

# Blends of Polyamide 6 and Bisphenol-A Polycarbonate. Effects of Interchange Reactions on Morphology and Mechanical Properties

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## SYNOPSIS

Blends of polyamide 6 (PA6) and polycarbonate (PC) were prepared in a Brabender mixer, at 240°C, applying long mixing time, for 45 min. It was observed that the morphology and the mechanical properties tend to resemble those of a homogeneous material as the mixing time and PA6 concentration increase. This is attributed to chemical reactions taking place between the two homopolymers. Acidolysis, amidolysis, and aminolysis, catalyzed by the terminals and the amide groups of the polyamide, should in principle be possible. Our results indicate that the aminolysis is the main process, inducing simultaneously scission of PC chains and formation of PC-PA6 copolymer chains. The latter act as interfacial agents between incompatible PA6 and PC, improving the mechanical properties of PA6-rich blends, in agreement with the predictions of some theoretical models assuming good phase interpenetration. © 1992 John Wiley & Sons, Inc.

## INTRODUCTION

Through continuing developments polymer blends have become a source of new and ever-improving materials. The increased knowledge of the relationships among molecular structures, processing conditions, and final properties has remarkably progressed so that it is often possible to design and produce tailor-made materials with controlled phase structure and morphology. Within this framework, a relatively recent field of investigation involves the blending of high-performance polymers in the attempt to create synergistic effects leading to new "super materials."

Polyamide 6 (PA6) and bisphenol-A polycarbonate (PC) are important commercial polymers, each of them providing outstanding performances in a variety of applications. Nevertheless, they present

some negative aspects that are in some way symmetrical. PA6 is strongly resistant to most solvents while PC is not; PC is insensitive to moisture and dimensionally very stable while PA6 greatly suffers from its considerable hygroscopicity. Taking this into account, it is very logical to consider whether they may complement each other in a blend, overcoming the drawbacks, while maintaining the good properties of parent polymers. As often in the field, the patent literature is vast and intricate,<sup>1,2</sup> involving all major producers of these technopolymers whereas the scientific works on the subject of PC-PA6 blends are relatively scarce.

We have conducted extensive research on this system, pointing out that PC and PA6 are thermodynamically immiscible polymers;<sup>3,4</sup> however, blends with a relevant extent of interactions and interesting properties can be obtained if suitable mixing conditions are applied.<sup>5-7</sup> These previous studies have shown that at high temperatures and long mixing times, PC and PA6 chemically react forming PA6-PC copolymers that act as compatibilizers. Similar results have been obtained by

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Eguiazabal et al.<sup>8-10</sup> In this work we intend to discuss more deeply the effect of the chemical reactions taking place during the melt mixing on the blend morphology and consequently on the mechanical properties of the material.

## EXPERIMENTAL

### Materials

The polymers used in this work were hydrolytic polyamide 6 and bisphenol-A polycarbonate manufactured and kindly supplied by SNIA and Eni-chem, respectively. PA6 had a  $M_w$  of 37,000, PC of 32,000.

### Blending Procedure

The homopolymers, carefully dried in vacuum at 110°C overnight, were mixed in a Brabender Plasticorder model PLE 330 equipped with cam mixer, at 240°C and at a rotational speed of 30 rpm. The mixing time ranged from 15 to 45 min. The compositions of PA6-PC blends examined were 90/10, 75/25, 50/50, and 25/75 w/w. Sheets of about 1 mm thickness were obtained by compression molding in a Carver press at 260°C, under a pressure of 10 MPa and slowly cooled in air. All following characterizations were performed on such sheets.

### SEC Analysis

To separate the polycarbonate fraction, the blends were dissolved in formic acid and centrifuged at 10,000 rpm. After that, all blends gave a clear solution and a precipitate; only for PA6-PC 90/10 no precipitate was observed, and the solution contained some gel. The precipitated PC was thoroughly washed, dissolved in methylene chloride, and chromatographed in a Waters HPLC model 6000 A, equipped with a UV detector 440,  $\lambda = 254$  nm, and a column set microstyragel  $10^5$ - $10^4$ - $10^3$ -500 Å. The separation and the analysis were performed at room temperature.

### Morphology

The morphology of blends was investigated with a scanning electron microscope, model Cambridge Stereoscan 250 MK2. The samples, cut from the same sheets used for the tensile tests, were cryogenically fractured and the surfaces gold coated with a sputtering coating unit, model Agar Aid PS3. The compositions PA6-PC 90/10 and 75/25 were also

observed after etching with methylene chloride for 4, 8, and 24 h at room temperature.

### Melt Flow Index

The melt flow index (MFI) was measured using a melt indexer CEAST. For all samples the conditions were  $P = 2.16$  kg and 230°C. Before testing, the samples were dried by baking them at 110°C for 24 h in an oven in the presence of silica gel.

### Mechanical Properties

Stress-strain curves were obtained with an Instron tensile testing machine model 1122, at room temperature, and an initial velocity gradient of 0.33  $\text{min}^{-1}$ . The tests were carried out on strips ( $5 \times 1 \times 50$  mm) cut out from the compression-molded sheets. The tests were performed both on wet and dry samples. All results are average of 10 measurements.

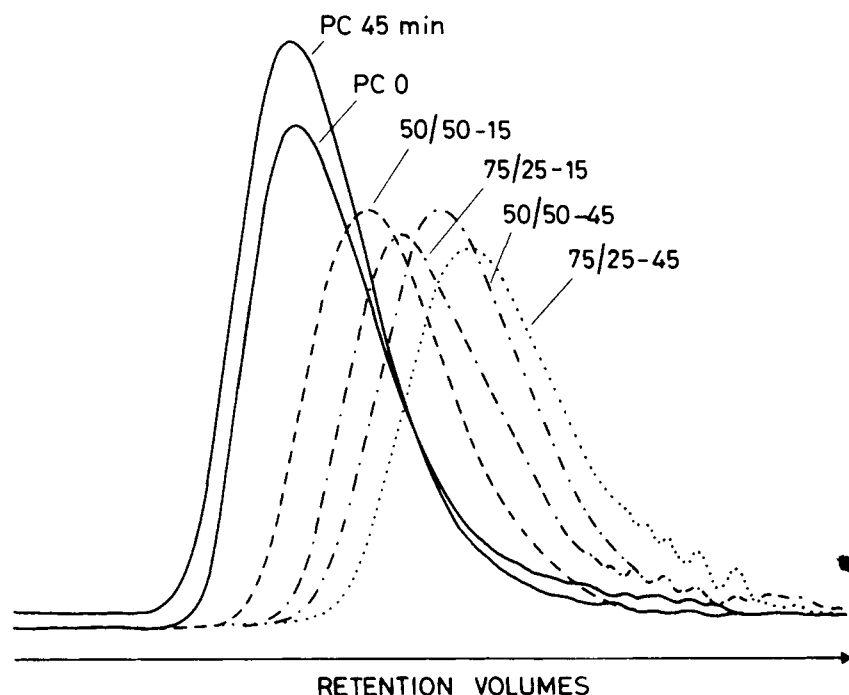
## RESULTS AND DISCUSSION

### Molecular Weight Distribution of Polycarbonate in the Blends after Melt Mixing

During the blend preparation, it was observed that, in some cases, sample discoloration occurred. Furthermore, a thorough analysis of patent literature revealed that degradation can be a relevant problem for such blends. Therefore, before analyzing the properties of the material, we decided to pay some attention to the molecular characterization of the blends.

It is well known that polycarbonate is very sensitive to basic compounds: The carbonate group is easily attacked by alkaline solutions, the chemical bonds destroyed, and the polymeric chain undergoes scissions that can lead to complete depolymerization.<sup>11</sup> Photo and thermal degradation have also been reported.<sup>12-15</sup> During the degradation, volatile compounds and small molecular species such as carbon dioxide, phenol, bisphenol-A, and diphenylcarbonate may be formed. Polyamide 6 has alkaline characteristics, its basicity residing in the nitrogen atom of the amide group and much more in the amine chain terminals.

In order to find out if and to what extent degradation reactions occurred, as effect of melt blending, we extracted the PC from the blends and analyzed the molecular weight distribution (MWD) by size exclusion chromatography (SEC). The chromato-



**Figure 1** SEC of PC as received, thermally treated for 45 min and extracted from PA6-PC blends after 15 and 45 min of mixing at 240°C.

grams of PC extracted from blends of different composition and mixing time are shown in Figure 1. Table I reports the values of weight-average molecular weight  $\bar{M}_w$  and number-average molecular weight  $\bar{M}_n$ , obtained elaborating the SEC curves with a polystyrene-based calibration curve. The compared analysis of these results shows the following:

1. The SEC curves shift toward lower molecular weights as the PA6 concentration in the blend, as well as the mixing time, increases. Beside this shift, the fraction of low molecular weight compounds, visible as shoulders or peaks in the right side of the curves, becomes more and more relevant.
2. The PC average molecular weights  $\bar{M}_w$  and  $\bar{M}_n$  decrease dramatically (e.g., from 33,900 to 9,600 for  $\bar{M}_w$  in the case of 75/25 PA6-PC, after 45 min blending. Interestingly, the concentration of PA6 and mixing time are equally important as can be seen comparing the  $\bar{M}_w$  of PC in the blends 50/50-45 min and 75/25-15 min.
3. The degradative effect on PC is only due to the chemical reactions with PA6 and not to the thermomechanical treatment; in fact, pure PC treated in Brabender for 45 min at

240°C maintains the same MWD of the virgin polymer, as expected when this polymer is carefully dried.

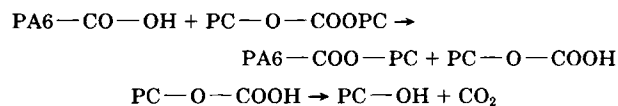
Possible reactions between the two polymers could be represented as follow:

#### Acidolysis

Reaction of PA6 acidic terminals and formation of ester bonds:

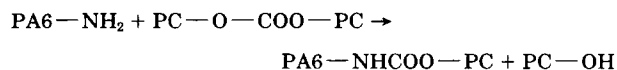
**Table I** Effect of Mixing Time and Blend Composition on PC Molecular Weight Distribution ( $T_{\text{mix}} = 240^\circ\text{C}$ )

Blend Composition PA6/PC	Mixing (min)	$\bar{M}_w \times 10^{-3}$ (g/mol)	$\bar{M}_n \times 10^{-3}$ (g/mol)
0/100	0	33.9	15.5
0/100	45	32.5	15.0
50/50	15	22.6	9.0
50/50	45	13.5	4.3
72/25	15	14.3	4.7
75/25	45	9.6	2.7
90/10	45	No PC extractable	

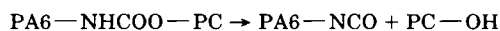


### Aminolysis

Reaction of PA6 amine terminals and formation of urethane bonds:<sup>16</sup>

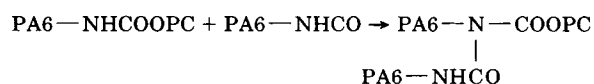


As is well known<sup>16</sup> above 200°C the urethane bonds are unstable and degradate to isocyanate as follows:

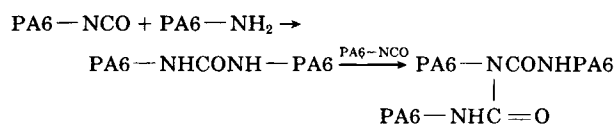


The isocyanates can undergo a large number of reactions:<sup>17</sup> Among the many possible chemical pathways, we would like to underline the following:

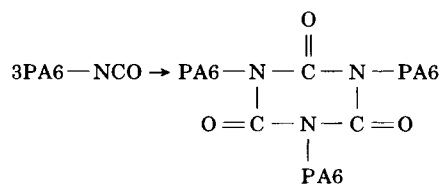
a) Allophanates Formation:



b) Biurets formation via urea compounds:



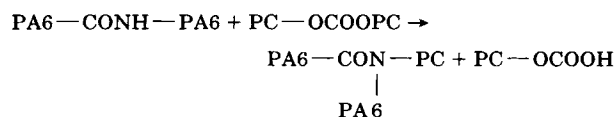
c) Cyclization to isocyanurates:



These reactions are relevant since they lead to branched and crosslinked species that may play a significant role in the properties of the material.

### Amidolysis

Reaction of PA6 amide groups:



This latter reaction is less probable and kinetically slower than the other ones, at least on the ground

of our blending conditions. In fact, the extent of degradation strongly increases with PA6 concentration and confirms that the PA6 terminals are much more active than the amidic groups.

By and large this sketch would account for the formation of linear and branched block copolymers as well as for crosslinked networks and also for the reduction of PC chains down to low molar masses. We would like to stress that PC-PA6 copolymers, insoluble both in formic acid and chloroform, were effectively isolated as reported in Ref. 5.

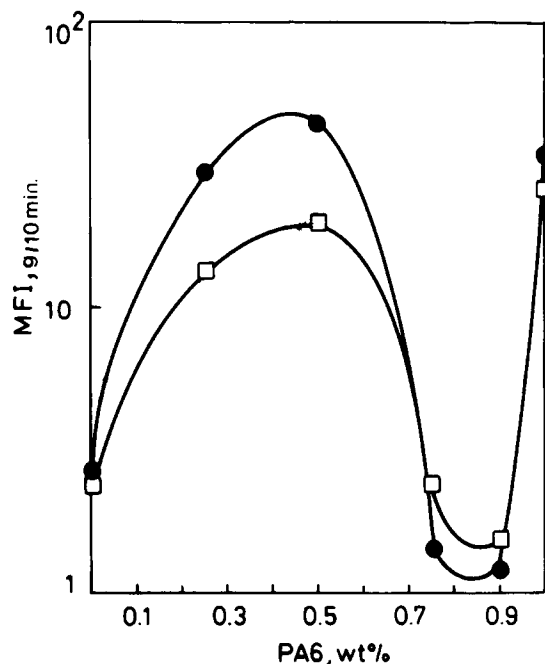
The strong reduction in the PC's  $\bar{M}_w$  suggests that the scission takes place randomly along the chain as an effect of chemical attacks. Although remarkable amounts of oligomers and low mass species are found, we have not detected significant amounts of bisphenol and/or phenol: It is possible that these species further react contributing to the overall degradation with their known catalytic effects.

Also PA6 degradates due to thermomechanical treatment and the catalytic effect provided by developed acidic phenols. Viscosity measurements show that PA6  $\bar{M}_n$  is reduced from 18,600 to about 12,000, as the overall reaction goes on. The solubility tests with concentrated formic acid of 90/10 PA6-PC blend, 45 min mixing, gave a gel-like solution, with no evidence of precipitated PC. That supports the idea that PC should be completely bonded to PA6 chains. Since PC chains are not soluble in formic acid, a gel-like solution could be due to PA6-PC copolymer chains partially soluble and/or to lightly crosslinked material. However, a common solvent of both polymers such as hexafluoroisopropanol, easily dissolves the major part of the above product, while a small amount ( $\approx 10\%$  by weight) remains highly swollen.

This analysis proves that long mixing times and large concentrations of PA6 induce considerable chemical reaction between PC and PA6 with significant reduction of the molecular weight of both homopolymers. Moreover, in extreme conditions (e.g., 10% by weight of PC and 45 min of mixing time) crosslinking is possible, although not extensive.

### Rheological Behavior

The melt flow index (MFI) values of the investigated blends are reported in Figure 2 as a function of PA6 content. The two curves, referring to  $\tau_{\text{mix}}$  equal to 15 and 45 min, present a maximum and a minimum located around 50 and 90% PA6, respectively. To understand the origin of this rather complex shape, it is necessary to take into account the various phys-



**Figure 2** Melt flow index of PA6-PC systems as a function of composition:  $T = 230^{\circ}\text{C}$ ;  $\tau_{\text{mix}} = 45$  (●), and 15 min (□).

ical and chemical interactions occurring in the blends.

First, it must be noted that, as already observed by SEC, the mixing time does not affect significantly the melt viscosity of the pure PC while PA6 is more sensitive to the thermomechanical treatment. In addition, this effect on the pure polymers is much smaller than the large variations of the melt viscosity observed in the blends, which are due to the interactions between PA6 and PC, mainly the chemical transformations occurring during the mixing. The formation of copolymers, as well as a significant reduction of PC molecular weight, drives the viscosity of the system toward opposite direction insofar the degradation reduces the melt viscosity (i.e., increase the MFI) while the presence of copolymers acting as compatibilizers increases the interaction between the phases and then the melt viscosity.

Where the MWD prevails, associated to a very low degree of interactions, namely up to 50% PA6, the MFI curve goes to a maximum, i.e., highest deformability of the two molten phases, while when the interactions are very strong and a small amount of crosslinked material is present, such as in PA6-PC 90/10 blends, the viscosity increases and the MFI curve reaches a minimum. This behavior is emphasized changing the mixing time from 15 to 45 min, since longer processing times induce higher MWD, as shown in Table I, as well as a larger con-

tent of crosslinked material and copolymer when the PA6 percentage is high.

### Morphology

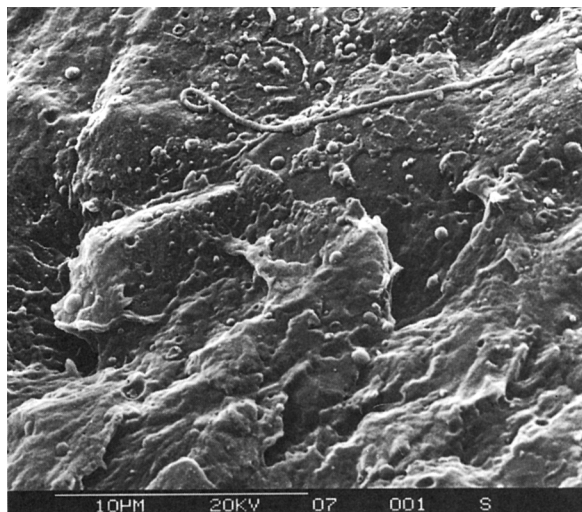
In previous works,<sup>4,5</sup> we have shown that the morphology of PA6-PC blends, prepared in a single-screw extruder, is characterized by the presence of two well-segregated phases over the full range of composition. This is no more the case when the mixing time is increased up to several tenths of a minute (instead of tenths of a second).

Figure 3(a) to (c) shows that after 15 min, PC and PA6 phases are well distinguishable in all the blends studied. However, while in the 75/25 [Fig. 3(b)] and 50/50 [Fig. 3(c)] mixtures the two phases show very poor adhesion, in the 90/10 blend [Fig. 3(a)] partial interaction is observed, with the PC phase more or less strongly bonded to the PA6 matrix. In this case the PC component is mainly dispersed as spherical domains, whose dimensions range between 0.1 and 0.5  $\mu\text{m}$  and are smaller than those of the other blends (0.8–2.4  $\mu\text{m}$  for 75/25 blend), obviously bearing in mind the volume fraction of PC in both blends; moreover, in the 90/10 blend [Fig. 3(a)] it is possible to see some material that we consider to be PA6-PC copolymer, laying over the PA6 matrix. If the blend 90/10 is prepared with a single-screw extruder, the PC phase is dispersed as rodlike domains, whose diameters range from 0.2 to 0.6  $\mu\text{m}$ .<sup>4</sup>

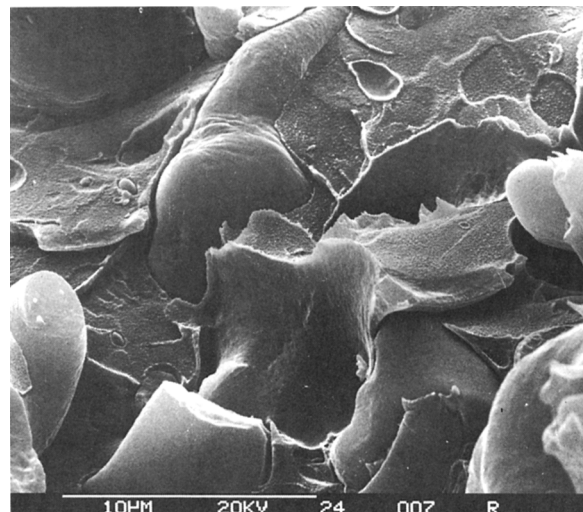
We underline that, as shown in previous works,<sup>3,4</sup> the phase inversion occurs when the PA6 content goes down to about 60% by weight: PA6 is the matrix in the 90/10 and 75/25 blends [Fig. 3(a), (b)], but constitutes the dispersed phase in 50/50 blend [Fig. 3(c)].

On increasing the mixing time to 45 min, the interactions between the polymers increase as well [Fig. 4(a) to (c)]. Apparently homogeneous mixtures are obtained at concentrations of 90 and 75% PA6 [Fig. 4(a) and 4(b), respectively]. In the case of 50/50 blend [Fig. 4(c)], the segregated phase is well defined; however, most PA6 domains are locked in the polycarbonate matrix, owing to a small amount of copolymer material, in situ synthesized during the melt blending, which acts as compatibilizing agent between the two homopolymers.

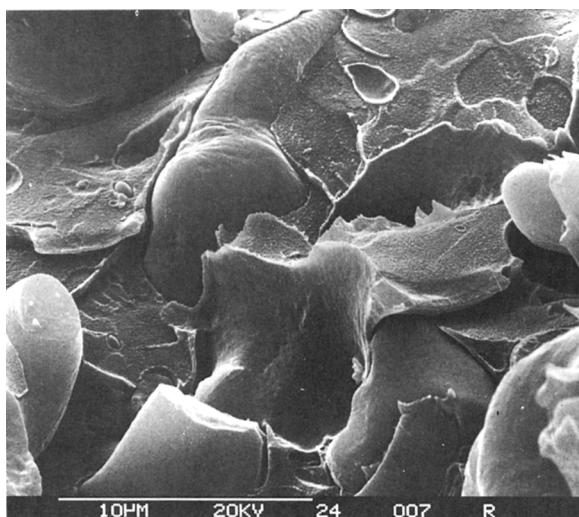
The morphology of 90/10 blend is in agreement with the solubility characteristics discussed previously. In other words this blend, even after 24 h etching with methylene chloride, exhibits an unchanged structure, and no evidence of extraction is observed (Fig. 5); all the polycarbonate is chemically



a



b



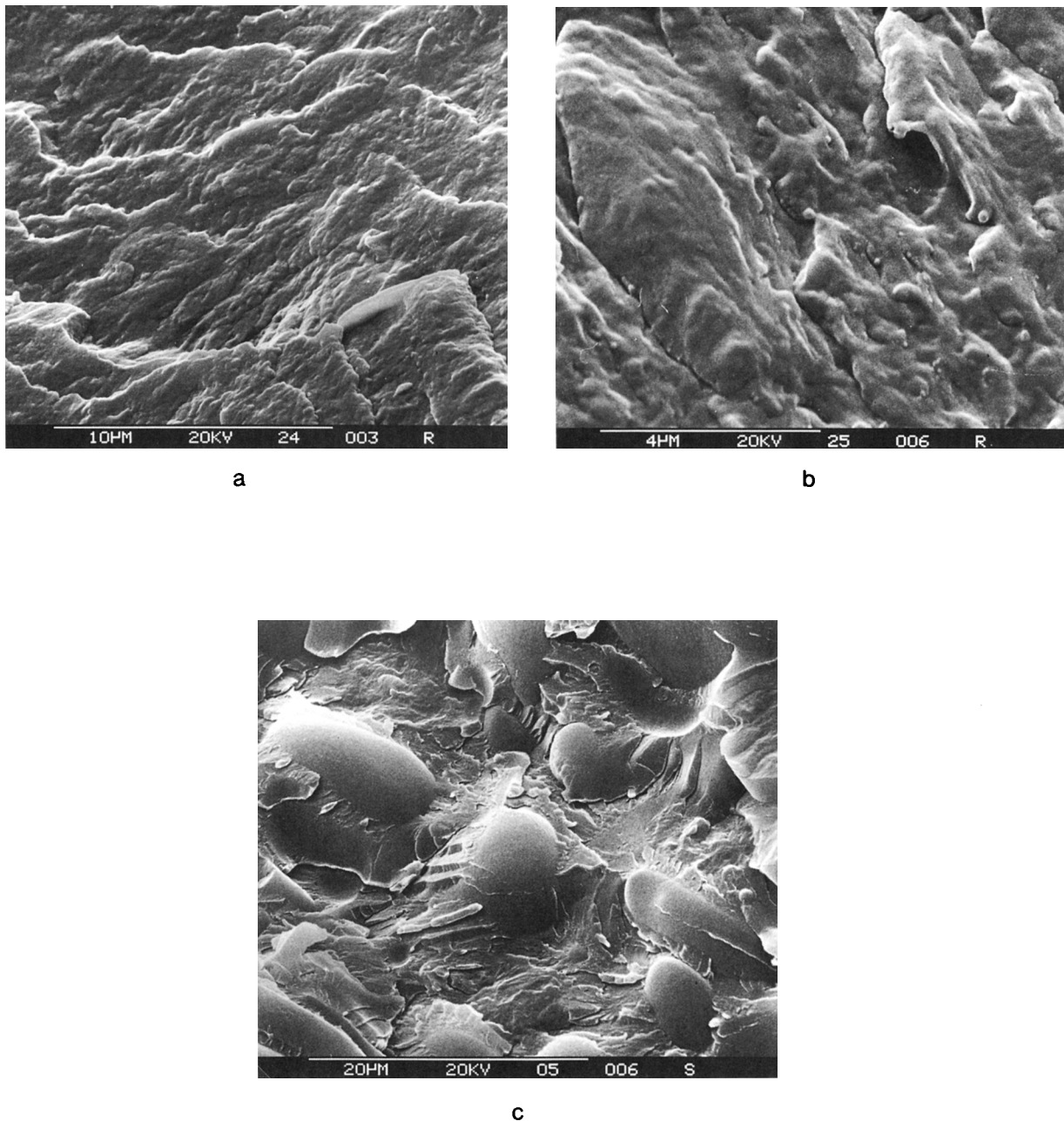
c

**Figure 3** SEM of fracture surface of PA6-PC blends:  $\tau_{\text{mix}} = 15$  min;  $T_{\text{mix}} = 240^\circ\text{C}$ . (a) 90/10; (b) 75/25; (c) 50/50.

linked to the polyamide. On the contrary, pure PC is easily separated from the 75/25 blend. The domains of PC are not easily visible in the microscope, as shown in Figure 4(b), because they are covered by the copolymer material interacting with both homopolymers. This is confirmed by 4 h etching treatment with methylene chloride: The cavities shown in Figure 6 indicate the initial places of the segregated phase of extracted PC.

### Mechanical Properties

Figure 7 shows the elastic modulus of dry samples for  $\tau_{\text{mix}} = 15$  (curve a) and 45 min (curve b) and wet sample for  $\tau_{\text{mix}} = 45$  (curve c), as a function of the PA6 content. Modulus data of PA6- or PC-rich blends are affected by a scattering of about  $\pm 4\%$ , whereas it is of about  $\pm 6\%$  for the intermediate compositions.

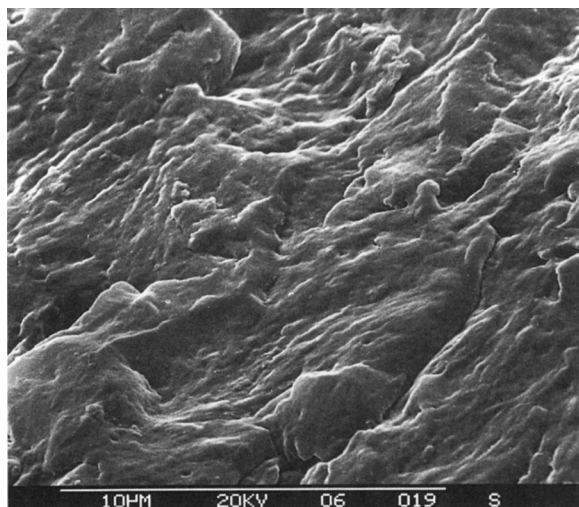


**Figure 4** SEM of fracture surface of PA6-PC blends:  $\tau_{\text{mix}} = 45 \text{ min}$ ;  $T_{\text{mix}} = 240^\circ\text{C}$ . (a) 90/10; (b) 75/25; (c) 50/50.

By and large the trend is the same for all the samples, regardless of mixing time as well as of humidity absorbed from the material. Increasing the polyamide amount, the value of  $E$  diminishes down to a minimum corresponding to the 50/50 blend; then  $E$  increases and reaches a maximum for the blend containing 90% PA6, which is definitely stiffer than the pure polyamide. Modulus values of all the blends are higher, in respect of the pure components,

than those reported for the same systems mixed in a single-screw extruder.<sup>4</sup>

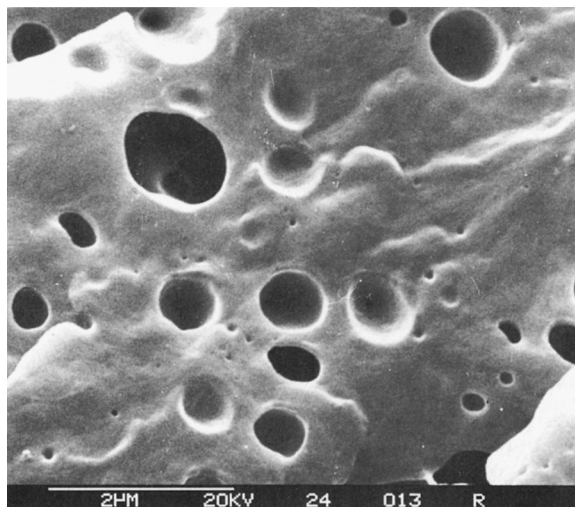
The mixing time clearly affects the modulus for PA6 percentage higher than 50%; its effect, however, is again present even if less remarkable in samples with PA6 content lower than 50%, where practically the same values of  $E$  are obtained. These two apparently different trends can be explained remembering that the morphology of PA6-rich blends pre-



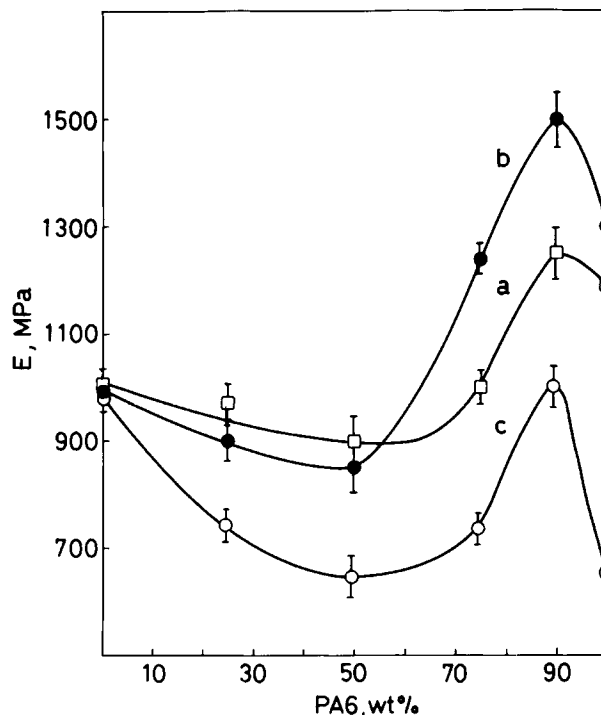
**Figure 5** SEM of fracture surface of PA6-PC 90/10 blend of Fig. 4(a), after 24 h of etching with methylene chloride.

sents PC domains dispersed in the polyamide matrix, while for an amount of PA6 less than about 60%, phase inversion occurs and PA6 is the segregated phase. The 90/10 blend always presents the highest modulus. This could be caused by several factors, such as a different supermolecular crystalline structure of PA6, synergic phenomena, cross-linked material, etc.

As regards the crystalline phase of polyamide, in a previous work<sup>5</sup> we showed that its melting tem-



**Figure 6** SEM of fracture surface of PA6-PC 75/25 blend of Fig. 4(b), after 4 h of etching with methylene chloride.



**Figure 7** Young's modulus of PA6-PC blends as a function of PA6 content. Dry samples:  $\tau_{\text{mix}}$  45 (●) and 15 (□) min; wet samples (○):  $\tau_{\text{mix}} = 45$  min.

perature changes from 220 to 214 and 215.5°C for PC content equal to 0, 10 and 25% by weight, respectively, and  $\tau_{\text{mix}} = 45$  min. Moreover, from the melting heat data we calculate that the crystallinity in 90/10 blend amounts to about 23% and about 32% for pure PA6, while in the other mixtures it corresponds to that of the pure polymer. These results are the same ones reported by us for the blends with the same compositions, but prepared in a single-screw extruder.<sup>3</sup> This indicates that even if a low extent of interaction between PC and PA6 macromolecules is established, it is enough to disturb the crystallization process of polyamide, allowing a lower development of the crystallinity and/or the formation of crystals with higher defect numbers. As a consequence, the final structural characteristics of the PA6 matrix should have a negative influence on the rigidity and the strength of the blends. In our case we feel that the slight increase of the modulus of PA6-rich blends can be attributed to synergic phenomena, promoted by the formation of PA6-PC block copolymers, and to the crosslinking, although crosslinked material is only about 10% by weight and with a very low crosslinking density.

The modulus values of the blends with PC matrix



are about 10% lower than that of pure PC, even if the molecular weight of the latter one is about twice as much as that of PC in the blends. In the previous works<sup>3,4</sup> we reported a dramatic reduction of the tensile modulus for the blends with same composition, prepared in a single-screw extruder and characterized by a foamlike morphology of the PC matrix. The results of Figure 7 for PC > 60% w/w are due to the significant contribution of PA6 through partial adhesion realized during the blending, between the two polymers, as SEM micrographs reveal [Fig. 4(c)].

On the other hand, the large degradative effects of the mixing time on the molecular weight of PC must be invoked to justify the lower values of  $E$  of blends mixed for 45 min in comparison to those obtained after 15 min.

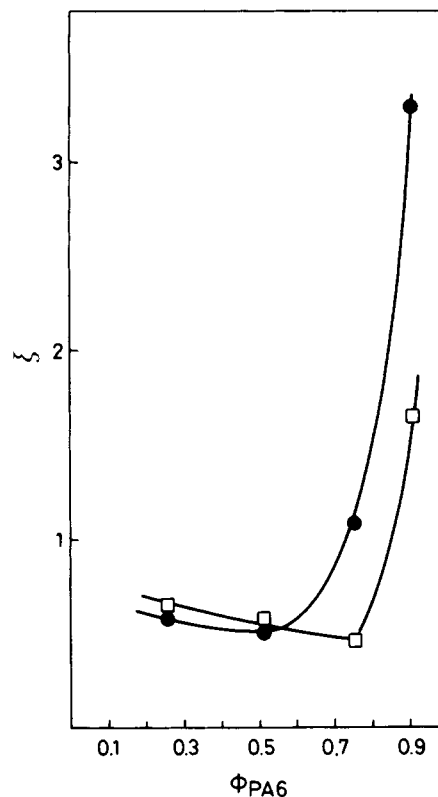
As expected all wet samples are less rigid, proportionally to PA6 weight fraction, but interestingly the difference between wet and dry samples, normalized over the PA6 content, seems to be constant. This would suggest that the presence of PC does not influence the PA6 water uptake at the equilibrium.

To give further indications about the structure of these blends, we will briefly consider here the evaluation of the fraction of reinforcing material in the filler space,  $\xi$ , according to Dobkowski.<sup>18</sup> By extending the suspension model of Guth<sup>19</sup> and its parallel voids model, Dobkowski suggested that the parameter  $\xi$  can be defined as follows:

$$\xi = \frac{E_m}{E_d} \left( 1 + \frac{E_b - E_m}{E_m \phi_d} \right) \quad (1)$$

in which  $E_d$  is the elastic modulus of the dispersed phase. The values give indications about the structure of the blend. In particular,  $\xi = 1$  means perfect adhesion,  $\xi < 1$  means presence of voids, and  $\xi > 1$  means that the macromolecules are penetrating their neighboring material at the boundary. The  $\xi$  values, calculated using the data relative to the dry materials of Figure 7, are reported in Figure 8. For the blends with PA6 content larger than 75% and mixed 45 min, the  $\xi$  values are larger than 1, indicating an interpenetration between the two phases. On the contrary at lower PA6 content  $\xi$  is less than 1 suggesting the presence of voids in the blends. For the blends mixed for 15 min,  $\xi$  is larger than 1 only at very high PA6 concentration.

The strength and the elongation at break shown in Figures 9 and 10 present analogous trends characterized by a minimum at 50/50 composition,



**Figure 8** Fraction of reinforcing material in the filler space as a function of volume fraction of polyamide in the blend.  $\tau_{mix} = 45$  (●) and 15 (□) min.

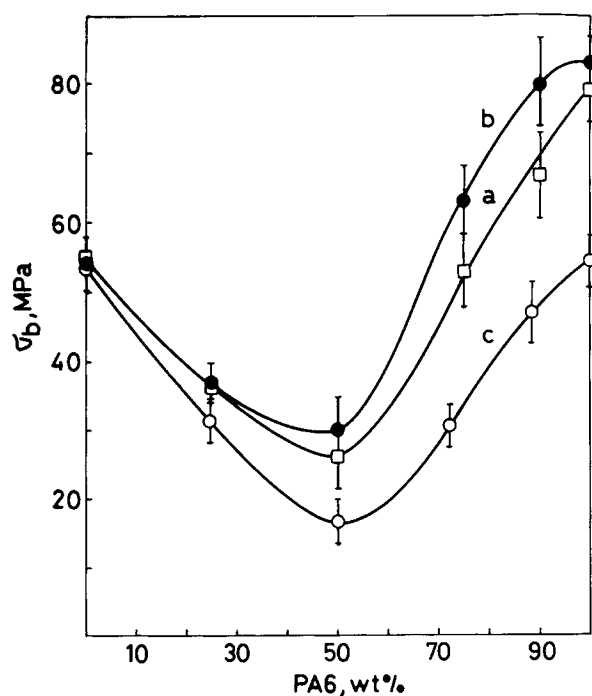
clearly the weakest material. A scattering of about  $\pm 10\%$  and  $\pm 20\%$  characterizes strength and elongation data, respectively, of PA6- or PC-rich blends. In the case of 50/50 blends the larger error ( $\pm 16\%$  and  $\pm 23\%$ , respectively) could be due to the presence of rodlike structures [Figs. 3(c) and 4(c)] with higher stress concentrations than the spherical inclusions [Figs. 3(a), (b) and 5]. Differently from tensile modulus, tensile strength (Fig. 9) is very little influenced by the high miscibility that characterizes the PA6-richest blend. However, as expected, the reduction of the molecular weight of PC is responsible for the very low strength of the 50/50 and 25/75 PA6-PC blends. Still the 90/10 blend has the best properties, further improved if  $\tau_{mix}$  is 45 min (curve b), but always inferior to those of pure PA6. As expected the wet samples (curve c) always display lower values than the dry ones (curves a and b).

As regards the elongation at break,  $\epsilon_b$ , the trend shows again a minimum at 50/50 concentration (Fig. 10). More precisely for PA6 content lower than 50% w/w, the time of blending does not show any

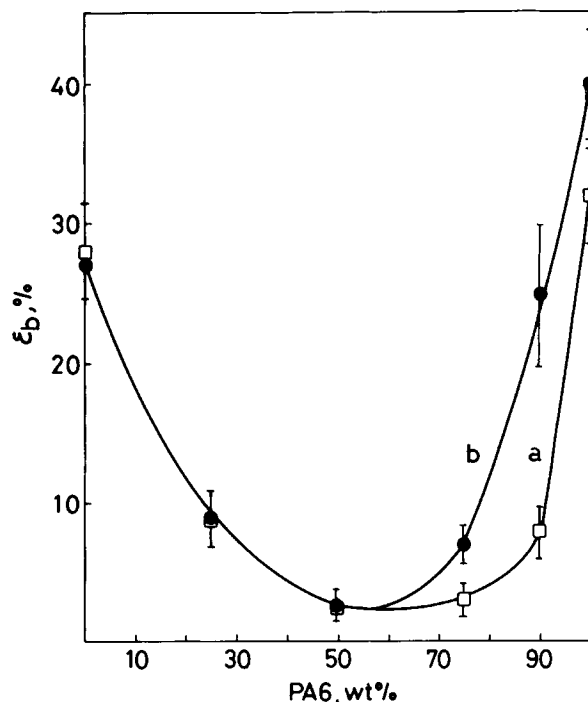
influence, while  $\epsilon_b$  depends on it when that percentage extends beyond 50.

## CONCLUSIONS

In previous work on PA6-PC systems, prepared in a single-screw extruder at 260°C, we showed that these polymers are incompatible and that only in polyamide-rich blends can some interactions seem to be present. The mechanical properties are very poor and both the elastic modulus and the tensile strength are much inferior to those of the pure components. In this work we have reported evidence that an intensive mixing of PC and PA6, in the melt state for long times at 240°C, produces blends with increased compatibility. This is due to the interchange reactions between the polymers leading to the formation of block copolymers, linear and cross-linked, as well as other mixed molecular species. As a consequence, morphology, mechanical properties, and solubility are profoundly affected and tend to resemble those of a single-phase material for high polyamide percentage. However, the mechanical properties are still lower than the pure components, with the exception of the 90/10 mixture, which pre-



**Figure 9** Tensile strength of PA6-PC blends as a function of PA6 concentration. Dry samples:  $\tau_{\text{mix}} = 45$  (●) and 15 (□) min; wet samples (○):  $\tau_{\text{mix}} = 45$  min.



**Figure 10** Elongation at break of PA6-PC blends as a function of PA6 concentration. Dry samples:  $\tau_{\text{mix}} = 45$  (●) and 15 (□) min.

sents the highest modulus, maintaining also good strength and elongability. Nevertheless, we underline that the properties of these blends are better than those of the corresponding samples prepared in the single-screw extruder.

The reactions depend on mixing time and PA6 concentration thus suggesting a primary role of terminal groups of polyamide chains, and mainly  $-\text{NH}_2$ . Since synergic phenomena clearly affect the mechanical properties of PA6-rich blends, an extension of this work centered on the exploitation of  $-\text{NH}_2$  terminals in PA6 molecules to achieve a good extent of compatibilization in the processing time, acceptable by the industry, is at present under way and will be the matter for further study.

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