

Thermal Properties and Mechanical Characteristics of Cationic Dyeable Poly(trimethylene terephthalate)/Metalocene Isotactic Polypropylene Conjugated Filaments

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ABSTRACT: Cationic dyeable poly(trimethylene terephthalate) (CD-PTT) and metallocene isotactic polypropylene (m-iPP) polymers were extruded (in proportions of 75/25, 50/50, and 25/75) from two melt twin-screw extruders to prepare three CD-PTT/m-iPP conjugated filaments of the island–sea type. This study investigated the thermal properties and mechanical characteristics of the CD-PTT/m-iPP conjugated filaments with gel permeation chromatography, differential scanning calorimetry, thermogravimetric analysis, potentiometry, rheometry, density gradients, wide-angle X-ray diffraction, extension stress–strain measurements, and scanning electron microscopy. The rheological behavior of the CD-PTT/m-iPP polyblended polymers exhibited negative-deviation blends, and the 50/50 CD-PTT/m-iPP blend showed a minimum value of the melt viscosity. The experimental results from differential scanning calorimetry indi-

cated that CD-PTT and m-iPP molecules formed an immiscible system. The tenacity of the CD-PTT/m-iPP conjugated filaments decreased initially and then increased as the m-iPP content increased. Morphological observations revealed that the blends were in a dispersed phase structure. A pore/filament morphology of a larger size (0.5–3 μm in diameter) was observed after a 1,1,1,3,3,3-hexafluoro-2-propanol (CD-PTT was removed)/decalin (m-iPP was removed) treatment in the cross section of a CD-PTT/m-iPP conjugated filament. The CD-PTT and m-iPP polymers were identified as an immiscible system. Blends with 10 wt % compatibilizer exhibited the maximum improvement in the tenacity. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 103: 2387–2394, 2007

Key words: blends; fibers; mechanical properties; thermal properties

INTRODUCTION

The applications of polyblends are important developments for the plastic and synthetic-fiber industries. Polyblends are mixtures of two or more polymers that can either mix completely on a molecular scale or form a two-phase structure. Polyblends can exhibit new combinations of properties that depend on the properties of the components and strongly on the morphology of the blended materials. The morphology resulting from a blending process depends mainly on the rheological and interfacial properties of the molten components, the blending conditions, and the weight ratio of the blended polymers.^{1–3} Polyblends can be characterized by their phase behavior as either miscible or immiscible. The thermal, mechanical, and rheological properties of a polyblend depend strongly on its state of miscibility.⁴

There are some reports on the physical properties of poly(trimethylene terephthalate) (PTT) with other polymers such as poly(ethylene terephthalate),⁵ poly(butylene terephthalate),⁶ poly(ethylene naphthalate),⁷ and clay.⁸ Until now, there have been no reports in the literature for polyblended polymers and conjugated filaments of cationic dyeable poly(trimethylene terephthalate) (CD-PTT) with metallocene isotactic polypropylene (m-iPP).

Polypropylene (PP) resins have been produced with Ziegler–Natta catalysts for over 45 years now. These polymers are typically resins with high molecular weights and broad molecular weight distributions that are produced in a polymerization reactor. In recent years, these resins have frequently been given a post-reactor treatment to narrow their molecular weight distributions and lower their molecular weights when they are to be used for fiber spinning. This treatment typically consists of extrusion in the presence of a peroxide compound, which produces the desired result by thermooxidative degradation of the reactor resin. This method is known as the controlled rheology pro-

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TABLE I
Characteristics of the CD-PTT and m-iPP Pellets

Chip	Intrinsic viscosity (dL/g) ^a	MFR (g/10 min) ^b	5-SSDMI (mol %)	M_w (g/mol) ^c	M_w/M_n ^d	R-COOH (mequiv/kg)	T_m (°C)	T_d (°C) ^e
CD-PTT	0.76	—	2.03	23,000	1.91	20	225.3	376
m-iPP	—	32	—	138,000	1.92	0	149.8	371

^a Obtained for the CD-PTT polymer with a mixed solution of phenol and tetrachloroethane (60/40 w/w) at a concentration of 0.5 g/100 mL and at 30°C.

^b Measured for the m-iPP polymer according to the ASTM D 1238 method (230°C/2.16 kg).

^c Weight-average molecular weight.

^d Weight-average molecular weight/number-average molecular weight (i.e., the molecular weight distribution of the polymer). The molecular weight distributions of the CD-PTT and m-iPP polymers were measured in HFIP and TCB solvent systems, respectively.

^e Degradation temperature of the polymer.

cess. A major advantage of metallocene catalysts in the preparation of PP is that narrow molecular weight distribution resins can be obtained directly from the reactor without the need for secondary processing.⁹

For this study, m-iPP was chosen as a blended material. The CD-PTT polymer possesses 5-sodium sulfonate dimethyl isophthalate (5-SSDMI; —SO₃Na groups). Furthermore, the m-iPP polymer does not possess any functional groups. The interfacial adhesion plays a critical role for CD-PTT/m-iPP conjugated filaments. This study thoroughly investigated the rheological behavior, thermal behavior, crystallinity, tenacity, density, morphology, and influence of a maleic anhydride grafted polypropylene (PP-graft-MA) compatibilizer for CD-PTT/m-iPP conjugated filaments of the island-sea type.

EXPERIMENTAL

Materials and melt spinning

CD-PTT pellets were obtained from the Material and Chemical Research Laboratories/Industrial Technology Research Institute (Hsinchu, Taiwan). The CD-PTT polymer, a white pellet with an average diameter size of 3–5 mm, has been specially designed for the fiber-spinning process. Its intrinsic viscosity is 0.76 dL/g. Before spinning, CD-PTT was dried for 4 h at 120°C and for 8 h at 150°C. m-iPP pellets were obtained from Exxon Mobil Chemical Co. (Houston, TX). The m-iPP resin (Achieve 3825), a white pellet with an average diameter size of 3–5 mm, has been specially designed for the fiber-spinning process. Its nominal melt flow rate (MFR) is 32 g/10 min at 230°C. PP-graft-MA (Polybond 3200) was supplied by the courtesy of Crompton Corp. (Middlebury, CT). The grafting degree of maleic anhydride (MA) was 1 wt %, and its MFR was 110 g/10 min. Table I lists the synthetic characteristics of the CD-PTT and m-iPP polymers. The CD-PTT and m-iPP polymers were extruded in proportions of 75/25, 50/50, and 25/75 from two melt twin-screw extruders to prepare three CD-PTT/m-iPP conjugated filaments.

All conjugated filaments were prepared on an AW-909 bicomponent, precision-conjugated filament-winding machine (Barmag, Remscheid, Germany), which consisted of two 1-in. extruders, two metering pumps, a bicomponent spin pack purchased from Teijin Seiki Co. (Tokyo, Japan), a 15-ft stack with a cross-air-cooling channel, a spin finish applicator, and a speed winder. The extrusion temperature of the CD-PTT and m-iPP polymers was 260°C. The cross sections of the CD-PTT/m-iPP conjugated filaments were prepared for an island-sea type, and the combined melts were extruded through 24 round holes of a 0.2-mm diameter (32 islands/hole) at 260°C. Three different mass-flow-rate combinations of CD-PTT and m-iPP were used. The extruded conjugated filaments were quenched and subsequently wound to yield the conjugated partially oriented yarn (POY) filaments. Also, single-component CD-PTT POY and m-iPP POY were produced. The take-up speed of the winder for all POY filaments was 2500 m/min. Then, POY filaments were drawn 1.8 times to form fully drawn yarn (FDY) filaments by a drawn-winder machine (Dienes, Berlin, Germany). The draw temperature and take-up speed were 110°C and 300 m/min, respectively. The specification for the FDY filaments was 75d/24f×32 islands. A Cambridge Stereoscan 600 scanning electron microscope (London, UK) was used to examine the morphological structure of the sample, which was sputter-coated with Au to prevent oxidation. Table II shows the compositions of the CD-PTT/m-iPP conjugated filaments.

TABLE II
Compositions of the CD-PTT/m-iPP
Conjugated Filaments

Polymer	CD-PTT/m-iPP blend ratio
Sample 1	100/0
Sample 2	75/25
Sample 3	50/50
Sample 4	25/75
Sample 5	0/100

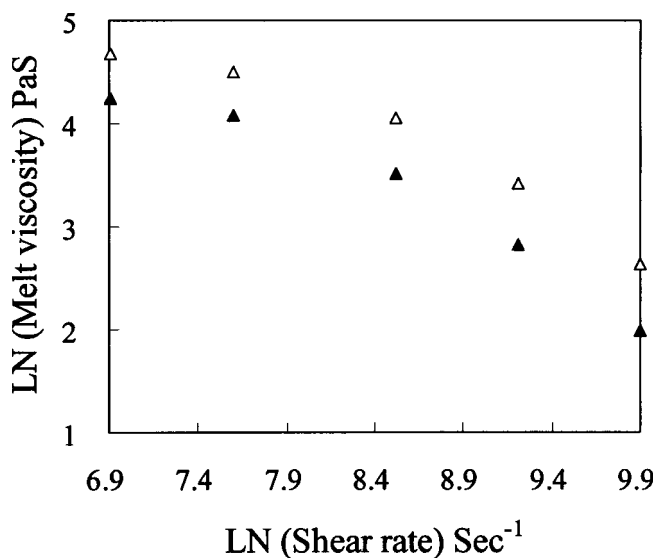


Figure 1 Relation between the melt viscosity and shear rate for CD-PTT and m-iPP polymers at 260°C: (Δ) CD-PTT and (\blacktriangle) m-iPP.

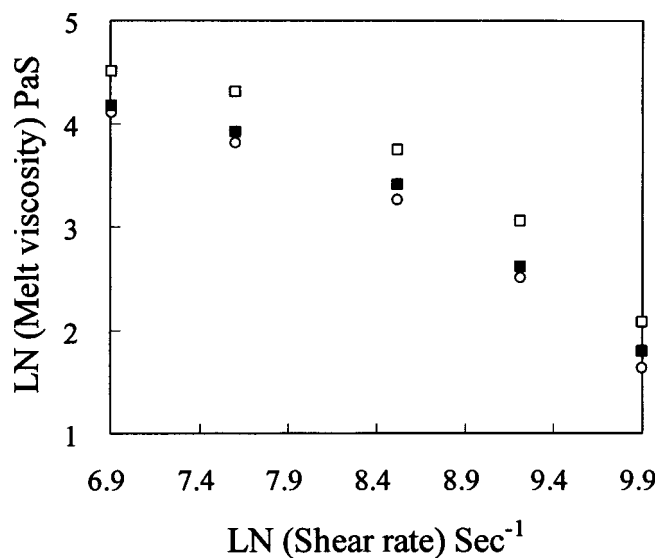


Figure 2 Relation between the melt viscosity and shear rate for CD-PTT/m-iPP polyblended polymers at 260°C: (\square) CD-PTT/m-iPP (75/25), (\circ) CD-PTT/m-iPP (50/50), and (\blacksquare) CD-PTT/m-iPP (25/75).

Measurements

Gel permeation chromatography (GPC) data were measured with a Waters model 510 instrument (Milford, MA). Two solvent systems were used for the GPC measurements. The molecular weight distributions of the CD-PTT and m-iPP polymers were measured in 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP) and trichlorobenzene (TCB) solvent systems, respectively. The intrinsic viscosity of the CD-PTT polymer was obtained with a mixed solution of phenol and tetrachloroethane (60/40) at a concentration of 0.5 g/100 mL and at 30°C.¹⁰ Wide-angle X-ray diffraction (WAXD) studies of the samples were conducted with a MAC Science X-ray unit (Tokyo, Japan) operated at 35 kV and 20 mA. X-ray diffraction was then used with Cu K α radiation and scanning from 5 to 40° (2 θ) at a scan speed of 4°/min.^{11,12} Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) measurements of the samples were made under an atmosphere of N₂ with a PerkinElmer Pyrix-1 (Boston, MA). The heating rate for the TGA measurements was 10°C/min from 30 to 600°C. Both the heating rate and cooling rate were 10°C/min from 30 to 300°C, and the temperature was held for 3 min at 300°C for DSC measurements. The crystallinity of the DSC method was calculated with the following equation: Crystallinity (%) = $\Delta H_m / \Delta H_m^0 \times 100\%$, where ΔH_m and ΔH_m^0 are the heats of fusion (J/g) of the repeating unit for the sample and 100% crystallinity, respectively. The ΔH_m^0 values of pure PTT and pure PP are 145.6 and 209.0 J/g, respectively.^{13,14}

The densities of the samples were determined with the density gradient method. A mixture of *n*-heptane and carbon tetrachloride was used for this purpose.

The densities of the *n*-heptane and carbon tetrachloride solvents were 0.684 and 1.595 g/cm³, respectively. The scale of the density gradient method was prepared from 0.850 to 1.500 g/cm³. The melt viscosities of the samples were measured with a capillary rheometer, and the length-to-diameter ratio of the capillary was 30. The observations of scanning electron microscopy (SEM) pictures were performed with a JEOL 200CX instrument (Tokyo, Japan). All stress-strain data for the samples were obtained on a Zwick 1511 Instron instrument (Zwick, Bamberg, Germany) at an extension rate of 200 mm/min.

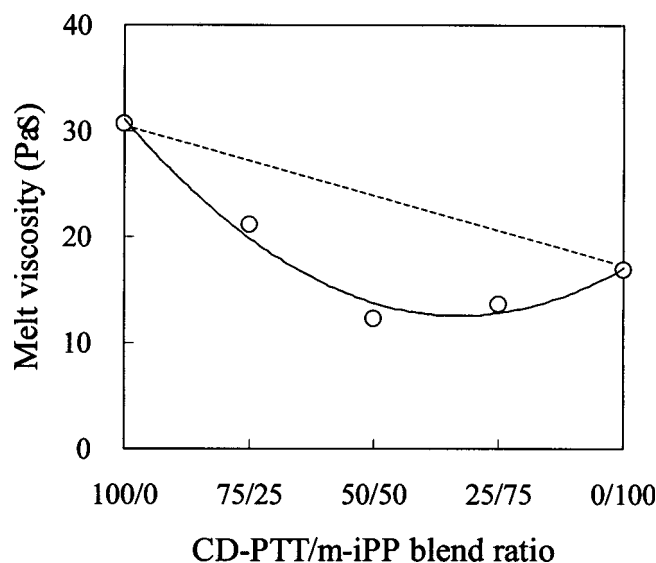


Figure 3 Relation between the melt viscosity and blend ratio for CD-PTT/m-iPP polyblended polymers at 260°C/10,000 s⁻¹: (---) calculated values and (\circ) experimental values.

TABLE III
Thermal Properties of the CD-PTT, m-iPP, and CD-PTT/m-iPP Conjugated Filaments

Polymer	Heating process				Cooling process					
	CD-PTT		m-iPP		CD-PTT			m-iPP		
	T_{m1} (°C)	ΔH_{m1} (J/g)	T_{m2} (°C)	ΔH_{m2} (J/g)	T_{cc1} (°C)	ΔH_{cc1} (J/g)	Half-time (s)	T_{cc2} (°C)	ΔH_{cc2} (J/g)	Half-time (s)
Sample 1	227.6	47.6	—	—	166.5	45.4	89.3	—	—	—
Sample 2	225.2	35.7	155.5	23.4	164.6	34.1	—	103.6	24.1	—
Sample 3	223.1	23.8	154.4	46.9	163.8	22.7	—	102.5	48.2	—
Sample 4	222.7	11.9	153.5	70.3	161.6	11.4	—	101.3	72.3	—
Sample 5	—	—	151.2	93.7	—	—	—	100.2	96.4	52.8

RESULTS AND DISCUSSION

Flow behavior

Figures 1 and 2 present capillary rheological data at temperatures and shear rates applicable to the spinning process. Figure 1 shows the melt viscosities of the CD-PTT and m-iPP polymers at 260°C versus the shear rate. From 1000 to 20,000 s⁻¹, the two polymers exhibited pseudoplastic flow behavior. The rheological curves indicated that the melt viscosity of the CD-PTT polymer was higher than that of the m-iPP poly-

mer over the entire shear-rate range. Figure 2 displays the melt viscosities of the CD-PTT/m-iPP polyblended polymers at 260°C versus the shear rate. The melt viscosities of the CD-PTT/m-iPP polyblended polymers also exhibited pseudoplastic flow behavior. This tendency was independent of the blend ratio.

Figure 3 shows that the melt viscosities of the CD-PTT/m-iPP polyblended polymers exhibited negative-deviation blends (NDBs).^{15–18} Notably, the 50/50 blend of CD-PTT/m-iPP showed a minimum value of the melt viscosity in comparison with that predicted by the additivity rule at 260°C and 10,000 s⁻¹. The rheological results indicated poor interfacial adhesion between the CD-PTT and m-iPP polymers. The melt viscosities of CD-PTT, m-iPP, and their blends followed this order: CD-PTT > CD-PTT/m-iPP (75/25) > m-iPP > CD-PTT/m-iPP (25/75) > CD-PTT/m-iPP (50/50).

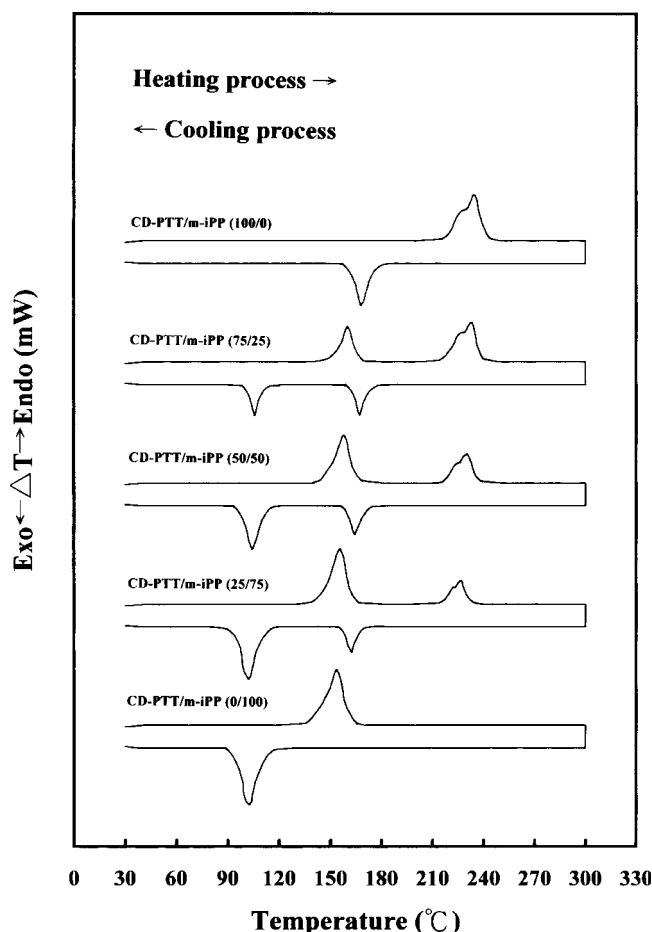


Figure 4 DSC curves of CD-PTT, m-iPP, and CD-PTT/m-iPP polyblended filaments.

Thermal properties and crystallinity

Table III displays the thermal properties of CD-PTT, m-iPP, and three CD-PTT/m-iPP conjugated filaments. In

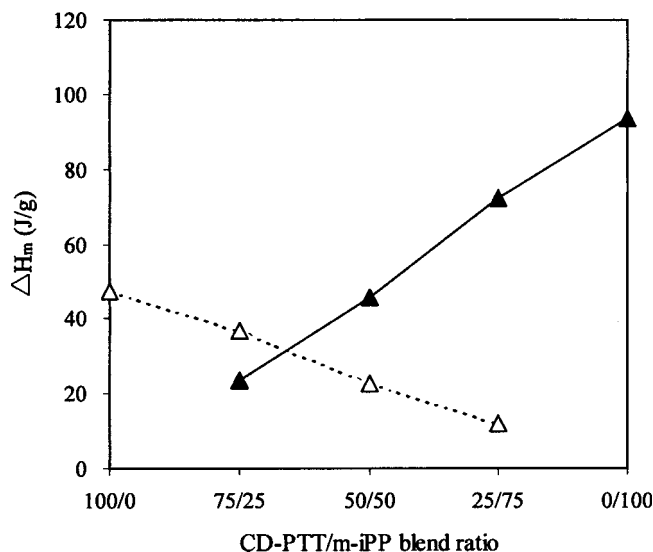


Figure 5 Relation between ΔH_m and the blend ratio of CD-PTT/m-iPP conjugated filaments: (Δ) CD-PTT and (\blacktriangle) m-iPP.

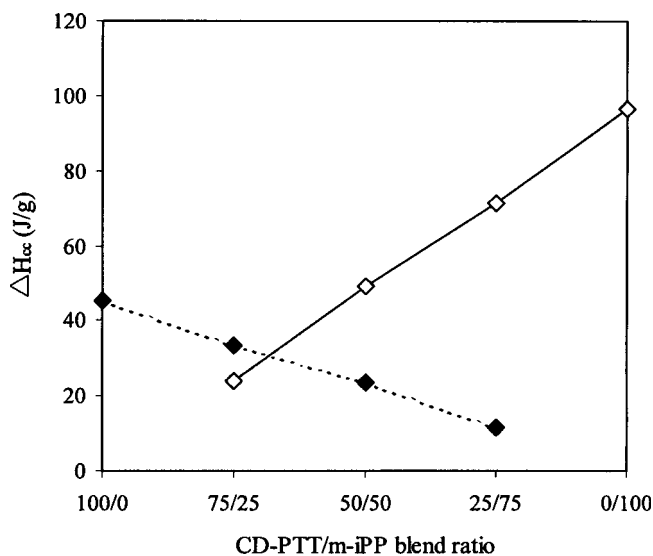


Figure 6 Relation between ΔH_{cc} and the blend ratio of CD-PTT/m-IPP conjugated filaments: (\diamond) CD-PTT and (\blacklozenge) m-iPP.

the DSC heating process, the endothermic peaks of the CD-PTT and m-iPP filaments occurred at 227.6 and 151.2°C, respectively. The endothermic peak [i.e., the melting temperature (T_m)] was due to the melting of the sample. T_m of the CD-PTT and m-iPP filaments was compared with that of the CD-PTT and m-iPP chips, as shown in Tables I and III. Because of the influence of the orientation and crystallization, T_m of the CD-PTT and m-iPP filaments was clearly higher than that of the CD-PTT and m-iPP chips. T_m of the CD-PTT filament was higher than that of m-iPP by approximately 76.4°C. These DSC data displayed a melting endothermic peak, indicating that the CD-PTT and m-iPP filaments were originally crystalline. Figure 4 shows that a double-melting endothermic peak was obtained from the CD-PTT filament, and only melting was observed for the m-iPP filament. Interestingly, large $-\text{SO}_3\text{Na}$ side groups in the CD-PTT polymers prevented the chain molecules from tightly coagulating and obstructed the formation of larger crystals.¹⁹ Because of the effect of a $-\text{SO}_3\text{Na}$ side group on the polymer, the thermal behavior of the CD-PTT/m-iPP polyblended filaments resembled that of the CD-PTT filament. The experimen-

tal phenomenon resembled the observation of Pal et al.²⁰ for cationic dyeable polyester yarns.

In the DSC cooling process, the exothermic peaks (i.e., the cold-crystallization temperatures) of the CD-PTT and m-iPP filaments occurred at 166.5 and 100.2°C, respectively. The exothermic peak was due to the recrystallization behavior of the melting polymer. Obviously, the half-time of the recrystallization for the m-iPP filament was shorter than that of the CD-PTT filament, implying that the recrystallization rate of the m-iPP filament was faster than that of the CD-PTT filament. For all CD-PTT/m-iPP conjugated filaments, the melting temperature of the CD-PTT segments (T_{m1}) nearly did not shift and appeared around 222–225°C. Additionally, the melting temperature of the m-iPP molecules (T_{m2}) also did not clearly change and appeared at 153–156°C. In other words, the T_m change was independent of the blend ratio.

Figure 5 displays linear variations of ΔH_m with the blend ratio of the CD-PTT/m-iPP conjugated filaments. The heat of fusion of the CD-PTT molecules (ΔH_{m1}) proportionally declined with increasing m-iPP content. Meanwhile, the heat of fusion of the m-iPP molecules (ΔH_{m2}) proportionally increased with increasing m-iPP content. For all CD-PTT/m-iPP conjugated filaments, the cold-crystallization temperature of the CD-PTT molecules (T_{cc1}) also nearly did not change and appeared around 161–165°C. The cold-crystallization temperature of the m-iPP molecules (T_{cc2}) was observed around 101–104°C. Figure 6 also shows the linear variations of the recrystallization heat of the exothermic peak (ΔH_{cc}) with the blend ratios of CD-PTT/m-iPP conjugated filaments. The recrystallization heat of the exothermic peak of the CD-PTT molecules (ΔH_{cc1}) proportionally decreased with increasing m-iPP content. Meanwhile, the recrystallization heat of the exothermic peak of the m-iPP molecules (ΔH_{cc2}) proportionally increased with increasing m-iPP content. The DSC experimental results indicated that CD-PTT segments and m-iPP molecules were an immiscible system.

The heat of fusion was used to indicate the crystalline fraction of the material. A higher value of the heat of fusion was expected to result in a higher crystallinity. Table IV reveals the crystallinities of CD-PTT,

TABLE IV
Crystallinities of the CD-PTT, m-iPP, and CD-PTT/m-iPP Conjugated Filaments

Polymer	DSC method				Total X_c (%) of the filament	
	CD-PTT part		m-iPP part		DSC method	WAXD method
	ΔH_{m1} (J/g)	X_c (%) of CD-PTT	ΔH_{m2} (J/g)	X_c (%) of m-iPP		
Sample 1	47.6	32.7	—	—	32.7	31.3
Sample 2	35.7	24.5	23.4	11.2	35.7	33.4
Sample 3	23.8	16.3	46.9	22.4	38.8	36.8
Sample 4	11.9	8.2	70.3	33.6	41.8	40.5
Sample 5	—	—	93.7	44.8	44.8	43.6

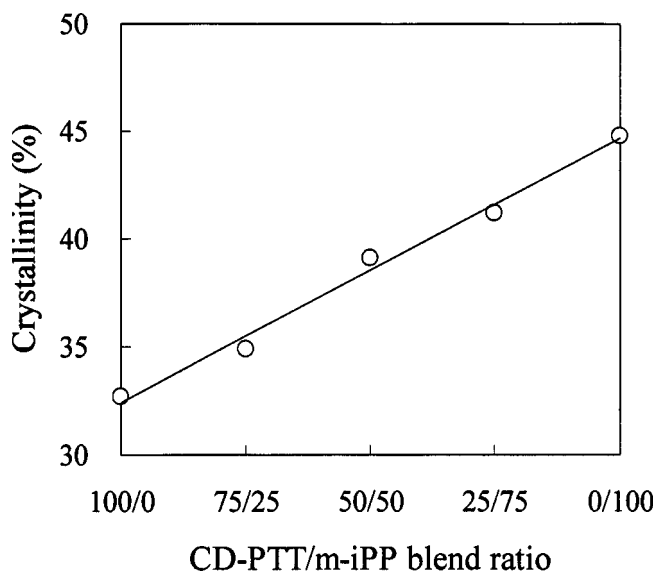


Figure 7 Relation between the crystallinity and blend ratio of CD-PTT/m-iPP conjugated filaments.

m-iPP, and CD-PTT/m-iPP conjugated filaments. The heat of fusion of melting was used to calculate the crystallinity. Values of 145.6 and 209.0 J/g corresponding to 100% crystallinity were used for pure PTT and pure PP, respectively. The crystallinities of CD-PTT and m-iPP filaments were 32.7 and 44.8%,

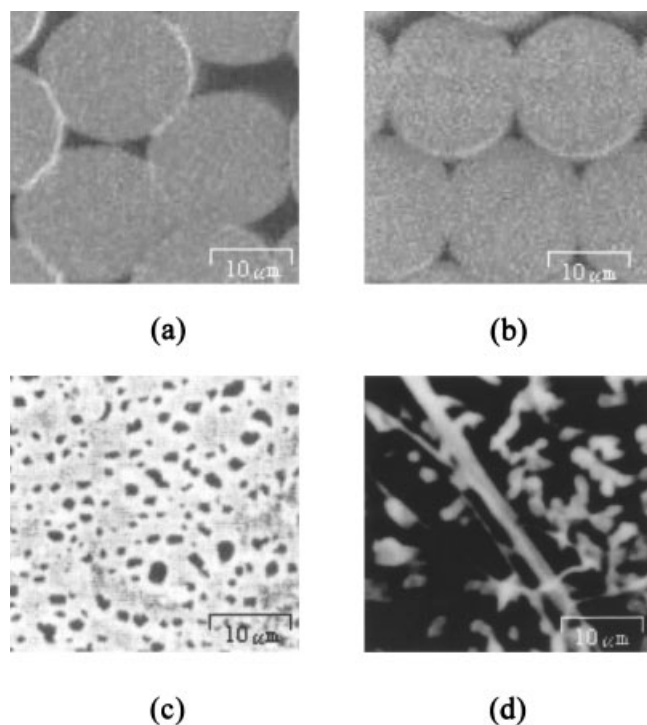


Figure 8 Morphologies of CD-PTT, m-iPP, and CD-PTT/m-iPP conjugated filaments: (a) CD-PTT/m-iPP (100/0), (b) CD-PTT/m-iPP (0/100), (c) CD-PTT/m-iPP (50/50) after an HFIP solvent treatment, and (d) CD-PTT/m-iPP (50/50) after a decalin solvent treatment.

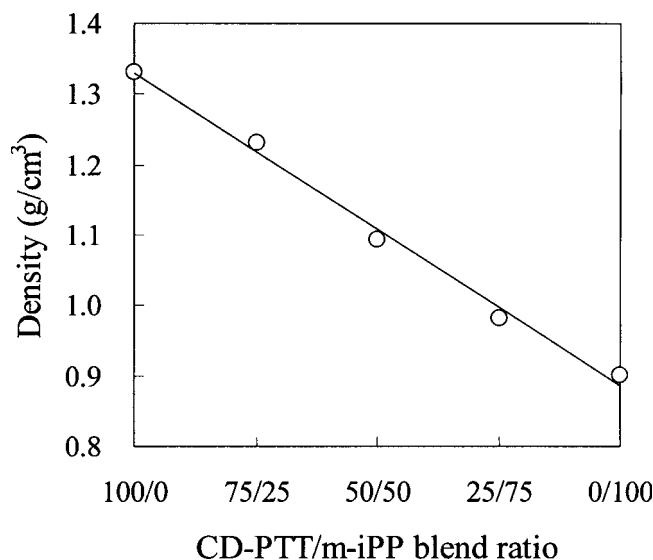


Figure 9 Relation between the density and blend ratio of CD-PTT/m-iPP conjugated filaments.

respectively. The crystallinity of the m-iPP filament was higher by 12.1% than that of the CD-PTT filament. After blending, the total crystallinity of the CD-PTT/m-iPP conjugated filaments increased as the proportion of m-iPP increased. The crystallinities by the WAXD method were slightly lower than those by the DSC method. Figure 7 displays the relation between the crystallinities and blend ratios of CD-PTT/m-iPP conjugated filaments. For all CD-PTT/m-iPP conjugated filaments, the experimental crystallinities agreed fairly well with the calculated values. This indicated that CD-PTT and m-iPP formed an immiscible system.

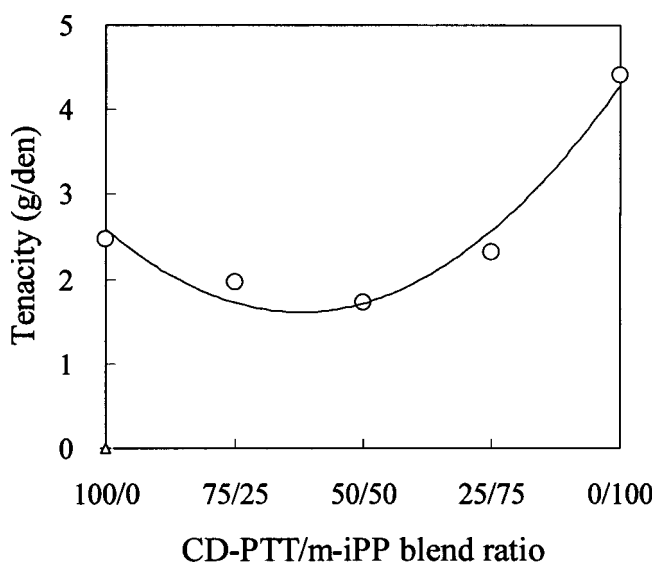


Figure 10 Relation between the tenacity and blend ratio of CD-PTT/m-iPP conjugated filaments.

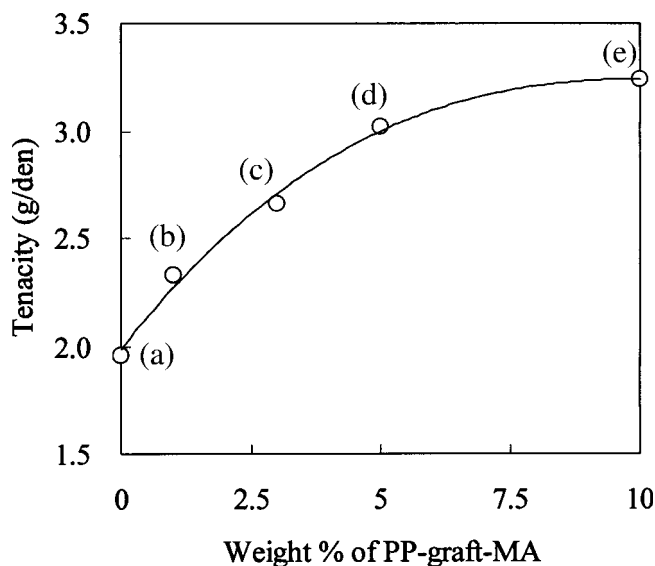


Figure 11 Effect of the concentration of PP-graft-MA on the tenacities of CD-PTT (75)/m-iPP (25) conjugated filaments: (a) no PP-graft-MA, (b) 1 wt % PP-graft-MA, (c) 3 wt % PP-graft-MA, (d) 5 wt % PP-graft-MA, and (e) 10 wt % PP-graft-MA.

Morphology

Morphological observations revealed that the blends had a dispersed phase structure. As shown in Figure 8(a,b), both the CD-PTT and m-iPP filaments had a uniform surface structure in cross sections. As shown in Figure 8(c,d), a pore/filament morphology of a larger size (0.5–3 μm in diameter) was observed after an HFIP (CD-PTT was removed)/decalin (m-iPP was removed) treatment in a cross section of the CD-PTT/m-iPP(50/50) conjugated filament. The morphological results could imply that CD-PTT and m-iPP could be divided into two parts. Meanwhile, the CD-PTT and m-iPP polymers were identified as an immiscible system.

Density and tenacity

Figure 9 displays the relation between the densities and blend ratios of CD-PTT/m-iPP conjugated filaments. For all CD-PTT/m-iPP conjugated filaments, the experimental densities agreed fairly well with the calculated values. This indicated that CD-PTT and m-iPP formed an immiscible system. Figure 10 shows the relation between the tenacities and the blend ratios of CD-PTT/m-iPP conjugated filaments. The tenacities of the CD-PTT/m-iPP conjugated filaments declined initially and then increased with the proportion of m-iPP. Obviously, the 50/50 CD-PTT/m-iPP blend displayed a minimum value. The poor interfacial interactions between CD-PTT and m-iPP indicated poor mechanical properties. The tendency agreed with the flow behavior, and the 50/50 CD-

PTT/m-iPP blend displayed a minimum tenacity value. The experimental results for the tenacities were consistent with the thermal behavior, flow behavior, crystallinity, tenacity, and morphologies from SEM pictures of the CD-PTT/m-iPP conjugated filaments.

Figure 11 displays the variation of the tenacities of CD-PTT (75)/m-iPP (25) as a function of PP-graft-MA. The compatibilizer acted as an interfacial emulsifying agent. As for the tenacities, the addition of PP-graft-MA increased the tenacities considerably, and the maximum value for the tenacity was found for the blend containing 10 wt % compatibilizer. Here, a 65% increase in the tenacity was noticed. The reason for the increase in the tenacity with the addition of the compatibilizer is clear. The compatibilization effect between CD-PTT and PP-graft-MA was based on the reaction between $-\text{OH}$ groups of CD-PTT and MA groups in PP-graft-MA.^{21,22}

CONCLUSIONS

A CD-PTT polymer possesses 5-SSDMI ($-\text{SO}_3\text{Na}$ groups) functional groups. Furthermore, an m-iPP polymer does not possess any functional groups. The interfacial adhesion plays a critical role for CD-PTT/m-iPP conjugated filaments. The flow behavior of CD-PTT/m-iPP polyblended polymers exhibited NDBs. A 50/50 blend of CD-PTT and m-iPP showed a minimum value of the melt viscosity in comparison with that predicted by the additivity rule. The rheological results indicated poor interfacial adhesion between the CD-PTT and m-iPP polymers. The experimental results of DSC indicated that CD-PTT and m-iPP molecules easily formed individual domains. In particular, a double endothermic peak was observed from CD-PTT and CD-PTT/m-iPP conjugated filaments. The tenacities of the CD-PTT/m-iPP conjugated filaments declined initially and then increased with the proportion of m-iPP. Obviously, the 50/50 CD-PTT/m-iPP blend displayed a minimum value. The experimental crystallinities and densities agreed fairly well with the calculated values. This indicated that CD-PTT and m-iPP formed an immiscible system. The morphological result could imply that CD-PTT and m-iPP could be divided into two parts. From the flow behavior, thermal behavior, crystallinity, mechanical properties, density measurements, and morphological observations of the conjugated filaments, the CD-PTT/m-iPP polymers were identified as immiscible systems. The presence of the PP-graft-MA compatibilizer improved the tenacity considerably. Blends with 10 wt % compatibilizer exhibited the maximum improvement in the tenacity. The compatibilization effect between CD-PTT and PP-graft-MA was based on the reaction between $-\text{OH}$ groups of CD-PTT and MA groups in PP-graft-MA.

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