

Thermal Behavior and Morphology of Polycaproamide (PCA)/Cationic Dyeable Polycaproamide (CD-PCA) Polyblended Filaments

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ABSTRACT: Polycaproamide (PCA) polymer and cationic dyeable polycaproamide (CD-PCA) polymer were blended mechanically in the proportions of 75/25, 50/50, and 25/75 in a melt twin-screw extruder to prepare three PCA/CD-PCA polyblended polymers. Blends of PCA and CD-PCA were spun into filaments. The molar ratio of 5-sodium sulfonate dimethyl isophthalate (5-SSDMI) for CD-PCA polymer was 1.99%. This study investigated the physical properties of PCA/CD-PCA polyblended filaments using gel permeation chromatograph (GPC), nuclear magnetic resonance (NMR), gas chromatography (GC), potentiometer, thermogravimetric analysis (TGA), a rheometer, differential scanning calorimetry (DSC), the density gradient method, wide-angle X-ray diffraction (WAXD), extension stress-strain measurement, and scanning electron microscope (SEM). Flow behavior of PCA/CD-PCA polyblends exhibited positive-deviation blends (PDB), and the 50/50 blend of PCA/CD-PCA showed a maximum value of the melt vis-

cosity. Experimental results of the DSC indicated PCA and CD-PCA molecules easily formed miscible domains. The crystallinities of PCA/CD-PCA polyblended filaments decreased as the 5-SSDMI content increased. Tenacities/densities of PCA/CD-PCA polyblended filaments were also found to decline as the 5-SSDMI content increased. The surface of PCA/CD-PCA polyblended filament exhibited a uniform morphology from the SEM data. PCA and CD-PCA polymers were proved to be a compatible system. The weight loss percentages of the PCA/CD-PCA polyblended filaments increased as the 5-SSDMI content increased in aqueous NaOH solution. The porous morphology of a larger size from 0.01 to 2 μm in diameter was observed after alkali treatment of PCA/CD-PCA polyblended filament. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 97: 1220–1226, 2005

Key words: PCA; CD-PCA; 5-SSDMI; polyblend; crystallinity; morphology

INTRODUCTION

The applications of polyblends are important developments of the plastics 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 upon the morphology of the blended materials. The morphology resulting from a blending process depends mainly upon the rheological and interfacial properties of the molten components, the blending conditions, and the weight ratio of the polymers blended.^{1–3} Polyblends can be characterized by their phase behavior as being 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 polycaproamide (PCA) with other polymers such as poly(ethylene terephthalate) (PET), cationic dyeable poly(ethylene terephthalate) (CD-PET), poly(butylene terephthalate) (PBT), polyethylene (PE), and polypropylene (PP).^{5–13} PCA polymer possesses amide functional groups (–NHCO– groups). Furthermore, CD-PCA polymer possesses not only amide functional groups, but also 5-sodium sulfonate dimethyl isophthalate functional groups (–SO₃Na groups; 5-SSDMI). The interfacial adhesion plays a critical role for PCA/CD-PCA polyblended filaments. This study thoroughly investigated the flow behavior, thermal behavior, crystallinity, mechanical property, and morphology of PCA/CD-PCA polyblended filaments.

EXPERIMENTAL

Materials

Polycaproamide (PCA) chips were purchased from the Rhodia Engineering Plastics Co., Ltd. in Taipei, Taiwan. Cationic dyeable polycaproamide (CD-PCA)

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TABLE I
Characteristics of PCA and CD-PCA Polymers

Polymer	5-SSDMI (mol %)	R.V. ^a	\bar{M}_w (g/mol)	\bar{M}_w/\bar{M}_n (MWD) ^b	R-COOH (meq/kg)	T _m (°C)	T _{d5} ^c (°C)
PCA	0	2.55	32,227	2.35	50.6	220.8	320
CD-PCA ^d	1.99 ^e	2.39	28,089	2.66	42.2	208.7	312

^a Relative viscosity of polymer was obtained by a solution of 1.0 g/dl in 85% formic acid at 20°C.

^b Molecular weight distribution of polymer.

^c Degradation temperature of the weight loss percentage at 5%.

^d Synthetic torque of the CD-PCA polymer was controlled with the same as that of the PCA polymer in the reaction stop step.

^e Data was measured from NMR measurement.

chips were obtained from the Shang Chen Glass Products Co., Ltd. in Dongguan, China. Table I lists the synthetic characteristics of PCA and CD-PCA polymers. PCA and CD-PCA polymers were blended mechanically in the proportions of 75/25, 50/50, and 25/75 in a melt twin-screw extruder to prepare three PCA/CD-PCA polyblended polymers. Prior to blending, PCA and CD-PCA were dried for 24 h at 105°C. Melt spinning was carried out using a capillary rheometer. Undrawn yarns (UDY yarns) were extruded at 260°C, and at a constant wind speed of 600 m/min were taken up for all samples. Then, UDY yarns were drawn 2.5 times to form fully oriented yarns (FOY yarns) by a drawn-winder machine. The draw temperature and take-up speed were 110°C and 300 m/min, respectively. The specification of FOY is 30 d/10f (3dpf). Table II shows the compositions of the PCA/CD-PCA polyblended filaments.

Measurements

Gel permeation chromatograph (GPC) data were measured from a Waters Model 510 Type. Relative viscosities of PCA and CD-PCA chips were obtained by a solution of 1.0 g/dL in 85% formic acid at 20°C.¹⁴ The 5-SSDMI content of CD-PCA polymer was obtained by a Bruker AM-400 nuclear magnetic resonance (NMR) spectrometer.

Wide-angle X-ray diffraction (WAXD) studies of samples were conducted using a MAC Science X-ray unit operated at 35kV and 20 mA. X-ray diffraction

was then used with Cu K α radiation and scan from 5 to 35° (2 θ) with a scan speed of 4°/min. The crystallinity of WAXD method is given by the follow expression: crystallinity (%) = C/(C+A) \times 100 (%), where C is the area under the crystalline curve and A is the area under the amorphous curve.¹⁵⁻¹⁷

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) measurements of samples were made using a Perkin-Elmer Pyrix-1. The heating rate of TGA measurement 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 measurement. The crystallinity of the DSC method was calculated from the following equation: crystallinity (%) = $\Delta H_m / \Delta H_m^0 \times 100$ (%), where ΔH_m and ΔH_m^0 are the heat of fusion in J/g of repeating unit for the sample and 100% crystallinity, respectively. ΔH_m^0 of PCA is equal to 230.1 J/g.¹⁸ The densities of samples were determined using the density gradient method. A mixture of *n*-heptane and carbontetrachloride was used for the purpose. Densities of *n*-heptane and carbontetrachloride solvents were 0.684 g/cm³ and 1.595 g/cm³, respectively. The scale of density gradient method was prepared from 1.100 g/cm³ to 1.200 g/cm³. The crystallinity was calculated from the following equation: crystallinity (%) = $\rho_c(\rho - \rho_a) / \rho(\rho_c - \rho_a) \times 100$ (%), where ρ_c is the crystalline density and ρ_a is the amorphous density, 1.230 g/cm³ and 1.084 g/cm³ were used as their values, respectively.¹⁹

The melt viscosities of samples were measured by a capillary rheometer, and the length-to-diameter ratio of the capillary is 30. The observations of scanning electron microscope (SEM) pictures were performed from a JEOL-200CX Type. All stress-strain data of samples were obtained on a Zwick 1511 type Instron instrument at an extension rate of 10 mm/min. The specimen was 50 mm long, 10 mm wide, and 0.5 mm thick. The measured weight lost by the samples after treatment with alkali was calculated as follows: weight loss (%) = $(W_1 - W_2) / W_1 \times 100$ (%), where W_1 and W_2 were the weight of the samples before and after alkaline hydrolysis, respectively.²⁰

TABLE II
Compositions of the PCA/CD-PCA Polyblended Filaments

Polymer code	PCA/CD-PCA blend ratio	5-SSDMI content (mol %)
Sample 1	100/0	0
Sample 2	75/25	0.5
Sample 3	50/50	1.0
Sample 4	25/75	1.5
Sample 5	0/100	2.0

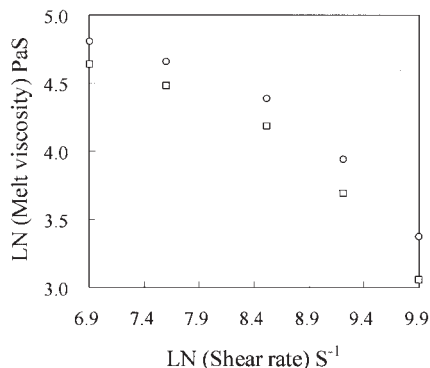


Figure 1 Relation between the melt viscosities and shear rate for PCA and CD-PCA polymers at 260°C: (○) PCA; (□) CD-PCA.

RESULTS AND DISCUSSION

Flow behavior

Figure 1 shows the melt viscosities of PCA and CD-PCA polymers at 260°C versus shear rate. From 1000 to 20,000 S^{-1} , two polymers exhibited pseudoplastic flow behavior. The rheological curves indicated the melt viscosity of the PCA polymer was higher than that of the CD-PCA polymer over the entire shear rate range. The melt viscosity of polymer was deeply influenced by the molecular weight of polymer. In this work, the synthetic torque of the CD-PCA polymer was controlled the same as that of the PCA polymer in the reaction stop step. Table I reveals that the weight-average molecular weight (\bar{M}_w) and the T_m of the CD-PCA polymer were lower than those of the PCA polymer. Therefore, the melt viscosity of the CD-PCA polymer was lower than that of the PCA polymer at the same rheological condition.

Figure 2 displays the melt viscosities of PCA/CD-PCA polyblended polymers at 260°C versus shear rate. The melt viscosities of PCA/CD-PCA poly-

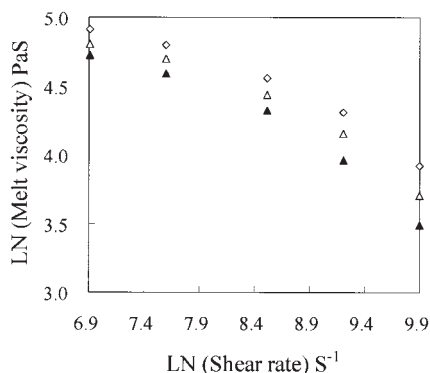


Figure 2 Relation between the melt viscosities and shear rate for PCA/CD-PCA polyblended polymers at 260°C: (△) PCA/CD-PCA (75/25); (◇) PCA/CD-PCA (50/50); (▲) PCA/CD-PCA (25/75).

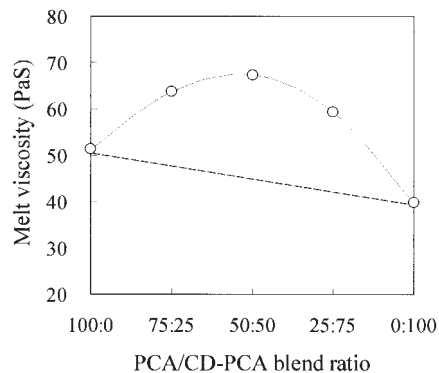


Figure 3 Relation between the melt viscosities and blend ratio for PCA/CD-PCA polyblended polymers at 260°C/10,000 S^{-1} ; —: Calculated value; ○: Experimental value.

blended polymers also exhibited pseudoplastic flow behavior. This tendency was independent of the blend ratio. Figure 3 shows that the melt viscosities of PCA/CD-PCA polyblended polymers exhibited positive-deviation blends (PDB).^{21–25} Clearly, the 50/50 blend of PCA/CD-PCA showed a maximum value of the melt viscosity than that predicted by the additivity rule. The rheological results displayed a good interfacial adhesion between the PCA and CD-PCA polymers. This trend is enough to prove compatibility between the PCA and CD-PCA polymer. The melt viscosities of PCA, CD-PCA, and their blends followed the order Sample 3>Sample 2>Sample 4>Sample 1>Sample 5.

Thermal behavior and crystallinity

Table III and Figure 4 display the thermal properties of PCA, CD-PCA, and three PCA/CD-PCA polyblended filaments. In the DSC heating process, the endothermic peaks of the PCA and CD-PCA filaments occurred at 223°C and 212°C, respectively. The endothermic peak (T_m) is due to the melting of the sample. The T_m of the PCA/or CD-PCA filament is compared with that of the PCA/or CD-PCA chip, as seen between Table I and Table III. Because of the influences of the orientation and crystallization, the T_m of the PCA/or CD-PCA filament was clearly higher than that of the PCA/or CD-PCA chip. The T_m of the CD-PCA filament was lower than that of the PCA at approximately 11°C. Particularly, a double endothermic peak was observed from the CD-PCA filament and only a single melting peak was obtained for the PCA filament. The double endothermic peak of CD-PCA filament included the chief peak (212°C) and the shoulder peak (209°C). The occurrence of the shoulder peak for the CD-PCA filament resulted from a large $-SO_3Na$ side group in the CD-PCA filament preventing the chain molecules from crystallizing close to each other and making the formation of two crystalline particles. The experimental phenomenon resem-

TABLE III
Thermal Properties of PCA, CD-PCA, and PCA/CD-PCA Polyblended Filaments

Polymer code	Heating process				Cooling process		
	T_g (°C)	T_m (°C)	ΔH_m (J/g)	X_c (%)	T_{cc} (°C)	ΔH_{mc} (J/g)	X_{cc} (%)
Sample 1	60.5	222.6	70.2	30.5	185.1	62.8	27.3
Sample 2	59.6	219.9	67.8	29.5	177.2	60.9	26.5
Sample 3	58.7	217.1	65.4	28.4	169.3	59.0	25.6
Sample 4	57.7	214.4	63.0	27.4	161.3	57.0	24.8
Sample 5	56.8	211.6	58.2	25.3	153.4	53.2	23.1

bled the observation of S. K. Pal et al. for cationic dyeable polymer.²⁶ The glass transition temperature (T_g) is related to the initiation of the micro-Brownian motion of the amorphous chains and is for all samples between 57 and 61°C. Clearly, the T_g of the PCA/CD-PCA polyblended filaments declined as the 5-SSDMI content increased.

The crystallinities of PCA, CD-PCA, and PCA/CD-PCA polyblended filaments were determined using DSC, the density gradient, and WAXD methods. In the DSC method, the heat of fusion was used to indicate the crystalline fraction of the material. A higher value of heat of fusion was expected to relate to a higher crystallinity. Table IV reveals the crystallinities of PCA, CD-PCA, and their blends. Notably, increasing the 5-SSDMI content in the PCA/CD-PCA polyblended filaments reduced the heat of fusion, and thus, decreased the crystallinity. The data obtained by the DSC method was consistent with that obtained by the density gradient method and the WAXD method.

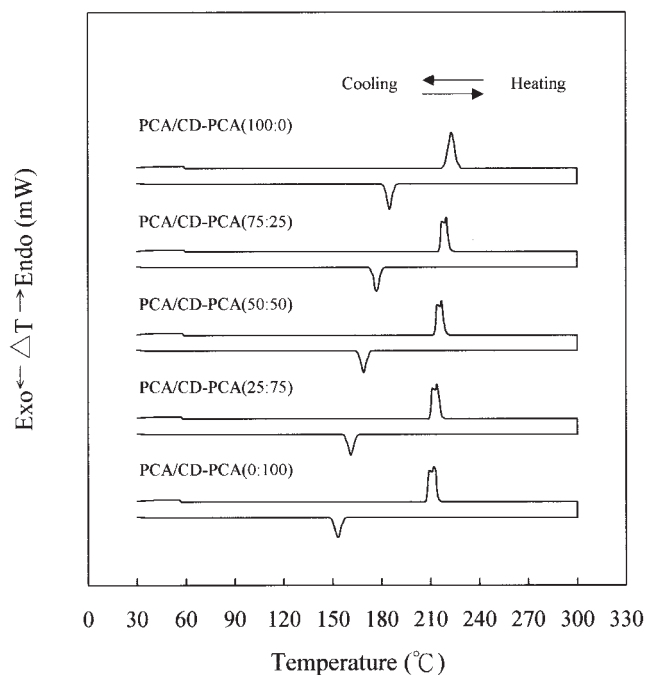


Figure 4 DSC curves of PCA, CD-PCA, and PCA/CD-PCA polyblended filaments.

The degree of crystallinity was compared from DSC, the density gradient, and WAXD methods for all samples. According to the comparison, quantifying the crystallinity by the DSC method and the density gradient method was difficult. In the DSC analysis, the heat of fusion of repeating unit was theoretically computed for 100% crystallinity of the PCA polymer. Regarding the density gradient method, the density of crystalline and amorphous PCA polymer could not be measured accurately, and literature values for crystalline and amorphous density were assumed theoretically as well. Therefore, the crystallinity of the WAXD method was often used in the quantitative analysis of the crystallinity for polymer. Meanwhile, the crystallinity of the WAXD method was slightly lower than that of the DSC method and the density gradient method.

Figure 5 reveals that the crystalline peak of the PCA filament was sharper than that of the CD-PCA filament. Meanwhile, the peaks of diffracting angles (2θ) for (200) and (002,202) reflecting planes in equator direction, which are 20.5° and 24.0°, did not clearly shift except for the diffraction intensities. These peaks are the equivalent reflections of the PCA polymer α phase.^{27–29} In the DSC cooling process, the crystallization exothermic peak (T_{cc}) of the PCA filament was higher than that of the CD-PCA filament. This implies that the crystallization rate of the PCA filament was faster than that of the CD-PCA filament. Because of the influence of a $-\text{SO}_3\text{Na}$ side group on the polymer, the thermal behavior of PCA/CD-PCA polyblended filaments resembled that of the CD-PCA filament.

TABLE IV
Crystallinities of PCA, CD-PCA, and PCA/CD-PCA Polyblended Filaments

Polymer code	DSC		Density		WAXD
	ΔH_m (J/g)	X_c (%)	ρ (g/cm ³)	X_c (%)	X_c (%)
Sample 1	70.2	30.5	1.1306	34.7	29.3
Sample 2	67.8	29.5	1.1288	33.5	28.3
Sample 3	65.4	28.4	1.1270	32.2	27.2
Sample 4	63.0	27.4	1.1253	30.9	26.2
Sample 5	58.2	25.3	1.1217	28.3	24.1

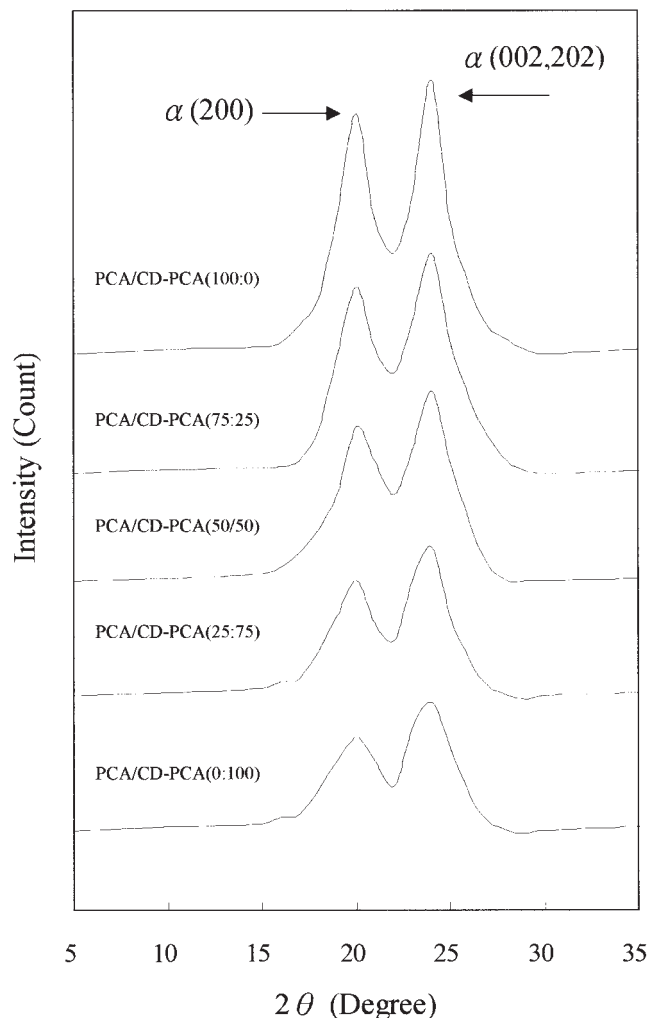


Figure 5 WAXD curves of PCA, CD-PCA, and PCA/CD-PCA polyblended filaments in the equator direction.

Mechanical property and density measurement

Figure 6 shows the relation between the tenacity/density and the blend ratio of PCA/CD-PCA polyblended filaments. The tenacities/densities of PCA/CD-PCA polyblended filaments were found to decline as the proportion of CD-PCA content increased. Ther-

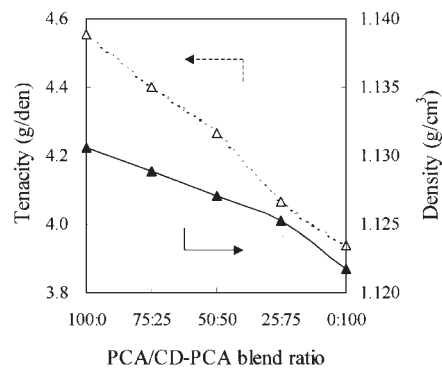


Figure 6 Relation between the tenacities/densities and blend ratio for PCA/CD-PCA polyblended filaments: (Δ) Tenacity; (▲) Density.

mal behavior showed that the crystallinity decreased with an increasing 5-SSDMI content. Adding a compound containing the $-\text{SO}_3\text{Na}$ side group disturbed the regularity of the CD-PCA polymer segments and made the structure of the CD-PCA filament less compact than that of the PCA filament. This physical property was reflected in the lower tenacity/density of the CD-PCA filament. Experimental results also indicated the trend that the tenacity/density of the CD-PCA filament was lower than that of the PCA filament. The phenomenon is due to the effect of a higher 5-SSDMI amount.

Solubility and morphology

Table V displays the solubility of PCA, CD-PCA, and PCA/CD-PCA polyblended filaments. All the former samples were dissolved into formic acid (85%), *m*-cresol, and conc. sulfuric acid (98%) solvents. Figure 7 shows the weight loss percentage of PCA and CD-PCA filaments respectively, on treatment with 0.5, 1.0, 1.5, 2.0, and 2.5% aqueous sodium hydroxide (NaOH) solution at 100°C for various time periods. Clearly, the weight loss percentage of the PCA filament hardly decreased in aqueous NaOH solution. The weight loss percentage of the CD-PCA filament was higher than

TABLE V
Solubility^a of PCA, CD-PCA, and PCA/CD-PCA Polyblended Filaments

Polymer code	HCOOH (85%)	<i>m</i> -Cresol	conc. H ₂ SO ₄ (98%)	NaOH (2.5 g/L)	NMP ^b	Py ^b	DMF ^b	DMAc ^b	DMSO ^b	Toluene
Sample 1	+	+	+	+ -	-	-	-	-	-	-
Sample 2	+	+	+	+ -	-	-	-	-	-	-
Sample 3	+	+	+	+ -	-	-	-	-	-	-
Sample 4	+	+	+	+ -	-	-	-	-	-	-
Sample 5	+	+	+	+ -	-	-	-	-	-	-

^a Solubility: +, soluble at room temperature; + -, partially soluble at 100°C/120 min; -, insoluble.

^b NMP: *N*-methyl-2-pyrrolidone; Py: pyridine; DMF: *N,N*-dimethylformamide; DMAc: *N,N*-dimethylacetamide; DMSO: dimethyl sulfoxide.

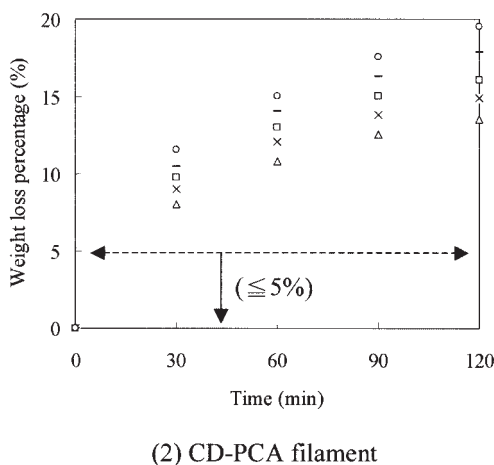
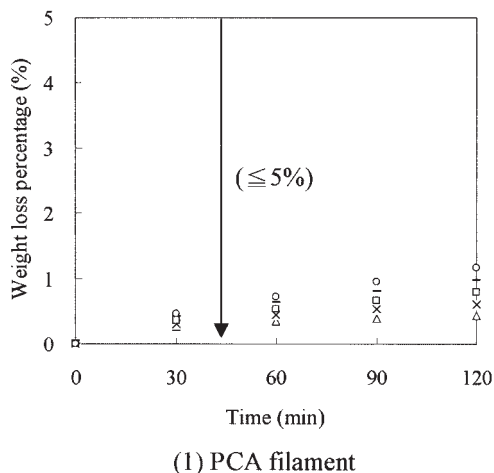


Figure 7 Weight loss percentages of (1) PCA and (2) CD-PCA filaments at 100°C: (Δ) 0.5% [NaOH]; (×) 1.0% [NaOH]; (□) 1.5% [NaOH]; (–) 2.0% [NaOH]; (○) 2.5% [NaOH].

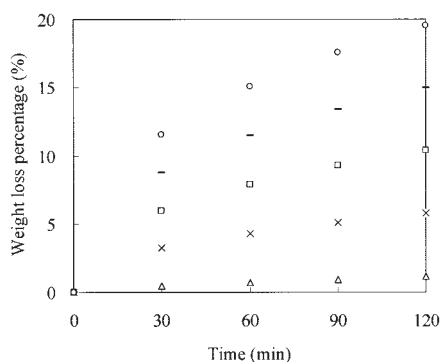


Figure 8 Relation between the weight loss percentages and time of PCA/CD-PCA polyblended filaments at 2.5% [NaOH] and 100°C: (Δ) PCA/CD-PCA (100/0); (×) PCA/CD-PCA (75/25); (□) PCA/CD-PCA (50/50); (–) PCA/CD-PCA (25/75); (○) PCA/CD-PCA (0/100).

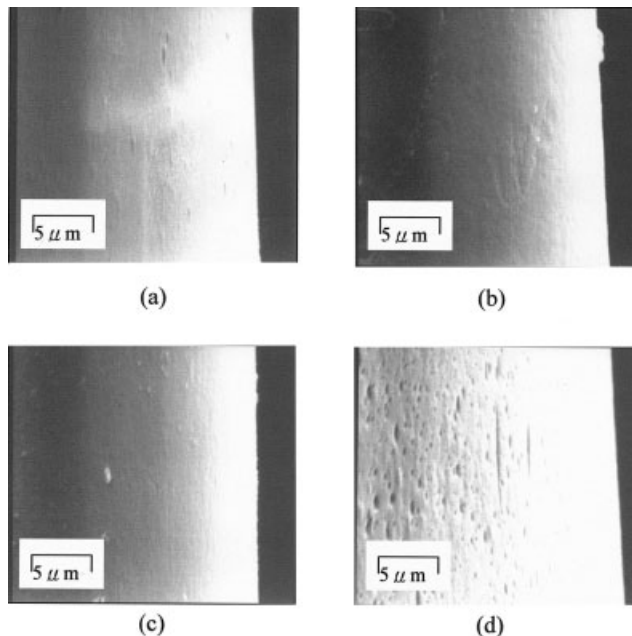


Figure 9 Morphologies of PCA, CD-PCA, and PCA/CD-PCA polyblended filaments: (a) PCA/CD-PCA (100/0); (b) PCA/CD-PCA (0/100); (c) PCA/CD-PCA (50/50)-before alkali treatment; (d) PCA/CD-PCA (50/50)-after alkali treatment.

that of the PCA filament. Figure 8 summarizes the weight loss percentage of PCA/CD-PCA polyblended filaments on treatment with 2.5% [NaOH] at 100°C for various time intervals. Obviously, the weight loss percentage of PCA/CD-PCA polyblended filaments increased with an increasing 5-SSDMI content in aqueous NaOH solution.

The DSC results indicated PCA and CD-PCA segments easily formed miscible domains. As seen in Figure 9, both PCA and CD-PCA filaments possessed not only uniform surfaces, but also their blends exhibited smooth surfaces with increasing the amount of the CD-PCA polymer. The SEM observations of morphologies were consistent with the DSC results for PCA/CD-PCA polyblended filaments. The porous morphology of a larger size from 0.01 to 2 μm in diameter was observed after alkali treatment (2.5% [NaOH], 100°C, 120 min) of PCA/CD-PCA polyblended filament (Sample 3).

CONCLUSION

The combination of amide chains in PCA polymer and amide/5-SSDMI segments in CD-PCA polymer functional groups was found to be a good interfacial adhesion between PCA and CD-PCA segments. Flow behavior of PCA/CD-PCA polyblended polymers exhibited positive-deviation blends (PDB). The 50/50 blend of PCA/CD-PCA showed a maximum value of the melt viscosity than that predicted by the additivity

rule. Rheological results indicated a good interfacial adhesion between PCA and CD-PCA polymers. The experimental results of the DSC indicated PCA and CD-PCA molecules easily formed miscible domains. The crystallinities of PCA/CD-PCA polyblended filaments declined as a 5-SSDMI content increased. Tenacities/densities of PCA/CD-PCA polyblended filaments were also found to decrease as the 5-SSDMI content increased. The surfaces of PCA/CD-PCA polyblended filaments exhibited uniform morphologies from the SEM data. From the flow behavior, thermal behavior, mechanical property, density measurement, and the SEM observation of the morphology, PCA/CD-PCA polyblended polymers were proved to be a compatible system. The weight loss percentage of the PCA/CD-PCA polyblended filaments increased as the 5-SSDMI content increased in aqueous NaOH solution. The porous morphology of a larger size from 0.01 to 2 μm in diameter was observed after alkali treatment of PCA/CD-PCA polyblended filament.

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