

# Preparation of the Anhydride Terminated Polycarbonate and Its Reactive Compatibilization with Polystyrene

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**ABSTRACT:** Polycarbonate with anhydride end groups (PC-anh) was prepared by the reaction between polycarbonate having hydroxyl end groups (PC-OH) and trimellitic anhydride chloride (TMAC). Hydroxyl or anhydride terminated polycarbonates were characterized by Fourier transform infrared spectroscopy (FTIR) and nuclear magnetic resonance (NMR) spectroscopy. The reaction of PC-anh with polystyrene containing oxazoline reactive groups (RPS) was confirmed not only by the torque measurement during melt blending of these two but also by FTIR spectroscopy of the reactive blend obtained. Polycarbonate (PC)/polystyrene (PS) compatibilized blends were prepared by melt blending along with their reactive counterparts, PC-anh and RPS in the Haake mixer. The morphologies of these blends were examined by the scanning electron microscope (SEM). The compatibilized blends with reactive components showed relatively finer morphologies than the uncompatibilized blend without reactive components. Izod impact strength and rheological property of these blends were also investigated. © 2000 John Wiley & Sons, Inc. *J Appl Polym Sci* 77: 1338–1347, 2000

**Key words:** polycarbonate; anhydride terminated polycarbonate; hydroxyl terminated polycarbonate; polystyrene; reactive blends; compatibilization; coalescence

## INTRODUCTION

There is of intense interest in multiphase polymer blends because of their commercial versatility.<sup>1</sup> Lately, with the environmentally conscious market for claiming resins free of halogenated flame retardant, a Polycarbonate/polystyrene (PC/PS) alloy has been commercialized and is competing with PC/acrylonitrile–butadiene–styrene (ABS) alloys.<sup>2</sup> It has been known that PC/PS blend is not compatible even though there is some evidence of partial mixing and adhesion of the two at the interface,<sup>3–5</sup> thus, numerous studies focused on

compatibilization of the blend.<sup>6–10</sup> Quite often simple melt blending of these two components shows inherent immiscibility, which leads to a large interfacial tension in the melt and makes it difficult to properly disperse the components during mixing and drives coalescence of phase morphology at low stress or quiescent conditions. It generally leads to poor interfacial adhesion in the solid state, which results in poor material properties. Compatibilization for these blends can be obtained by the addition of block and graft copolymers properly designed, which can act as interfacial agents. When preformed block and graft copolymers are used for this purpose, some of them would form micelles or reside within the bulk phases of the blend in addition to the presence at the interface.<sup>11</sup> Not only because rela-

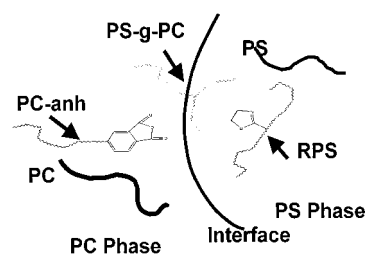
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tively large amounts of block or graft copolymers are required, but also they are quite expensive, these surface active interfacial modifier have not been used extensively. Recently more attention has been focused on in situ formation of graft or block copolymer via interfacial reaction of added functionalized polymeric components, i.e., reactive compatibilization. Block copolymers formed by reaction between two polymers with counter functional groups would preferentially locate at the interface between two polymers since they can meet at the interface. This approach has been demonstrated to be effective and cost-efficient for the morphological control of the immiscible blends. Reactive compatibilization has been most widely practiced for blends involving polyamides by making use of the reaction of available amine end groups with anhydride units in the other phase.<sup>12,13</sup> The amine-anhydride reaction to form an amide linkage has been known to be very rapid at melt processing temperatures.

It is hardly found in the literature about in situ reactive compatibilization for PC/PS blend, whereas several studies have been reported to use the preformed graft or block copolymers. Wildes and coworkers<sup>14,15</sup> reported about reactive compatibilization of PC/ABS blends through reaction between carbonate functional group of PC main chains and secondary amine groups attached to styrene-acrylonitrile (SAN) copolymer. Because most commercial polycarbonates do not have reactive end groups, they suggested a new route to form block or graft copolymer through the reaction between the carbonate groups of PC main chains. They showed that the secondary amine groups attached the SAN backbone that would be miscible with ABS materials might react with PC at the carbonate linkage to form a SAN-g-PC copolymer. The graft copolymer would reside at the PC/ABS interface and provide improved morphological stability at the elevated temperature by suppressing the phase coalescence. However, there are some disadvantages that the reactions between carbonate groups of PC main chains and amine group may also cause chain scission reaction so that a low molecular weight polycarbonate with a phenol group at the end would be generated. Thus in order to minimize this kind of chain scission reaction, PC with end-group functionality should be prepared in the first place. Therefore it will react with other functionality such as epoxide, amine, or oxazoline in the corresponding phase, to offer us a useful way to develop polycarbonate-based blends.



**Figure 1** Schematic diagram of PC/PS blend compatibilized by reactive components, PC-anh and RPS.

On the other hand, extensive research has been performed on the reactive compatibilization using the reaction between PS containing oxazoline (RPS) and other polymer with counter functional groups, amine end groups of nylon, or carboxylic acid groups of polyesters and acrylonitrile rubber having by Baker and coworkers.<sup>16–21</sup> The compatibilization through reaction between polyethylene having anhydride functional group and PS containing oxazoline group has been also studied by Liu et al.<sup>22</sup> Their investigation revealed that the reactive blends have a fine and uniform morphology to result in the enhanced mechanical properties.

In this study, an anhydride terminated PC (PC-anh) was firstly synthesized and was used for the reactive component of PC phase. On the other hand, a commercial RPS having 5 wt % oxazoline functionality was employed for PS phase. Thus it is expected that PC-anh and RPS would preferentially be located in the respective PC and PS phase and performed as reactive components like the scheme displayed in Figure 1. The formation of graft copolymer through reaction between these reactive components has been examined with Fourier transform infrared spectroscopy (FTIR) and other techniques. Change in morphology, impact strength, and rheological property of the reactive blends were also investigated.

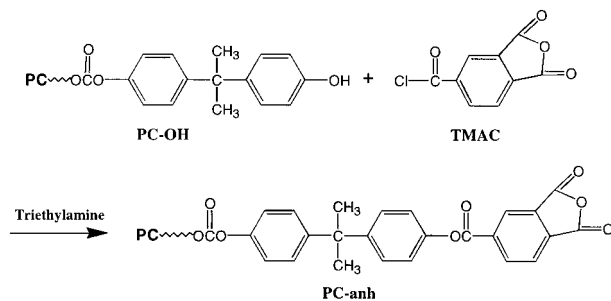
## EXPERIMENTAL

### Preparation

Hydroxyl terminated PC (PC-OH) was prepared by melt transesterification of diphenyl carbonate (DPC) with bisphenol A (BPA) monomer in a batch reactor. Because DPC exhibits moderate vapor pressure at the reaction temperature 250–300°C, some DPC monomer may be lost from the reactor during the initial reaction stage. Any loss

of DPC during the reaction will cause significant variations in the concentration of reactive end groups so that it will make difficult to obtain the high molecular weight polymers. Thus the appropriate temperature and pressure profiles should be employed in order to minimize the loss of DPC and to maintain the initial mole ratio of phenyl carbonate to hydroxyl group during the reaction in the same manner as Kim and Choi.<sup>23</sup> According to the procedure described in this literature, the reaction was carried out with five stages. In the first stage, the reaction is conducted under nitrogen atmosphere at 180°C for 2 h. In the second stage, the reaction temperature is raised to 250°C without reducing the pressure. At the beginning of third stage, the pressure in the reactor is reduced gradually to 150 mmHg while the reaction temperature is kept at 250°C for 1 h. In stage four, the reactor temperature is again raised from 250 to 280°C with the reduced pressure, 50 mmHg. In stage five, the reactor pressure is finally lowered to 1 mmHg and the reaction temperature is maintained at 280°C for 1 h. In this reaction system, the mole ratio of end groups can be controlled by the initial mole ratio of reactants, DPC and BPA. The polymerized PC-OH was precipitated in the acetone. Precipitation was repeated twice to remove any unreacted monomers. Then the precipitated PC-OH was dried in a vacuum oven at 70°C in 2 days before further usage.

PC-anh was made from PC-OH by the reaction with trimellitic anhydride chloride (TMAC) as shown in the following alkoxy-de-halogenation reaction scheme.<sup>24</sup> The reaction was carried out in a reactor containing the dissolved PC-OH and TMAC in methylene chloride. Triethyl amine (TEA) was used as an acid acceptor.



About four times excess amount of TMAC compared to that required for reaction of phenolic end groups of PC-OH was dissolved in methylene chloride. After the TMAC solution was introduced into the functionalization reactor, a small amount

of TEA was added to the TMAC solution. This functionalization reactor was kept under dried N<sub>2</sub> atmosphere in advance. The PC-OH was dissolved at 10% concentration (w/v) in methylene chloride and the solution was put into the functionalization reactor drop by drop over 1 h. By this procedure, excess amount of TMAC in the reactor was presented throughout the functionalization reaction so that the possible side reaction would be relegated. After the addition of PC-OH solution, the reaction was proceeded further 4 h and then precipitated into acetone. Then this procedure was repeated twice more. After the purification, the polymer was dried in a vacuum oven at 70°C for 2 days. This procedure is very similar to the examples described in the references.<sup>24-26</sup>

## Materials

Most of materials used in this study are commercially available polymers and their details are summarized in Table I.

## Melt Blending

PC/PS blends were prepared in composition of 70/30 by weight with varying amount of PC-anh and RPS as reactive components in the Haake Buchler Rheomix model 600P with roller blades. Same amount of PC-anh and RPS were introduced in all of reactive blends. The mixer was operated at 50 rpm and 230°C. After 10 min of melt blending, the blend samples were taken out carefully and quenched in ice water to freeze the blend morphology.

## Scanning Electron Microscope Analysis

The fractured morphologies of PC/PS and PC/PS/reactive component blends were observed using a Jeol 840A scanning electron microscope (SEM). These samples were prepared by quenching in the liquid nitrogen and subsequent fracturing. And then they were etched to remove PS components or unreacted RPS components in cyclohexane at 60°C during 1 day. The number average particle diameter in the dispersed phase was calculated from analysis of 300–500 particles taken from SEM photomicrograph using an image analysis software program.

## Izod Impact Test

One of the methods for evaluating compatibilization effect of the reactive components is to test the

**Table I** Polymers Used in This Study

Material	Designation	Molecular Weight		Maker	Grade
		$M_n$	$M_w$		
Polycarbonate	PC	10 500	18 400	Teijin Chem. Ltd.	Panlite L-1225LL
Polycarbonate having Hydroxyl end groups	PC-OH	10 800	15 300		
Polycarbonate having Anhydride end groups	PC-anh	9200	13 800		
Polystyrene	PS	82 000	218 000	Cheil Ind. Inc.	Starex HF2660
Polystyrene containing oxazoline	RPS	61 300	156 000	Nippon Schokubai Co.	Epocros RPS1005
Styrene-Butadiene-Styrene copolymer	SBS	—	—	Asahi Chem. Co.	Asaprene 420P

Molecular weight was measured by gel permeation chromatography using polystyrene standards.

impact property of the blends. In addition to PC/PS/reactive components blends, PC/PS/styrene-butadiene-styrene triblock copolymer (SBS) blends with reactive components were prepared in the Haake mixer. The specimens were notched according to the ASTM D256 by cutting the line in the compression molded sheet at 250°C with dimensions 150 × 200 × 2 mm. Izod impact tests were carried out on a Toseiki impact tester using notched specimen. Each impact value reported was the average of five tests.

### Dynamic Mechanical Spectroscopy

The dynamic moduli were measured with a Rheometrics Mechanical Spectrometer RDS-II with parallel plate geometry ( $r = 12.5$  mm). Isothermal frequency sweeps were recorded at 230°C with an accuracy of  $\pm 0.3^\circ\text{C}$  under inert  $\text{N}_2$  atmosphere. The strain was kept between 20 and 30 1/s to stay within the linear viscoelastic regime, which was tested by strain sweeps.

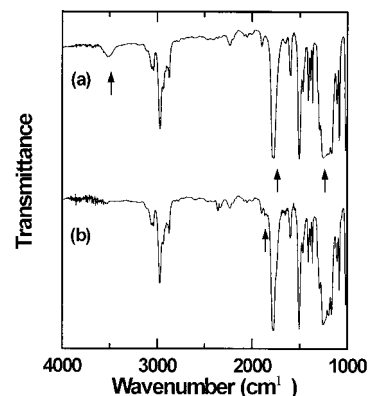
### Characterization

FTIR spectra for prepared PC-OH and PC-anh were observed on Perkin Elmer 1769X spectrometer in a film state. As seen in the Figure 2a, broad phenolic O-H stretching vibrations of PC-OH was also observed at around  $3500\text{ cm}^{-1}$  and C=O carbonyl stretching vibrations appeared at  $1772\text{ cm}^{-1}$  and O-C-O carbonate stretching vibrations at around  $1250\text{ cm}^{-1}$  are also apparent. Thus, it can be confirmed that PC-OH polymer

with hydroxyl end groups was successfully synthesized. Pryde and Hellman<sup>27</sup> reported a model consistent with these results.

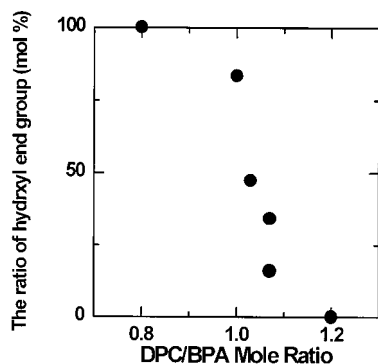
The characteristic doublet absorption band due to anhydride ring of PC-anh is observed at  $1863\text{ cm}^{-1}$  whereas phenolic OH stretching band at about  $3500\text{ cm}^{-1}$  almost disappears, as shown in Figure 2b. Another higher frequency band at  $1778\text{ cm}^{-1}$  associated with the symmetric vibration of anhydride ring<sup>25</sup> was not shown due to overlapping with broad carbonyl C=O band of PC at around  $1772\text{ cm}^{-1}$ . This must be one of the obvious evidences to show that functionalization of PC with anhydride end groups was done from PC-OH.

The  $^{13}\text{C}$  NMR spectra of PC-OH and PC-anh were obtained using a Bruker 500 MHz NMR



**Figure 2** FTIR spectra of PC-OH and PC-anh.



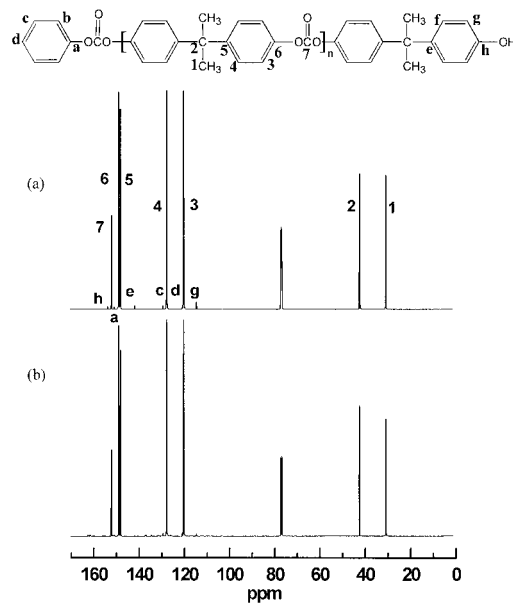


**Figure 3** The ratio of hydroxyl end groups of PC-OH with varying initial mole ratio of reactants, diphenyl carbonate (DPC), and bisphenol A (BPA).

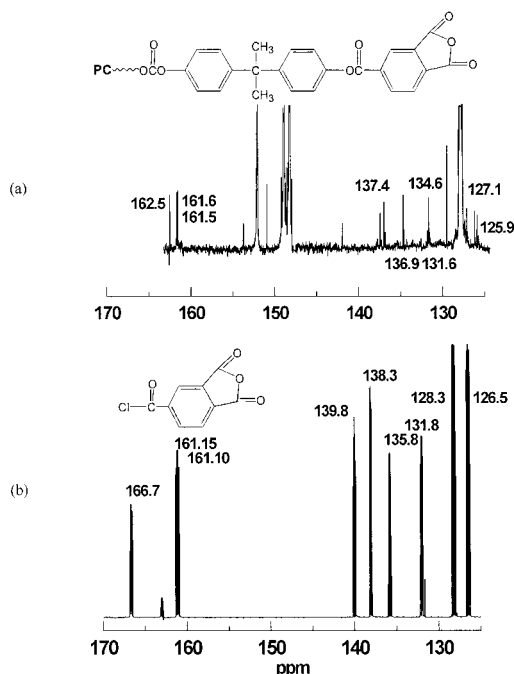
spectrometer. Test samples were examined by dissolving about 20% (w/v) concentration in a deuterated chloroform solvent. Traditional titration methods for measuring amounts of hydroxyl end groups were not employed because some carbonyl compounds might make an interference.<sup>28</sup> With proper attention paid to spin-lattice relaxation times ( $T_1$ ) and nuclear overhauser effect (NOE),  $^{13}\text{C}$  NMR can be useful tool to provide quantitative measurement of the ratio of hydroxyl end group to phenyl end group of PC-OH. The measurements were performed at the delay time 15 s with inverse gated decoupling mode. Schilling et al.<sup>29</sup> reported that these conditions were sufficient for polycarbonate materials to obtain quantitative data for eliminating NOE without paramagnetic quenching reagents. A preliminary study was performed in order to obtain the desirable contents of end groups at the chain end of PC-OH. In this experiment, the initial mole ratio of DPC/BPA was changed from 0.8 to 1.2, as displayed in Figure 3. When the initial DPC/BPA mole ratio was set at 0.8 and 1.2, all of the chain end groups were terminated with hydroxyl group or phenyl group respectively and synthesized PC have the small molecular weight with number average molecular weight at most 3000 g/mole. The ratio of hydroxyl end groups was changed significantly at the range of initial mole ratio 1.0 ~ 1.07, as seen Figure 3. In this study for obtaining anhydride terminated PC, PC-OH was prepared at the initial ratio 1.03. The respective peaks of PC-OH are assigned as shown in Figure 4a. The ratio of hydroxyl end groups of PC-OH could be calculated by way of comparing summation of e, g, h peak area with that of a, c, d peaks. The peaks b and f were excluded for this calcula-

tion because these appeared at the side of main chain peaks so that could give erroneous peak area. Reliability could be given by the comparison peak area of methyl resonance at the position 1 to that of quaternary carbon of PC-OH at the position 2 in Figure 4a. The ratio of these two carbons resonance was 2:1 as expected. Another investigation can be done to examine the peak ratio of end groups. Theoretically, the ratio of g peak at 114.7 and h peak at 153.7 ppm is expected 2:1, the value obtained through our experiment appears 1.84:1 respectively. This difference is considered to be in the reliable range. The mole ratio of hydroxyl end groups of PC-OH used in this study was 71.2%.

Anhydride functionalization of PC-OH can be also confirmed through  $^{13}\text{C}$  NMR analysis. The  $^{13}\text{C}$  -NMR spectra of PC-anh is shown in Figure 4b. Compared with that of PC-OH in Figure 4a, the peaks at the positions of e, g, and h resulting from hydroxyl end groups are considerably getting smaller whereas those at the positions of a, c, and d from phenyl end group maintain nearly constant in Figure 4b. Some peaks appear nearly between 125 ppm and 162 ppm. These peaks can be confirmed by comparison the  $^{13}\text{C}$  NMR enlarged spectra for PC-anh in the range of 125 and 140 ppm with that for only TMAC material, as seen in the Figure 5. In Figure 5b, the two peaks appeared at 161.1 and 161.2 are assigned for two anhydride ring carbons and one acyl carbon of



**Figure 4**  $^{13}\text{C}$  NMR spectra of (a) PC-OH (b) PC-anh in  $\text{CDCl}_3$ .



**Figure 5**  $^{13}\text{C}$  NMR spectra of (a) enlarged PC-anh in the range of 125 and 140 ppm (b) TMAC in  $\text{CDCl}_3$ .

TMAC attached to chlorine substituent appears at 166.7 ppm. The six peaks in the range of 126 and 140 ppm are attributable to phenyl ring carbons of TMAC. In Figure 5a,  $^{13}\text{C}$  NMR spectrum of PC-anh also show three peaks at around 162 ppm. Two peaks at 161.5 and 161.6 ppm are assigned as anhydride ring carbon and the other peak at 162.5 ppm is attributable to ester carbon generated by alkoxy de-halogenation reaction between hydroxyl end groups of PC-OH and acyl halide of TMAC. In addition, the newly occurred six peaks as shown in Figure 5a appear at the position of 125.9, 127.1, 131.6, 134.6, 136.9, and 137.4 ppm. These peaks are assigned as phenyl ring carbons of TMAC attached to the hydroxyl end groups of PC-OH through the reaction. The yield of the terminal anhydridization is estimated by comparing the ratio of peak areas resulting from respective end groups. The average peak

area from phenyl end groups and corresponding peak area due to hydroxyl end groups of PC-anh are calculated 0.083 and 0.053, respectively. The average peak area due to nine carbons of anhydride end groups shows 0.084, which was evaluated by averaging area of nine peaks generated anhydride end groups to minimize the calculation errors. Therefore the fraction of these phenyl, hydroxyl, and anhydride end groups functionality of PC-anh was composed at the ratio 37.7, 24.1, and 38.2%, respectively. At first, the ratio of hydroxyl end groups in the PC-OH was 71.2%, however the total ratio of anhydride end groups generated and hydroxyl end groups in the PC-anh is 62.3%. This difference is mainly thought to be attributable to generation of some of side reaction.<sup>25</sup> Results of this quantitative analysis are listed in Table II.

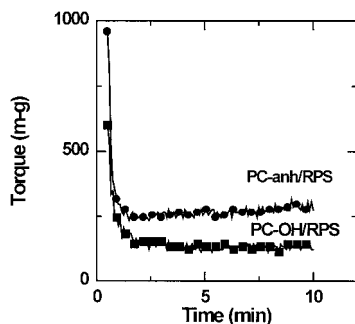
## RESULTS AND DISCUSSION

### Examination of Melt Reaction

In general, reactive blending of polymers in the melt results in the increase of molecular weight and melt viscosity, which in turn leads to apparent increase of torque during mixing. In this study, PC-anh/RPS blend as a model reaction was employed for examining melt reaction. High concentrations of functionality are required for further characterization. However, a higher loading of PC-anh is liable to generate the gelation to form the network polymer<sup>22</sup> and may lead to erroneous results. Thus the blend for a model reaction was prepared in composition ratio of PC-anh/RPS = 30/70 by weight. Figure 6 shows torque data for PC-anh/RPS and PC-OH/RPS blends during 10 min of mixing. During the early stage of melting, an increase in mixing torque showed in the PC-anh/RPS blend, whereas but no such increase was observed for the corresponding PC-OH/RPS blend. Even though phenolic hydroxyl is known to react with oxazline functionality,<sup>30</sup> it may be difficult for polymer containing oxazline

**Table II** Evolution of  $^{13}\text{C}$  NMR Peaks of PC-OH and PC-anh End Groups

Chemical Shifts (ppm)	114.7	125.9	126.2	127.1	129.5	131.6	134.6	136.9	137.4	141.9	150.9	153.7	161.5	161.6	162.5	
Positions		g		d		c				e	a	h				
Relative Intensities	PC-OH	0.408	—	0.085	—	0.152	—	—	—	0.177	0.089	0.222	—	—	—	
	PC-anh	0.120	0.074	0.092	0.079	0.142	0.076	0.092	0.069	0.071	0.045	0.086	0.053	0.099	0.121	0.071

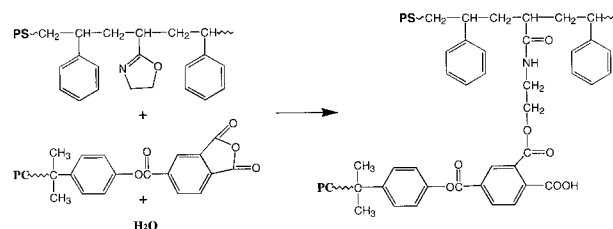


**Figure 6** Torque-time relationships for PC-anh/RPS blend and PC-OH/RPS blend.

to react with high molecular weight polymer having hydroxyl group in the melt. But anhydride functionality is known to be highly reactive with amine and oxazoline groups. Similar results were reported by Baker and coworkers<sup>22</sup> observing torque increase in melt blend of polyethylene with maleic anhydride and RPS in Brabender mixer. They demonstrated that the reaction was fast and might be over within 3 min as indicated on the torque-time curves during blending. This above-mentioned result is good evidence that a reaction between the two reactive components does occur in melt state.

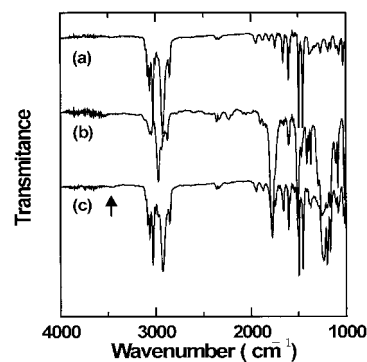
The compatibilized reaction was investigated using FTIR spectroscopy with the aim of confirming the generation of copolymers. Baker et al.<sup>16–22</sup> reported that the reaction of PS having oxazoline group with PE grafted maleic anhydride had been verified with conventional FTIR technique. They showed that the absorbance peaks at about  $3440\text{ cm}^{-1}$  appeared in the spectrum of reactive blends but not in that of nonreactive ones, which is a characteristic peak of the N-H stretching vibration of the secondary amide. And it was also shown that the new peaks at  $1743$  and  $1713\text{ cm}^{-1}$  were ascribed to the ester part of the inter-polymer linkage resulting of the opening of anhydride rings of the grafted PE. Figure 7 shows the FTIR spectra of PC-anh/RPS blend, RPS, and PC-anh respectively. The small broad absorbance peak at about  $3450\text{ cm}^{-1}$  appearing characteristic of the N-H stretching vibration of the secondary amide is observed at the PC-anh/RPS blend as shown in Figure 7c. This confirms that amide groups are formed through reaction between the functional groups of oxazoline and anhydride. A reaction scheme suggested by Baker et al.<sup>22</sup> is that anhydride groups in the presence of water can react with oxazoline functionality so that amide, ester

linkage, and carboxylic acid groups generate like the proposed reaction scheme. On the other hand Po' et al.<sup>31</sup> reported the reaction of anhydride and bis(2-oxazoline) monomer in the presence of diols led to linear polymers having ester-amide groups. Both routes proposed above can be expected in our experiments because there are appreciable amounts of hydroxyl end groups in the PC-anh as well as anhydride end groups, so that graft copolymers with amide and ester groups can be generated. The peak at  $1742\text{ cm}^{-1}$  that appeared is liable to overlap the peak with ones due to RPS in this region, as shown in Figure 7c. Therefore it is difficult to characterize this particular vibration due to the ester part of the inter-polymer linkage resulting of the opening of anhydride rings.

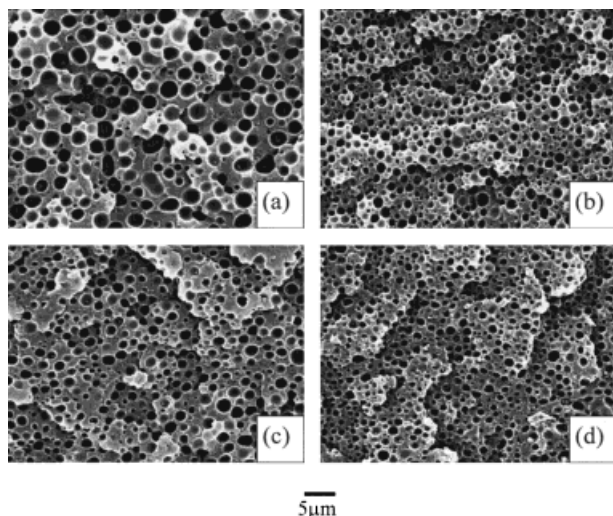


### Effect of Compatibilization

Figure 8 shows the morphology change with varying amount of reactive components. The addition of RPS and PC-anh causes the considerable change in the domain size of the PC/PS blends. (Domain size means number average diameter in the dispersed phase that is calculated from image analysis software program.) Image analysis indicates that the domain size in the reactive blends is smaller than that of the PC/PS nonreactive blend as seen in Figure 9. In addition, the narrow domain size distributions are observed in the re-



**Figure 7** FTIR spectra of RPS, PC-anh, and PC-anh/RPS blend.



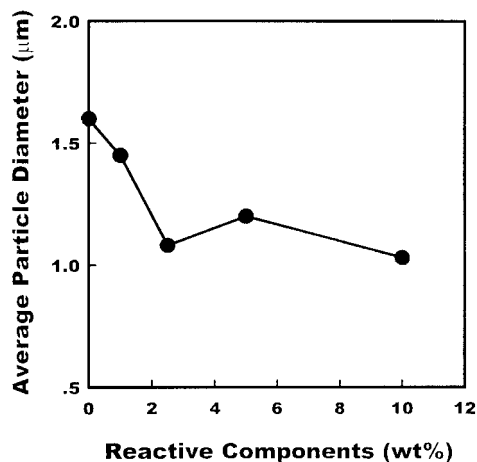
**Figure 8** SEM photomicrographs of PC/PS blends with varying reactive components. (a) 0%; (b) 2.5%; (c) 5%; (d) 10%. Each sample was cryofractured and etched for a day at 60°C in cyclohexane.

active blends. Among these reactive blends, the considerable decrease of the domain size shows up to the content of 2.5 wt % of reactive components, but further decrease of domain size can not be obtained with adding more reactive components. The refinement of the microstructure implies the formation of compatibilizing copolymers, which reduce the interfacial tension and stabilize the morphologies.

The morphology in the immiscible polymer blend depends on interfacial tension between the phases, rheological properties of the components and processing conditions, and so on. The morphology in melt state reflects a dynamic balance between break up of dispersed phase caused by stress imposed and phase coalescence driven by the tendency to minimize surface energy. When a molten blend stays in a quiescent or low-stress state, this dynamic balance is altered and morphology may change; that is, phase coalescence may occur. Such coarsening in the dispersed phase will affect the properties of the blend. A number of studies have revealed that in general compatibilized blends kept stable morphology during melt annealing without coarsening in the dispersed domain. The melt annealing effect for PC/PS blend at melt processing temperature 270°C was reported by Cheng et al.<sup>32</sup> They showed that the significant morphological changes occurred less than 2 min under these conditions and such morphological rearrange-

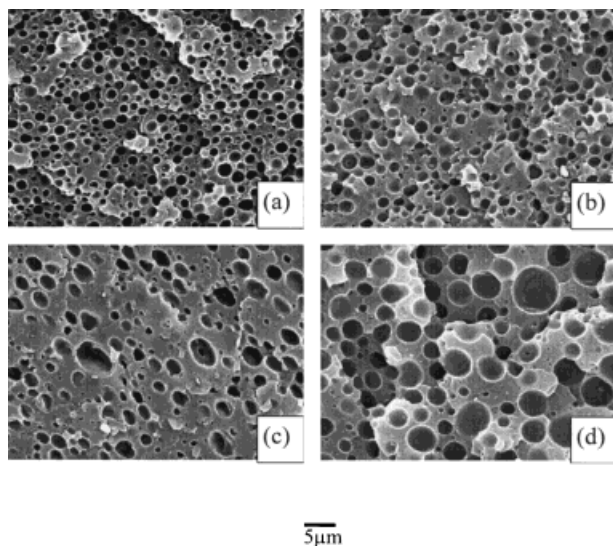
ments were detrimental to the mechanical properties. It is of interest to know about these changes that can occur in the reactive PC/PS blend and nonreactive PC/PS blend; that can be one of the effective tools to evaluate the performance of this PC/PS compatibilizing agent, which was formed through in situ reactive processing. In this study, a melt-annealing experiment was carried out in the Haake mixer. After the 10-min mixing under earlier mentioned processing conditions, 250°C and 50 rpm, the mixer was stopped and left for 10 min more. After then, the sample was taken out carefully and immediately quenched in ice water in order to avoid altering the blend morphology. Figures 10a and 10b show that the reactive blend containing 5 wt % reactive components exhibits little change in the dispersed domain size after melt annealing. However, domain size in the nonreactive blend displays appreciable growth after the melt annealing, as seen in Figures 10c and 10d. These results indicate that the copolymer was formed by the reaction between the reactive components through in situ reactive processing and acted as a compatibilizer. That is, these copolymers reside probably at the interface in the PC/PS blend and suppress the phase coalescence very much.

The result from the measurement of notched izod impact strength with varying amount of reactive components is shown in Figure 11. Little increase in the izod property was found in the reactive PC/PS blends only containing reactive components. However, izod impact strength starts to increase rapidly with addition of just



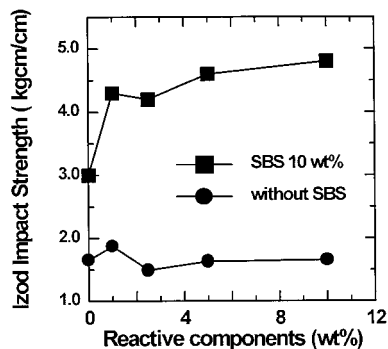
**Figure 9** Number average particle diameter as a function of reactive components for PC/PS/reactive components blends and PC/PS blend.



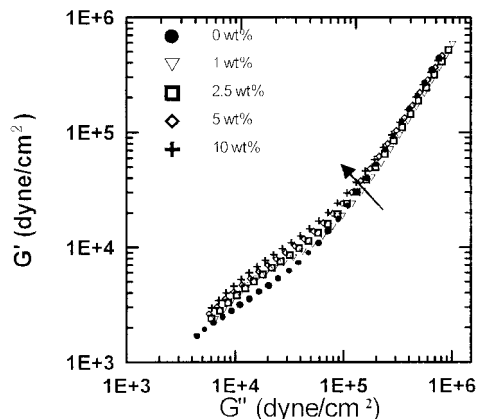


**Figure 10** SEM photomicrographs of PC/PS blends before and after 10 min melt annealing at 230°C (a) before, with reactive component 5%; (b) after, with reactive component 5%; (c) before, without reactive component; and (d) after, without reactive component. Each sample was cryofractured and etched for a day at 60°C in cyclohexane

SBS copolymer as an impact modifying agent in the PS phase. In these cases, 10 wt % SBS copolymer in place of PS components was put into blends. Appreciable enhancement is continued up to 5 wt % and then seems to increase much slowly until 10 wt % of reactive components. Similar experiments were conducted by Triacca et al.<sup>13</sup> for nylon 6/ABS blends. They showed that no improvement of izod impact strength appeared for nylon 6/SAN blend compatibilized by adding styrene maleic anhydride copolymer (SMA) as corresponding reactive components in the SAN phase



**Figure 11** Effect of the amount of reactive components on impact strength with or without SBS copolymer.



**Figure 12** Effect of increasing amount of reactive components on rheological properties,  $G'$  and  $G''$ .

with amine end groups of nylon 6. On the other hand, nylon 6/ABS blends were used the same SMA material as a reactive component showed the increase of izod strength, which relates to the fact that this ABS material contains high rubber content in the size of 0.5 to 1  $\mu\text{m}$ . In general, the reduction of domain size and the improvement of interfacial adhesion by addition of reactive components display the increase of mechanical properties, for example tensile properties and izod impact strength. However, when the material of domain phase deforms in a brittle manner under the test conditions, even though their blend has compatibilized morphology with finer dispersion, narrow distribution of domain size, and good interfacial adhesion, there may not be enhancement of such mechanical property. In this case, effective absorption of fracture energy is not achieved. In case of adding SBS triblock copolymer as an impact modifier, most of SBS copolymer may migrate and locate in the PS phase because of its miscibility with PS. Therefore the PS domain has ductile features and can act as an impact modifier. In those cases effective absorption of fracture energy was made with introducing reactive components.

Rheological properties in the polymer blends can serve as one of the effective tools for evaluating blends. In Figure 12, storage moduli  $\log G'$  for PC/PS blend and PC/PS/reactive components blends are plotted versus loss moduli  $\log G''$  with varying amount of reactive components. Palierne<sup>33</sup> and Lee and Park<sup>34</sup> suggested that the effect of particle size in the polymer blend on rheological properties could be predicted. Theoretically, there is close to  $G' \sim (G'')^2$  at high fre-

quency region and then a plateau region follows at low frequency. This plateau region is directly related to the interfacial tension with the longest relaxation time. The level of the plateau goes up but its width becomes narrow as the decrease of the domain size in dispersed phase. It is mainly due to the additional contributions of interfaces. There are widely reported for many other compatibilized blends.<sup>35</sup> In Figure 12, the plateau region appears from around loss moduli  $G''$   $10^5$  dyne/cm<sup>2</sup>. As loading amount of reactive components increases, these plateaus get higher. These results mean that the domain size in the PC/PS/reactive components blends get smaller with increasing amount of reactive components and compatibilized blends formed successfully. This result is consistent with that of aforementioned morphological observations.

## CONCLUSION

Polycarbonates with anhydride end groups can be prepared through the reaction between trimellitic anhydride chloride and hydroxyl terminated polycarbonate in methylene chloride solvent. Anhydride terminal functionalization was confirmed by <sup>13</sup>C NMR technique. Observation of the torque increase in the reactive blend and FTIR experiments revealed the inter-polymer reaction took place during the melt blending of polycarbonate having anhydride functionality and polystyrene with oxazoline functional groups. The reactive compatibilization of PC/PS blend using PC-anh and RPS as reactive components was obtained. The morphologies of PC/PS/reactive components blends were modestly refined relative to that of the comparable nonreactive PC/PS blend. Morphology in reactive blends showed to be suppressed phase coalescence fairly compared with that of nonreactive blend. Izod impact strength of reactive blends with impact modifier appeared to rise with increasing amount of reactive components. The measurements of rheological properties for the blend showed that the level of plateau goes up with increasing reactive components. This also indicates that adding reactive components makes it possible to form successfully compatibilized blend.

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