

Polystyrene/Bisphenol A Polycarbonate (PS/PC) Molecular Composite by *In Situ* Polymerization. II. Effect of Different Molecular Size of PC on the Blending Behavior and Properties

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ABSTRACT: The influences of different molecular sizes and contents of bisphenol A polycarbonate (PC) on the miscibility, morphology, and physical properties of a polystyrene/PC (PS/PC) molecular composite are examined. The polymerization of the matrix monomer, styrene, which is completely miscible with PC, affords an apparently homogeneous mixture that remains stable on heating to above the glass transition temperature (T_g) of the pure matrix and solubilizes in common organic solvents. Both the molecular and material characterizations for the purified products are examined in full detail by various instrumental analyses and microscopy. An enhanced miscibility with higher molar mass of PC was confirmed by both the unambiguous scanning electron microscopy (SEM) images, showing sub-inclusions of PS/PC microdomains in the matrix, and the characteristic size exclusion chromatograms (SEC) of the fractionated sample, exhibiting homogeneous molecular

mixing. With increased molecular size of PC and its content, the T_g increases proportionally and shows a shift to higher temperatures than that of pure PC, indicating the reduced segmental motion of PC chains due to the intimate chain entanglement with PS chains. Except for the case of the lowest molar mass of PC, both the thermal and tensile properties of the composite are also elevated. The conformational similarity between the two long flexible-chain polymers has clearly accentuated the tendency for miscibility. This effect is due mainly to the increase in entropy gained by mixing, which in turn leads to reinforcement of $n-\pi$ complex formation between the carbonate and benzene mixtures. © 2002 Wiley Periodicals, Inc. *J Appl Polym Sci* 87: 1610–1618, 2003

Key words: nanocomposites; polystyrene; polycarbonates; molecular reinforcement

INTRODUCTION

Since the concept of the molecular composite (a critical region where a single isotropic phase consisting of rods randomly dispersed in the coils can exist) was first revealed by Flory,¹ they have received considerable attention because of their potential for use in the development of new polymeric materials with enhanced performance and strength, along with other advantages. If the composite material could be fabricated into three-dimensional parts, a significant percent of molecular rods with excellent tensile and compressive properties would be in tension. Therefore, they would likely possess a high level of specific mechanical properties.

The polystyrene/bisphenol A polycarbonate (PS/PC) blend system, although it has quite limited miscibility^{2,3} because of the large size of the components and their weak mutual interactions, has been spotlighted because of its optical transparency, thermal

stability, and mechanical properties that might be used for an optical application such as compact discs and lenses. Hence, many ways to promote the miscibility of PS/PC have been proposed. The first is the use of tetramethyl polycarbonate (TMPC), which is well known to be miscible with PS, as a substitutive component for PC or a compatibilizer.^{4–9} The alternative is the modification of PS into the PS ionomer,^{10,11} and the alternative is the addition of a block or graft copolymer as a compatibilizer to those immiscible polymer blends.^{12–15} These efforts, however, have been unsuccessful because of the poor ratio of price–performance and the difficulties of the synthesis of copolymer.

The present work is a continuation of our previous study¹⁶ and aimed at investigating the effects of molar mass and composition on the miscibility, various physical properties, and morphological behavior of PS/PC blends, because they are considered to be highly dependent on the molecular size of PC. Accordingly, the influences of molar mass on the glass transition temperature (T_g) and mechanical properties, as well as the morphology were studied. The reason for the selection of PC and PS as the component polymers is based on both the strong possibility of $n-\pi$ complex

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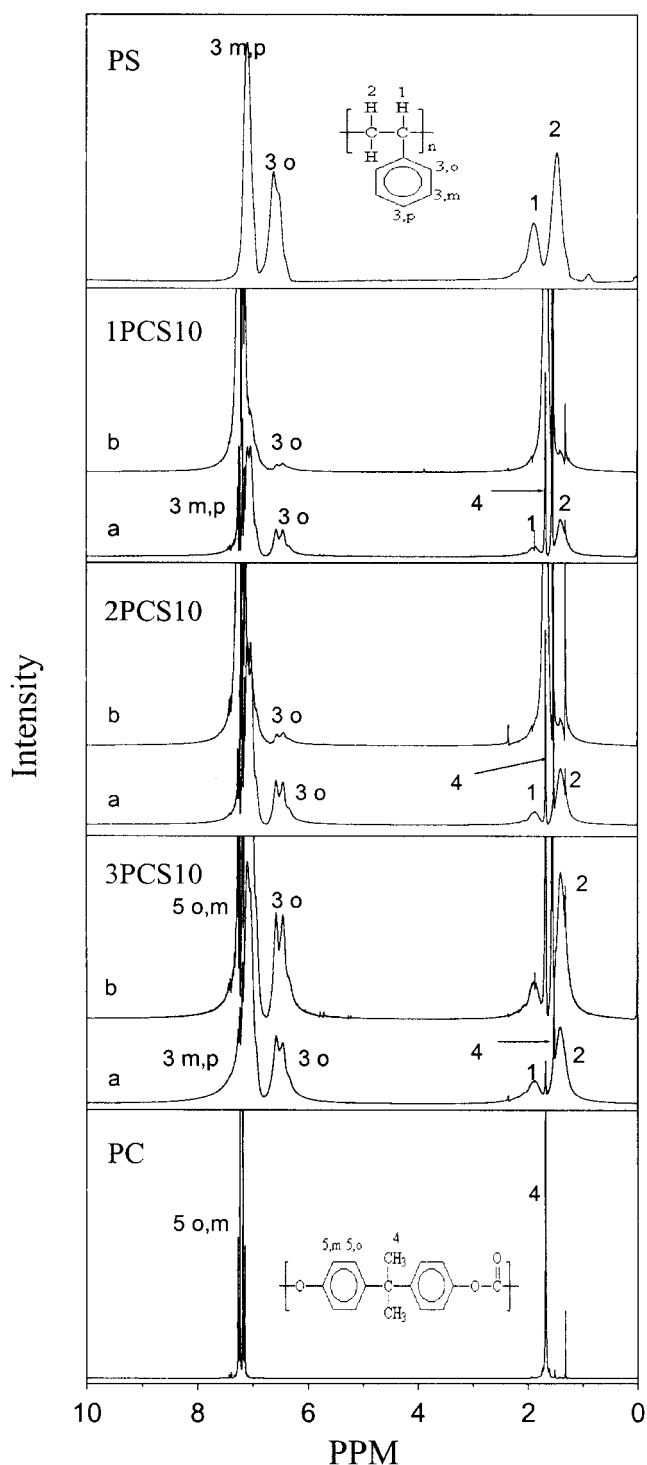


Figure 1 ^1H NMR spectra of pure PS, PC, and PS/PC molecular composites: (a) not purified (PCS series); and (b) purified by extraction (EPCS series).

formation¹⁷ and the increased entropy gained by mixing the two flexible chain polymers. In addition, PC is completely miscible in hot styrene, and hence, a polymerization of styrene monomer in the presence of PC is possible. Accordingly, styrene was polymerized with different molecular sizes of PC (M_w 19,000–

70,000 g/mol) in various amounts. In particular, efforts were made to determine the dependence of miscibility on the molar masses by employing various characterization techniques.

EXPERIMENTAL

Materials

Additive-free bisphenol A polycarbonates (PC) with different molecular masses ($M_w = 43,000$ and $70,000$ g/mol; $M_w/M_n = 1.64$ and 1.74) were obtained from Samyang Company (Korea) and purified by dissolution in chloroform and precipitation by methanol. Polymers were separated and dried under reduced pressure. In addition, a PC sample of M_w of $43,000$ g/mol was depolymerized in chloroform by addition of a small quantity of methanol containing ~ 0.6 wt % sodium hydroxide.¹⁸ After being continuously shaken for ~ 2 min, the solution was neutralized with trifluoroacetic acid. The polymer was recovered by reprecipitation into methanol, washed with methanol several times, and dried under reduced pressure. The M_w and M_w/M_n of the depolymerized product were $20,000$ g/mol and 1.56 , respectively. Chemical grade styrene monomer was obtained from Junsei Chemical Company (Japan) and purified by distillation under reduced pressure. Purification of other solvents was carried out by fractional distillation before use.

In situ polymerization

Mixtures of styrene monomer and PCs were made on a weight percent basis. The initial PC content was controlled not to exceed >30 wt % because of the

TABLE I
The Feed Ratio of Styrene Monomer/Polycarbonate (PC) and the Contents of Polymers in the Composite

Sample Code	Feed Ratio	Polymer Content ^b
	SM ^a /PC (g/g)	PS/PC (g/g)
1PCS-5	95/5	93/7
1PCS-10	90/10	83/17
1PCS-15	85/15	78/22
1PCS-20	80/20	79/29
1PCS-25	75/25	64/36
2PCS-5	95/5	93/7
2PCS-10	90/10	84/16
2PCS-15	85/15	78/22
2PCS-20	80/20	71/29
2PCS-25	75/25	64/36
3PCS-5	95/5	94/6
3PCS-10	90/10	83/17
3PCS-15	85/15	79/21
3PCS-20	80/20	70/30
3PCS-25	75/25	64/36

^a Styrene monomer.

^b Estimated from the ^1H NMR spectrum.

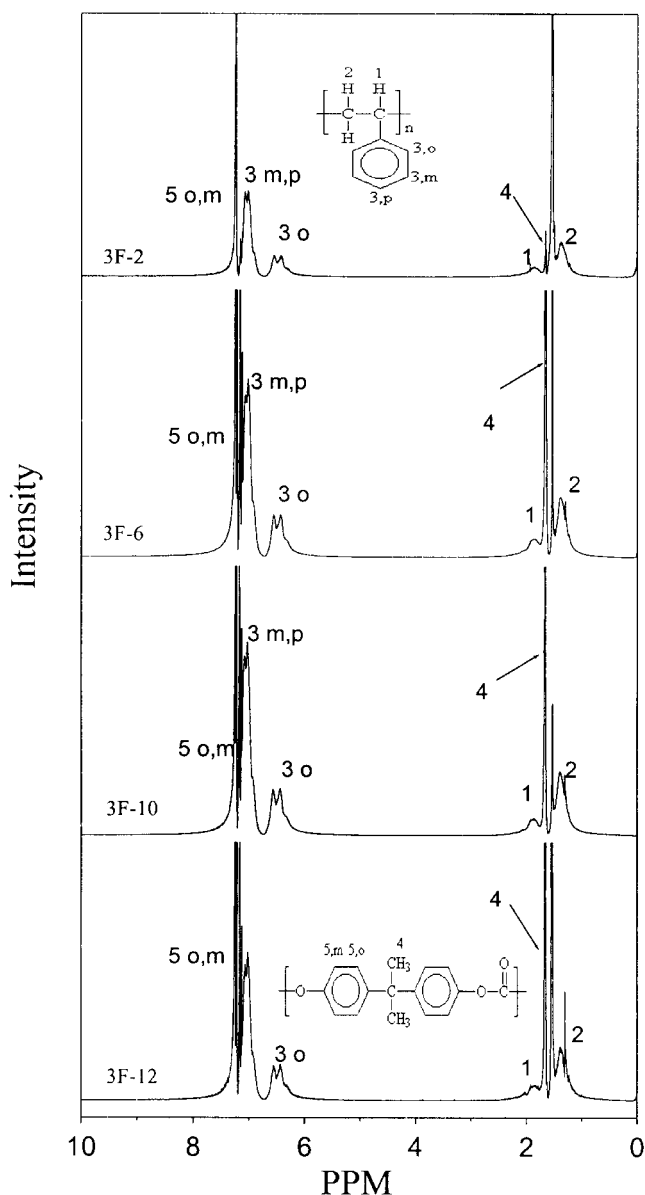


Figure 2 ^1H NMR spectra of fractionated PS/PC molecular composite (3PCS).

experimental infeasibility caused by the high viscosity of the reaction mixture at higher PC concentration. In particular, PC of high molar mass revealed low solubility in the styrene monomer. The polymerization was carried out by bulk method with neither addition of initiator nor other additives. The heterogeneous initial mixture containing PC was heated gently to 80–90°C where the PC was dissolved completely. With continuous vigorous stirring, the homogeneous reaction mixture was polymerized for 4 h at 90°C until stirring of the reaction mixture was infeasible because of its high viscosity. The polymerization was then terminated by cooling to room temperature. The chloroform solution of the product was poured into a large amount of methanol. The polymers that precipitated

were filtered and purified by several cycles of dissolution and reprecipitation from the chloroform/methanol system, which yielded white fibrous products (PCS series). The code was made in accordance with PC molar mass and its content (e.g., 1PCS-10 means the M_w of PC is 20,000 g/mol and PC content is 10 wt %). Separately, a part of the final product was exposed to hot toluene extraction for 2 days (EPCS series).

Fractionation

The 1PCS-10, 2PCS-10, and 3PCS-10 samples, with M_w of ~ 2.52 to 2.06×10^5 g/mol chosen from our stock, were separated into 12 or 13 fractions by fractional precipitation with chloroform as the solvent and methanol as the precipitant.

Measurements and characterizations

The proton nuclear magnetic resonance (^1H NMR) spectrum was recorded on a Bruker AC 250 spectrometer operating at 250 MHz. Chloroform- d containing 0.03% tetramethylsilane was used. The content of component polymer in a composite is estimated by the equations used in a previous work,¹⁶ which were derived from the values of the arithmetic mean of the integrated intensities of characteristic proton absorptions per mole of component polymers. The morphology of the fracture surfaces of composites was observed by scanning electron microscopy (SEM, JEOL JSM-5200). The accelerating voltage was 20 kV. Differential scanning calorimetry (DSC) data were obtained with a Perkin-Elmer DSC 7. Values were taken after one complete cycle of heating to 250°C and cooling at a rate of 20°C/min under a nitrogen atmosphere. Thermogravimetric analysis (TGA) of the products were carried out by employing a Dupont 9900 thermogravimetric analyzer. A sample of 20 mg mass was heated to 600°C at a rate of 20°C/min under nitrogen atmosphere. The stress-strain behavior of the composite was observed by tensile testing. An instron (Hounsfield Company H 25KS) operated with a 5 mm/min of crosshead speed was used. The specimen was prepared by the dumbbell molder (Model CS-183, Custom Scientific Instruments, Inc.) set at $250 \pm 5^\circ\text{C}$. Tests were carried out at 25°C and 60% relative humidity. The results of 10 specimens were averaged. Average molar masses and polydispersity indices of the products were measured by size exclusion chromatography (SEC) with a chromatograph equipped with a Waters 510 pump, infrared (IR) and ultraviolet (UV) detectors, a Rheodyne injector, and four linear columns packed with μ -Styragel. The flow rate of tetrahydrofuran (THF) was 1.0 mL/min, and the columns were calibrated with PS standards.

TABLE II
The Molar Masses and Polydispersity Indices of Those Representative Fractionated PC-PS Molecular Composites by Size Exclusion Chromatography and Contents of Polymers in the Composite

Sample Code		$\bar{M}_n \times 10^{-5}$	$\bar{M}_w \times 10^{-5}$	\bar{M}_w/\bar{M}_n	Polymer content ^a PS/PC (g/g)
1PCS-10	1F-2	3.98	5.33	1.34	— ^b
	1F-4	2.97	3.64	1.22	— ^b
	1F-6	2.20	2.52	1.15	99/1
	1F-8	1.58	1.79	1.13	90/10
	1F-10	0.87	1.03	1.18	65/35
	1F-12	0.22	0.28	1.28	56/44
2PCS-10	2F-2	3.56	4.88	1.37	99/1
	2F-4	2.12	2.79	1.30	94/6
	2F-6	1.89	2.21	1.17	92/8
	2F-8	1.52	1.76	1.16	88/12
	2F-10	1.10	1.26	1.15	77/33
	2F-12	0.65	0.83	1.26	60/40
3PCS-10	3F-2	4.16	5.22	1.25	91/9
	3F-4	2.16	2.53	1.17	95/5
	3F-6	1.80	2.06	1.15	97/3
	3F-8	1.04	1.21	1.15	89/11
	3F-10	0.50	6.13	1.21	72/28
	3F-12	0.21	0.27	1.29	68/32

^a Estimated from the ¹H NMR spectrum.

^b Not observed.

RESULTS AND DISCUSSION

Structural characterization

The ¹H NMR spectra obtained from the purified molecular composites are shown in Figure 1 along with those of PC and PS homopolymers, irrespective of the molar masses of PC. All characteristic proton absorptions corresponding to benzyl proton (1), methylene proton (2), and aromatic proton (3) of PS, and methyl proton (4) and aromatic proton (5) of PC can be observed in the spectra of PCS10s. As described in the previous work,¹⁶ the peak of benzyl proton (1) of styrene in the composites (PCS-10s) shows a slight shift toward lower frequency, which is attributed to the *n*- π complex formation between unshared pair of electrons of the carbonate groups in the PC and the π electrons of the aromatic rings of the PS.^{17, 19} Furthermore, the spectra of EPCS-10s demonstrate absorption due to the aromatic proton of PS, suggesting indirectly the occurrence of partial molecular mixing.

The feed ratios of styrene monomer against PC and the contents of component polymers in products, estimated from the equation in the previous work,¹⁶ are summarized in Table I. The variations of PS content in the products show a decreasing trend throughout when compared with the quantity of styrene monomer added initially. This trend is ascribed to the higher viscosity of the reaction mixture at the early stage of polymerization, which leads to a relatively low conversion; hence, a considerable amount of monomers might be either unreacted or oligomerized.

On the other hand, it is of interest to note that the spectra of the fractions (3PCS series) in Figure 2 ex-

hibit the proton of PC (3) throughout all the range of molar masses. One possible implication of this result is that as the polymerization advances, the PC chains begins to entangle with growing PS chains; the latter is likely to be due to the gain in entropy that comes from the similar conformational structure and the specific interaction originated from the *n*- π complex formation. This suggestion would imply the importance of the magnitude of PC molar mass for the enhancement of miscibility of PS/PC composite. The contents of component polymers in the respective sample matched with their molar masses are summarized in Table II. In the case of the 1PCS series, which has the lowest molar mass of PC, the proton of PC cannot be observed or is very small (>1 wt %) until the 3rd fraction. On the whole, the results in Table II show that the PC contents of fractions are inversely proportional to the molar mass of the fraction. These results are substantial evidence demonstrating the degree of miscibility of the given system.

Size exclusion chromatography

The PCS series that were completely dissolved in THF (1PCS series at 40°C, and 2PCS and 3PCS series at room temperature) not only give more evidence of the occurrence of molecular mixing but also allow measurements of molar masses and polydispersity indices of the products by SEC. The SEC chromatograms shown in Figure 3 are of PC with three different molar masses and their composites (PC content is 15 wt %) and were determined with IR and UV (254 nm wavelength) detector. Clearly, the different overall shapes

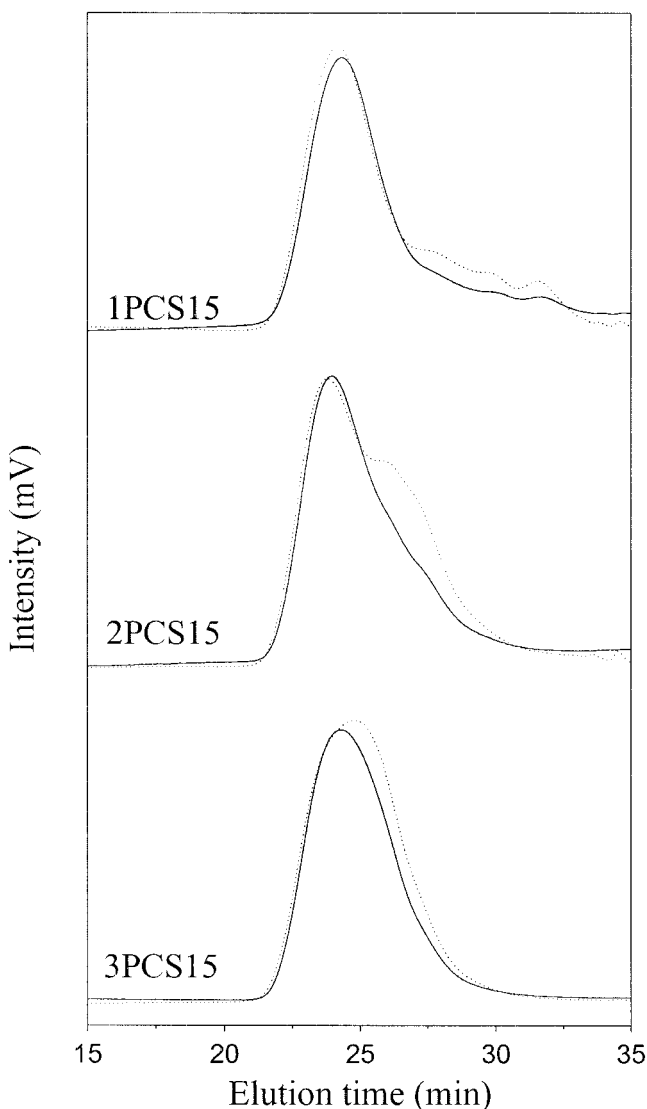


Figure 3 SEC chromatogram of PS/PC molecular composite: (a) IR detector; and (b) UV detector.

and the shifted peak position can be seen from Figure 3. The shoulder in the UV chromatograms, might be due to the aromatic chromophores ($\lambda_{\max} = 255$ nm) of the bisphenol A group in PC. Furthermore, the peaks of the composite corresponding to the PC are shifted toward a lower elution time than that of PC homopolymer; that is, PC in the composites has higher molar masses. It is also noticeable that the IR chromatogram of the representative fraction (3F-2, 3F-12) of the composite (3PCS-10), which was certified as a mixture of PC and PS by ^1H NMR, has quite a different shape than the UV chromatogram shown in Figure 4. The chromatogram of the fraction 3F-12, with the low molar mass, exhibits two peaks. The peak at the lower elution time corresponds to PS and the other to PC. On the other hand, the fraction of high molar mass (3F-2), although it has both PC and PS, shows the typical one peak. This trend of showing one peak

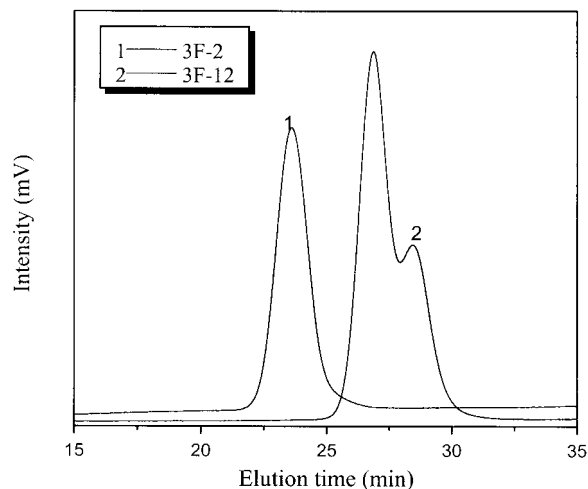


Figure 4 The IR chromatogram of fractionated PS/PC molecular composite (3PCS-10).

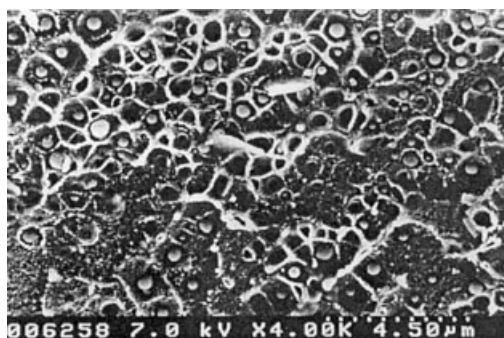
lasted until the 10th fraction. These results support the idea that two-component polymers act as a homopolymer, leading to a conclusion that the molecules of PC and PS are mixed in a molecular level.

The data obtained from the SEC measurements for the PCS series and PC are summarized in Table III. Of all the compositions studied, the PCS series exhibited higher molar masses than that of PC homopolymer, indicating the formation of high molar mass PS. In addition, the results in Table III also shows that the M_n values, which are known to be highly sensitive to the presence of low molar mass polymer, are inversely proportional to the input quantity of PC, whereas the M_w values are less diminished. Nevertheless, the vari-

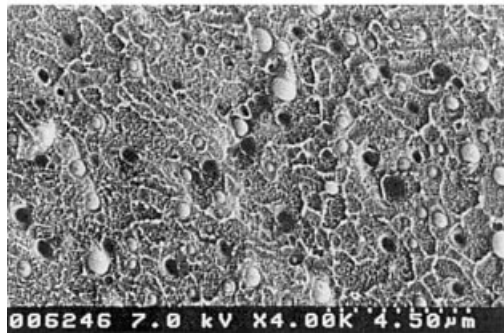
TABLE III
The Molar Masses and Polydispersity Indices of PC and PC-PS Molecular Composites by Size Exclusion Chromatography

Sample Code	$\bar{M}_n \times 10^{-5}$	$\bar{M}_w \times 10^{-5}$	\bar{M}_w/\bar{M}_n
1PC	0.13	0.20	1.56
2PC	0.26	0.43	1.64
3PC	0.40	0.70	1.74
1PCS-5	1.03	2.18	2.11
1PCS-10	0.75	2.11	2.82
1PCS-15	0.47	1.94	4.16
1PCS-20	0.38	1.72	4.99
1PCS-25	0.21	1.72	8.18
2PCS-5	1.05	2.37	2.27
2PCS-10	0.85	2.21	2.60
2PCS-15	0.75	2.16	2.90
2PCS-20	0.64	1.98	3.12
2PCS-25	0.48	1.71	3.54
3PCS-5	1.02	2.26	2.22
3PCS-10	0.91	2.18	2.42
3PCS-15	0.87	2.10	2.41
3PCS-20	0.76	2.13	2.81
3PCS-25	0.77	2.17	2.82

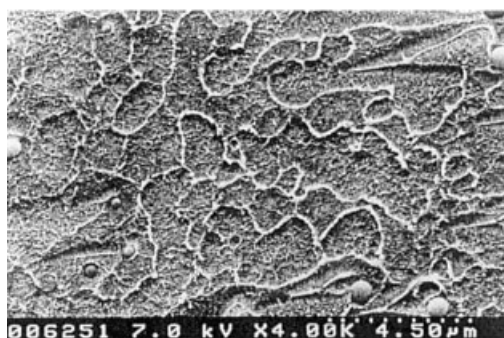
ations of the polydispersity index are proportional with the PC contents. The higher the PC molar mass, the smaller the degree of deviation. This trend is reasonably attributed to the low conversion of styrene monomer to high polymer because of the initial high viscosity of reaction mixture with increasing content and molar mass of PC.



(a)



(b)

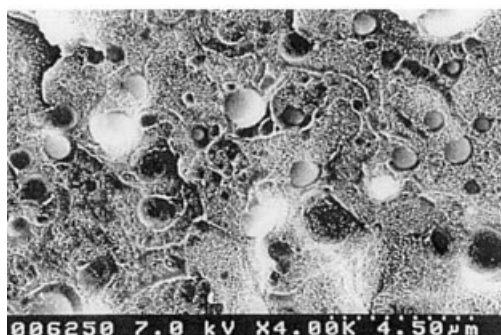


(c)

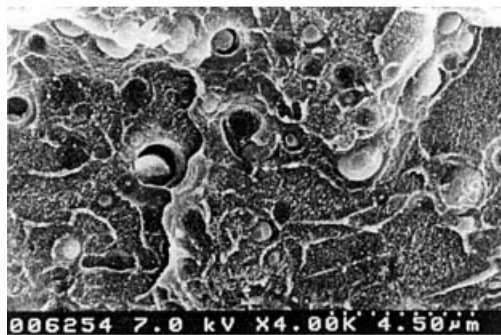
Figure 5 SEM micrographs of fracture surfaces of molecular composites containing 10 wt % PC: (a) 1PCS-10; (b) 2PCS-10; and (c) 3PCS-10.



(a)



(b)



(c)

Figure 6 SEM micrographs of fracture surfaces of molecular composites containing 25 wt % PC: (a) 1PCS-25; (b) 2PCS-25; and (c) 3PCS-25.

Morphological characteristics

As is known well, the use of SEM micrographs of fracture surfaces of the blends to observe their morphology, particle sizes, and degree of dispersion is often successful to elucidate the compatibility between the components. The previous work¹⁶ revealed that the phase separation did not appear during the variation of temperature, and the composite and the sample etched by NaOH solution to remove PC selectively

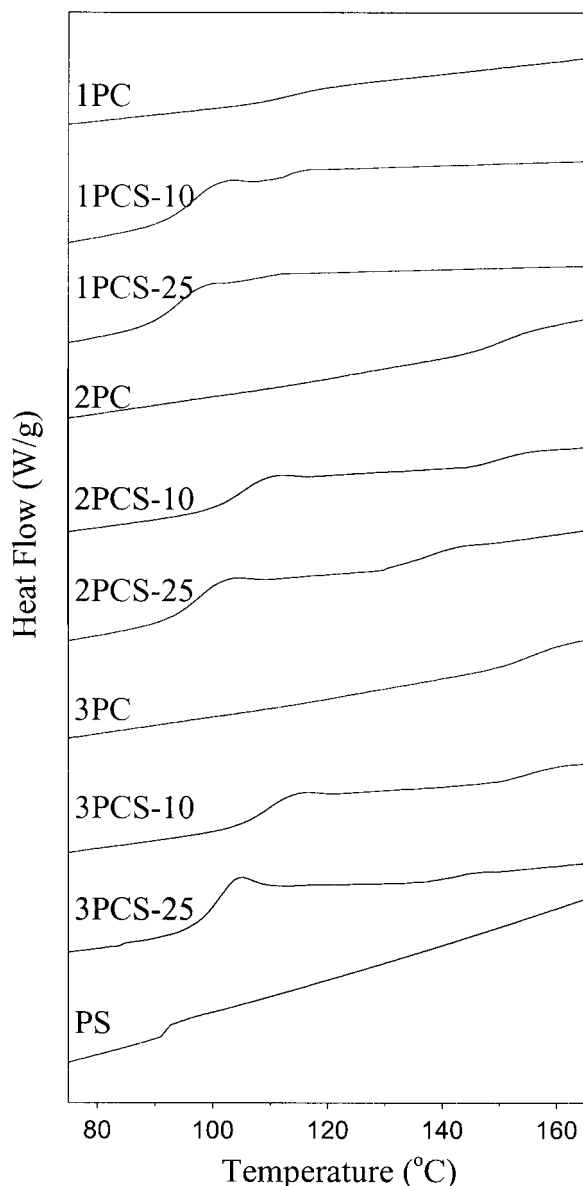


Figure 7 DSC thermograms of pure PS, three different molar masses of PC, and PS/PC molecular composites, as a function of temperature.

showed an interesting image. The dispersed phase domain contains subinclusions of PC and PS microdomains sized in tens of nanometers, which implies that adhesion between the continuous phase and the dispersed phase has been in action. On that ground, SEM micrographs taken from those composites prepared by using PCs of different molar masses are presented in Figure 5. In the case where the highest molar mass of PC (c) is concerned, the subinclusions of PS/PC microdomain in the continuous phase clearly can be seen, although a few of PC spheres sized $\sim 1 \mu\text{m}$ exist. In contrast, the boundaries of the PS/PC microdomain become ambiguous as the molar mass of PC is decreased. Concurrently, more PC spheres emerge from

TABLE IV
Glass Transition Temperatures of Composites from DSC Measurement

Sample Code	T_g		$T_g(p)$
	PS	PC	Linear Eq. ^a
PS	92	—	—
1PCS-5	94	—	94
1PCS-10	95	108	96
1PCS-15	96	109	97
1PCS-20	95	111	98
1PCS-25	98	113	100
1PC	—	114	—
2PCS-5	96	—	96
2PCS-10	98	139	101
2PCS-15	101	142	105
2PCS-20	104	145	109
2PCS-25	106	148	113
2PC	—	150	—
3PCS-5	96	—	96
3PCS-10	101	142	103
3PCS-15	104	145	105
3PCS-20	106	150	111
3PCS-25	110	154	115
3PC	—	156	—

^a Data estimated by a linear equation established from a completely miscible amorphous polymer system.

the continuous phase and their sizes are also enlarged, as illustrated in Figure 5b. Finally, the lowest molar mass fraction of PC has not only led to coalescence of the PC spheres but also eased the boundaries, whereas the PC domains became more clear. This result, taking into account the conformational similarities of both the polymer chains, can be anticipated because the longer chains of polymers can possibly be entangled more densely with each other due to the higher order of entropy gain. This situation, in turn, can provide chances of $n-\pi$ interactions; that is, formation between

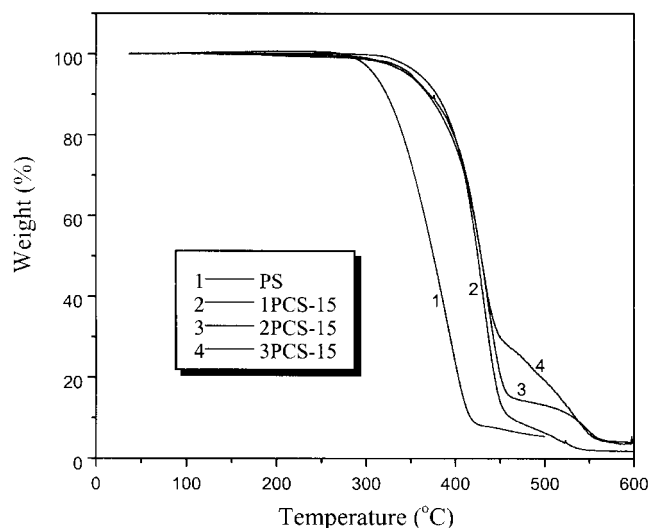


Figure 8 TGA thermograms of some selected fractions of PS/PC molecular composites.

the free electrons of the ester group of PC and the π electrons in the aromatic groups of benzene. Those samples of higher content of PC reveal this phenomenon more clearly, as is illustrated in Figure 6. However, it is interesting that the matrix exhibits the homogeneous phase, whereas the macrodomains constituted with only PC polymer show a complicated shape. This complexity, due mainly to the formation of a divided boundary of domain and matrix, is expected to originate from the increment of immiscibility and is regarded as the process of phase separation.

Glass transition temperature

The DSC traces of various composites as a function of temperature are shown in Figure 7. Three different molar masses of PC are also illustrated. The T_g of PC does not appear until the PC concentration reaches ~ 0.17 weight fraction (10 wt % of PC content) and then gradually decreases with increased PC content, whereas the elevated T_g s of PS are observable over the entire composition range and are proportional to the PC content. As mentioned in the previous work,¹⁶ although the composites containing PC contents > 0.17 weight fraction exhibit two T_g s, the overall trends of T_g variations of the composites appear different from that of the simple blend with the increment of the molar mass of PC. It can also be seen, from the results in Table IV, that the T_g s of the two components are shifted somewhat closer to each other, which is an indication of the enhancement for both the degree of dispersion of the PC component and the partial miscibility of the given system.^{20, 21} In the case of the lowest PC molar mass (1PCS series), the width of T_g variation of PC is $\sim 6^\circ\text{C}$, whereas when the molar mass of PC is higher, the width is elevated to $10\text{--}14^\circ\text{C}$. This result can likely be attributable to the potential that the specific interaction of the composite increases with the molar mass of PC.

Furthermore, the values of the predicted single $T_g(p)$ between the component polymers were estimated by following the simple linear equation derived by Stoelting et al.²² under an assumption that the polymers are completely miscible. Clearly, the $T_g(p)$ values come closer to actual values of the T_g of PS with the increment of molecular mass of PC.

Material characterization

The dependence of the PC molar masses on the thermal stability of those composites was evaluated by TGA. The measurements of the weight loss as a function of temperature for the selected samples are shown in Figure 8. In all the composites, the first sign of 1% initial weight loss was observed at $\sim 320\text{--}325^\circ\text{C}$. In contrast, the first sign of 1% weight loss for pure PS was noted at $\sim 270\text{--}275^\circ\text{C}$. However, the onset tem-

TABLE V
Stress–Strain Data of PC-PS Molecular Composites

Sample Code	Tensile Strength (MPa)	Elongation (%)	Modulus (GPa)
PS	32.2	3.02	1.48
1PC	—	—	—
2PC	63.2	120.6	1.25
3PC	70.6	122.3	1.21
1PCS-5	38.2	3.03	1.43
1PCS-10	34.7	2.27	1.44
1PCS-15	34.4	2.24	1.48
1PCS-20	33.3	2.22	1.55
1PCS-25	29.6	1.92	1.64
2PCS-5	32.8	3.05	1.37
2PCS-10	33.0	3.07	1.41
2PCS-15	33.8	3.13	1.44
2PCS-20	34.3	3.20	1.48
2PCS-25	35.2	3.37	1.51
3PCS-5	36.3	3.03	1.33
3PCS-10	37.8	3.13	1.35
3PCS-15	40.3	3.25	1.39
3PCS-20	42.2	3.31	1.42
3PCS-25	45.1	3.40	1.46

peratures of decomposition for the composites are shifted toward a higher range with increasing molar mass of PC. This elevation of thermal decomposition temperature of composites can be attributed to the enhanced miscibility of PS with PC, which is more thermally stable. Accordingly, this thermal stability of those composites gives more evidence for the elevated miscibility of the given system.

The tensile stress–strain behaviors of composites are summarized in Table V. Evidently, composites exhibit higher values of tensile strength at break than that of pure PS. Also, a trend is obvious that the tensile strength increases gradually with the increments of PC concentration. Moreover, the fracture elongations of the composite containing higher molar masses of PC are higher than that of the pure PS. This effect is undoubtedly responsible to the ductility of PC, which usually yields before fracturing. The tensile modulus of the composite, on the other hand, is slightly lower than that of pure PS. These relatively low values of modulus are primarily a consequence of the effect exerted by the ductile PC. On the whole, the entanglement of mechanical properties revealed by those composite composed of higher PC molar mass is ascribed to the contribution of the dispersion of macrodomain containing PS/PC microdomain into the PS matrix.

CONCLUSIONS

The homogeneous solution of styrene monomer and bisphenol A PC polymerized *in situ* yielded a partially miscible molecular composite. The two T_g values observed over the composition range studied changed significantly with the molar mass of PC and its com-

position, giving substantial evidence of partial miscibility. This miscibility increases with the molar mass of PC. The fracture morphology observed in the SEM micrograph revealed the subinclusion of PS/PC microdomain in a macrodomain, which is indicative of the good adhesion between the PS matrix and the PC dispersion phase. The size and number of the domain of PC are proportional to the composition, while inversely proportional to the molar mass of PC. The ^1H NMR spectra of the fraction divided by fractional precipitation show that the PC and PS are mixed homogeneously throughout the range of molar masses examined. The ^1H NMR spectrum obtained from the composite shows a chemical shift of benzyl proton of PS to the lower frequency. Also, the mechanical properties and thermal properties are enhanced compared with those of pure PS and are proportional to the molar mass of PC.

All the experimental results so far obtained lead us to conclude that the system has good miscibility. The entropy gained by mixing two large flexible component polymer chains, and in turn the formation of an $n-\pi$ complex between the unshared pair of electrons in the carbonate groups and aromatic rings of PS are considered to be responsible for the partial miscibility.

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