPP system [5–6]. However, more recent work indicates that improved interfacial bonding through covalent grafting and silane coupling treatments can significantly enhance stress transfer, stiffness, and overall mechanical integrity in iPP/CNT composites [3, 4].

Similarly, investigation on the thermal properties follows the same suit with some authors reporting no significant variation in degree of crystallinity and a few others indicating decrease and there are reports of increase in crystallinity also [7]. These conflicting reports motivated us to study the mechanical and thermal properties of iPP in iPP/CNT nanocomposites and to investigate the effect of 1-octadecanol functionalized (CNT-C18) on the dispersion of CNT into the polymer matrix. In this paper, two kinds of

multiwall carbon nanotubes: pure CNT and CNT-C18 modified with 1-octadecanol were chosen as reinforcing fillers in iPP composites.

2. Materials and Methods

Commercially available iPP, (density 905 kg/m^3 and MFR = 12 g/10 min), MWCNT (diameter 8-15nm, length 10-50μm, specific area 230m^2/g and purity of more than 95%), Nitric Acid, 1-octadecanol ($C_{18}H_{38}O$), Sulfuric Acid, Toluene and Deionized Water were used.

For the surface modification of CNT, firstly the CNT was oxidized with nitric acid to achieve CNT-COOH bonding following treatment with deionized water. The CNT-COOH was reacted with C_{18} to obtain CNT-C_{18} according to the scheme in Fig. 1.

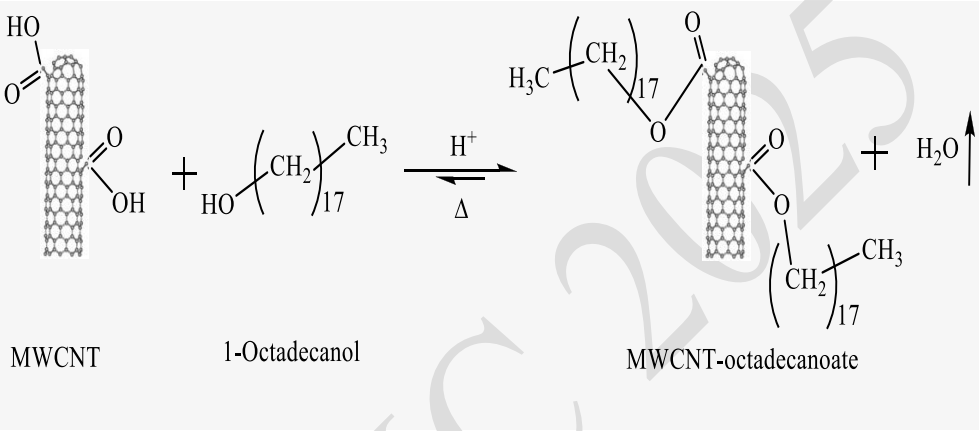

Figure 1: Modification chemistry of CNT surface with C_{18}

iPP/CNT nanocomposites was prepared via dry blending several fractions (0.1, 0.25, 1.0 and 5.0 wt. %) CNT and CNT-C_{18} in a Haake mini extruder respectively. The mixed samples were modeled to prepare the test specimen for the tensile experiments using Carver hydraulic hot-press.

The Fourier transform infrared spectroscopy (FTIR) was used for characterization of the different CNTs. Morphology and dispersion studies was conducted using Transmission electron microscope (TEM) while crystallization and melting temperatures were achieved using Differential Scanning Calorimetry (DSC). Tensile Testing (a minimum of five samples of each composite was tested) was done in Instron according to ASTM-D3638.

3. Results and Discussion

Fig. 2 shows the IR spectrum of both CNT and CNT-C18 nanotubes. The IR spectrum of CNT shows absorption band at 2920 cm^{-1} attributed to asymmetric and symmetric CH_2 stretching, 1698 cm^{-1} to carboxylic C=O stretching and 1097 cm^{-1} to C-O stretch in alcohols. The presence of these functional groups on the surface of CNT indicates their introduction during nanotubes purification processes. Carboxylic C=O stretching peak observed at 1693 cm^{-1} can be attributed to acid treatment of CNT which is reported [4]. Treatment of CNT-C_{18} gives indicative peaks at 2920 cm^{-1} correspond to CH_2 stretching of long alkyl molecule of C_{18}, 1473, 1458 and 1068 cm^{-1} corresponds to the ether formation of C_{18} [8].

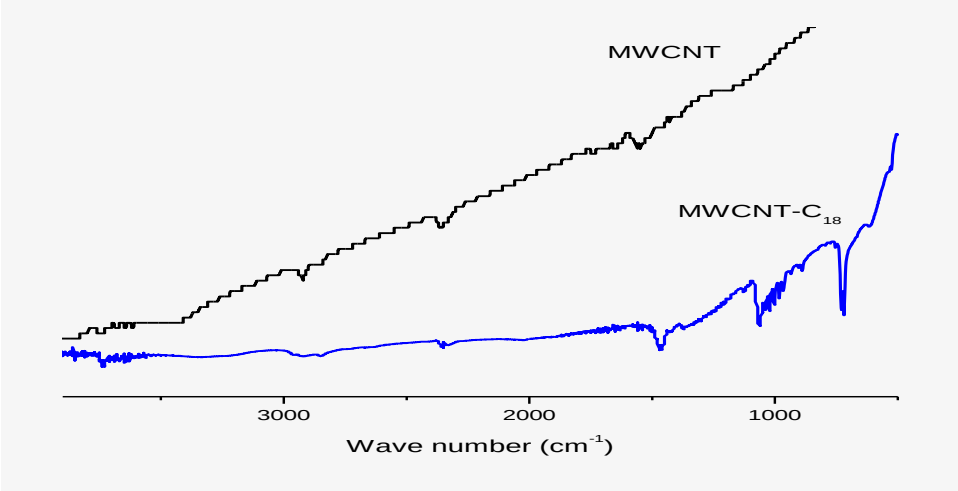

Figure 2: FTIR spectra of CNT, and CNT-C_{18}.

Figure 3 (a) and (b) shows the SEM images of CNT at low and high magnification respectively. From the images, it is clear that the CNTs are pure without impurities.

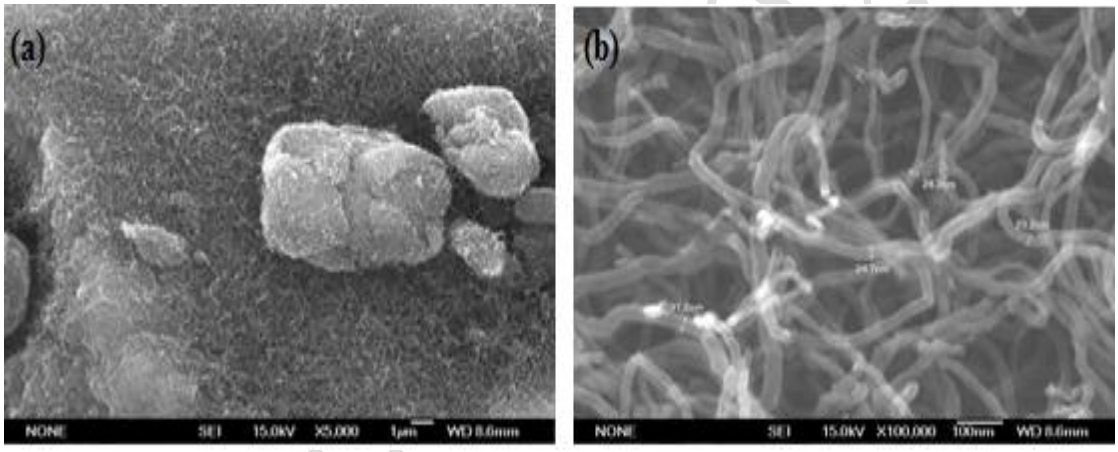

Figure 3: SEM Images of carbon nanotubes at (a) at low resolution (b) at high resolution

The DSC investigates the thermal properties of the composites. Non-isothermal DSC was used to study the effect of crystallization of the matrix. It is obvious from Table 1, that crystallization temperature of 0 wt.% loading is 119.1 ^{0}C while those of 0.25 wt.%, 1.0 wt.% and 5.0 wt.% CNT and CNT-C_{18} composites are respectively 125.8 ^{0}C, 129.4 ^{0}C, 132.1 ^{0}C and 119.7 ^{0}C, 123.7 ^{0}C, 129.2 ^{0}C. In general, a significant increase in percentage crystallinity with increasing CNT concentration is observed.

The mechanical properties of the iPP and the composites is summarized in Table 2. The Young's modulus of CNT composites increase with increase in CNT loading. Thus for example, at 1.0 CNT wt.% fillers, a remarkable 31% increase in Young's modulus is observed. Similar trend is seen for the composites with CNT-C_{18} modified, however, the extent of improvement is not so significant when compared to CNT composites.

Table 1: crystallization temperature (Tc), crystallization enthalpy ΔH (J/g), and the degree of crystallinity (Xc) of PP, iPP/MWCNT and iPP/MWCNT-C_{18} nanocomposites*

CNT loading (wt. %)	T_c (°C)	T_m (°C)	X_c (%)	ΔH (J/g)
0	119.1	161.1	44	97.03
0.10	122.1(118.4)	161.9(160.9)	46(49)	96.31(102.2)
0.25	125.9(119.7)	164.4(159.9)	46(48)	96.52(99.82)
1.00	129.4(123.7)	165.3(163.1)	49(48)	102.0(98.83)
5.00	132.1(129.2)	165.9(164.9)	50(49)	98.77(97.35)

*The values within parentheses correspond to the composites with CNT-C_{18}

The Maximum stress values shows similar trend with CNT showing higher performance followed by CNT-C_{18} composites. A decrease in elongation at break value shows the composites becomes stiffer with increase in loadings.

Table 2: Summary of mechanical properties of pure iPP and iPP/MWCNT composites sample as a function of Nanotubes loading*

CNT loading (wt. %)	Young's modulus (MPa)	Maximum Stress (MPa)	Elongation at break (%)
0	713.1	35.97	26.12
0.10	810.0(823.6)	35.90(37.27)	18.66(22.96)
0.25	899.6(861.0)	37.27(37.65)	16.36(20.05)
1.00	933.8(896.8)	38.76(38.40)	10.07(10.88)
5.00	986.3(945.1)	43.11(39.20)	8.07(7.02)

*The values within parentheses correspond to the composites with CNT-C_{18}

Fig. 4 show the TEM images of the composites with 1.0 wt.% CNT loading. The CNT displays rather good dispersion in the matrix, however, there are a few curled CNTs as well as agglomerates due to the interaction between the nanotubes themselves. The CNT-C_{18} composites show better dispersion than the CNT composites. There is also a few curled in CNT-C_{18} (b) but to a lesser extent compared to CNT composites (a).

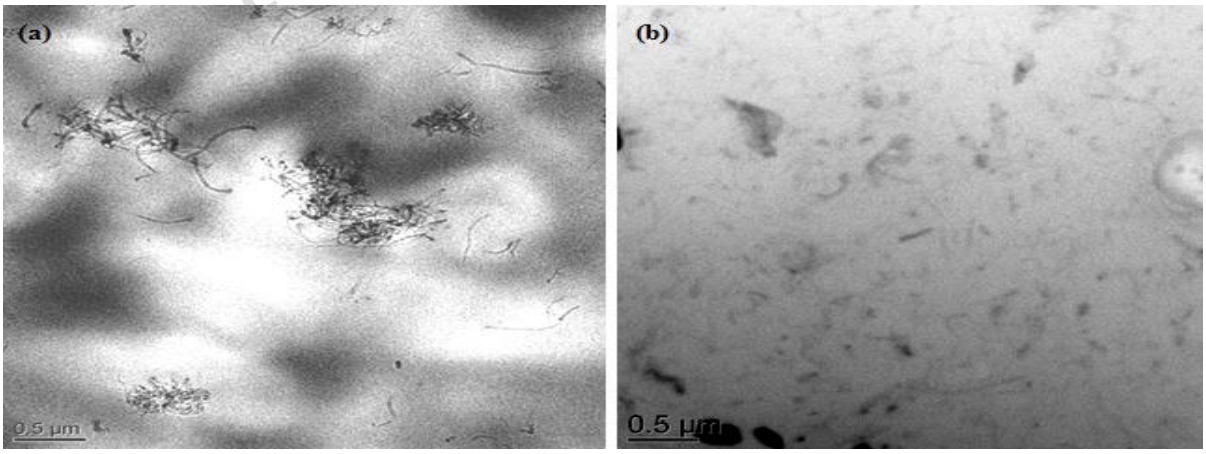

Figure 4: TEM images of 1 wt.% composites of iPP with (a) CNT and (b) CNT-C18

4. Conclusion

The results of the studies on the effects of surface modification of CNT with C_{18} on the mechanical and thermal properties of iPP matrix are reported in this article. The TEM image shows less aggregate and better dispersion in iPP/CNT-C_{18} matrix compared both neat iPP and iPP/CNT. Though a significant improvement in tensile stress and Young's Modulus of iPP reinforced with CNT-C_{18} was observed at 0.1 wt.% loading of the nanotubes, it falls below the reinforcing ability of the CNT nanotubes. Both the tensile properties and percentage crystallization were found to increase with increasing concentration of CNT.

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