

Effect of the mixing procedure on the morphology and properties of compatibilized polypropylene/polyamide blends

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Blends containing equal amounts of polypropylene (PP) and polycaprolactam (PA6) were prepared by extrusion mixing, using 5% by weight of polystyrene-*block*-poly(ethylene-*stat*-butylene)-*block*-polystyrene, modified by grafting with maleic anhydride (SEBS-MA), as the compatibilizer precursor. The blends were obtained using three mixing procedures, representing different ways of introducing the compatibilizer. The blends were prepared by (A) one-step mixing of the three components, (B) mixing a preblend of SEBS-MA and PP with PA6, and (C) mixing a preblend of SEBS-MA and about one-third of the total amount of PA6 with PP and the rest of the PA6. All of the blends contained dispersed PA6 domains, surrounded by a separate interphase of the self-assembling compatibilizing polymer. The morphology of blends A and B were similar and markedly different from that of blend C, which contained dispersed phase domains of two clearly distinguishable populations. Impact strength and elongation at break were lowest for C. The poor performance of blend C is due to the fact that a considerable fraction of the compatibilizer ended up in small, well dispersed phase domains with a high weight ratio of compatibilizer to PA6. © 1998 Elsevier Science Ltd. All rights reserved.

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INTRODUCTION

Many polymer pairs are incompatible in the sense that they have to be combined with a suitable compatibilizing agent in order to produce useful blends^{1–3}. Blends of incompatible polymer pairs are often prepared by extrusion mixing of the two polymers with either (a) a graft or block copolymer, having segments that may separately interact with each of the incompatible polymers, or (b) a functionalized polymer which reacts with one or both of the polymers, forming a compatibilizing polymer. The latter procedure has recently attracted a great deal of interest. It is often referred to as *in situ* compatibilization^{2–5}, to emphasize that the compatibilizer is formed during the mixing process.

The primary aim of the present study was to compare morphology and impact strength of compatibilized blends of polypropylene (PP) and polyamide-6 (PA6), having the same overall composition but prepared either using *in situ* compatibilization or by the addition of a preformed compatibilizer. The blends studied were prepared in processes differing only with respect to the order in which the three polymer components were mixed. Thus, in all cases, the same polystyrene-*block*-poly(ethylene-*stat*-butylene)-*block*-polystyrene (SEBS) triblock copolymer, modified by grafting with maleic anhydride (SEBS-MA), was used as the precursor for the compatibilizing polymer. This means that the compatibilizing polymer in all of the blends was a PA6 grafted SEBS-MA, formed under melt mixing conditions, in a reaction involving PA6 amino end groups and succinic anhydride groups on SEBS-MA⁶.

Although compatibilization of polymer blends has been the subject of many studies, very few have dealt with the

effect of mixing order. Cimmino *et al.*⁷ reported that rubber-modified PA6, with fine grain morphology and of higher impact strength, was obtained if the compatibilizer precursor (a rubber substituted with succinic anhydride groups) was first premixed with the rubber, instead of the three components being mixed in a single-step mixing process. Willis and Favis⁸ studied blends of PA6 with PP, or polyethylene (PE), compatibilized by a commercial ionomer. This study found that blends were formed with finer morphology than those prepared by one-step mixing. These blends were obtained by mixing PA6 with a pre-mix of the ionomer in the polyolefin. A coarser morphology was obtained if the polyolefin was mixed instead with a preblend of the ionomer in PA6. The morphology differences observed were partly ascribed as being due to the higher capacity of the compatibilizer to interact with polyamide than with PP, which would inhibit its transport from the PA6 phase to the interface. Dagli and Kamdar⁹ recently studied blends of polyethylene terephthalate and high density PE, using as the compatibilizer precursor a copolymer of ethylene and glycidyl methacrylate. They found that the finest morphology was obtained by adopting a one-step mixing process, i.e. using *in situ* compatibilization.

EXPERIMENTAL

Materials

The following commercial polymeric materials were used: isotactic polypropylene (MFI = 5), PY 6100 from Shell, polyamide-6 ($M_n = 18\,000$), Ultramid B3 from BASF and Kraton FG 1901X from Shell. The latter polymer is used as the compatibilizer precursor and is referred to as SEBS-MA. It has been reported that this is obtained by

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grafting a parent SEBS triblock elastomer with about 2 wt% of maleic anhydride¹⁰.

The polyamide was dried at 80°C for 20 h before processing. The other materials were used as received.

Processing

Blends and preblends were prepared by melt mixing, using a Berstorff ZE 25 × 43 D co-rotating intermeshing twin-screw extruder. The screw speed was 105 rpm and, except for the feed zone, the barrel set temperature was 250°C. The extruder was fed by dry blended pellets of the materials to be mixed. The feed rate (3.5 kg h⁻¹), corresponding to a residence time of about 2.4 min, was the same in all the blending operations.

Injection moulding of test bars for tensile testing and Charpy impact testing (SIS 161 351) were performed in a Engel ES 200/50 HL injection moulding machine. Moulding of the blends was carried out using a barrel temperature of 250°C and a mould temperature of 60°C.

Blend composition and mixing procedures

All blends were prepared from 47.5 parts PP, 47.5 parts PA and 5 parts SEBS-MA. The use of 5 wt% of SEBS-MA was found to be sufficient to produce a well compatibilized blend¹¹.

The final blends were prepared using three different mixing strategies (A, B and C), as described below.

- (1) A dry blend of all three polymer components (PP, PA and SEBS-MA) was fed to and mixed in the extruder.
- (2) In the first mixing step, SEBS-MA and PP were melt mixed to obtain a preblend containing SEBS-MA dispersed in PP. The final blend was obtained by mixing this preblend with PA6.
- (3) In the first mixing step, PA6-grafted SEBS-MA was prepared by melt mixing PA6 and SEBS-MA in the weight ratio 73.9/26.1. In the second mixing step, 100 parts of this preblend were mixed with 247.5 parts PP and 174.1 parts PA6 to obtain a final blend of target composition.

Morphology

The morphology of the blends was studied by transmission electron microscopy (TEM) using a Jeol 100 U instrument. Thin sections (thickness down to 80 nm) for TEM studies were cut with a glass knife in an LKB Ultratome V ultramicrotome supplied with a cryo-kit. Sample temperature was -80°C and knife temperature -40°C. A 50/50 DMSO/water mixture was used as trough liquid. The sections were exposed to vapour from a freshly prepared solution of ruthenium tetroxide for 20 min to develop contrast by staining the polystyrene domains in the SEBS-MA. The polyamide was also slightly stained by ruthenium tetroxide.

Values given for the average size of the dispersed phase domains were calculated from data extracted from TEM images.

Mechanical properties

Impact strength behaviour of dried (80°C for 20 h) and conditioned (50% relative humidity for 96 h at 23°C) blend samples was measured on injection moulded test bars, using a CEAST 6545/000 pendulum.

Tensile stress-strain curves were determined at room temperature by measurements on dried (80°C for 20 h) injection moulded bars in a Schenk tensile testing machine,

using a crosshead speed of 50 mm min⁻¹. It has to be pointed out that the rate of water transport in compatibilized blends of the present type is much slower than in pure PA6, and is also dependent on the morphology of the blend¹¹.

Thermal properties

The melting and crystallization behaviour of the blends was studied by differential scanning calorimetry (d.s.c.) (Mettler DSC 30), using samples (5–10 mg) sealed into aluminium pans. In order to avoid any influence from the previous thermal history of the blend, the sample was first heated to 250°C at a heating rate of 10°C min⁻¹. The crystallization process was then followed as the sample was cooled to -150°C at a rate of 10°C min⁻¹. The melting behaviour was studied by again heating the sample to 250°C at 10°C min⁻¹. The crystallization and melting temperatures were taken as the temperatures corresponding to the peak values of the crystallization exotherms and melting endotherms, respectively.

RESULTS AND DISCUSSION

The results to be presented demonstrate that the morphology and other properties of three-component polymer blends, prepared by extrusion mixing, can differ considerably depending on the mixing procedure adopted. The results presented are from the study of three blends, all made up from 47.5 parts PP, 47.5 parts of PA6 and 5 parts SEBS-MA, but prepared using the three different mixing procedures. The compatibilizer precursor (SEBS-MA) was a triblock thermoplastic elastomer, the elastomeric mid-block of which contains succinic anhydride side groups. In the present blends, the actual compatibilizer is the graft copolymer formed as succinic anhydride groups on SEBS-MA react with amino end groups on the polyamide. In all of the blends this reaction occurred in the extruder, i.e. under melt mixing conditions.

The different mixing procedures used (A, B and C) represent different ways of introducing the compatibilizer precursor (SEBS-MA) into the blend. In procedure A, a one-step mixing process, the component polymers were mixed during a single passage through the extruder. Procedures B and C both involved two extrusion mixing steps. In procedure B, a preblend of SEBS-MA and PP was prepared in the first step, and mixed with PA6 in the second step. In this case, the compatibilizer was formed in the second mixing step by a reaction at the interface between PA6 and a PP phase containing small, unassociated particles of SEBS-MA (see below). In procedure C, the compatibilizer was prepared in the first step by melt mixing of SEBS-MA with a part of the total amount of PA6. In the second step, this preblend, which contained 30% of the total amount of PA6 in the final blend, was mixed with PP and the remaining PA6. Assuming that SEBS-MA contained 2 wt% of succinic anhydride groups (see EXPERIMENTAL), the PA6/SEBS-MA mixing ratio used in the first step of procedure C would correspond to a (polyamide amino end groups)/(succinic anhydride groups) ratio of about 0.8.

Morphology

The morphology of the blends prepared using mixing procedures A, B and C is illustrated in *Figure 1*. The micrographs were obtained by transmission electron microscopy on thin sections taken from pellets of the final blends. The sections were taken at right-angles to the flow direction. Sections taken parallel to the flow direction showed that the

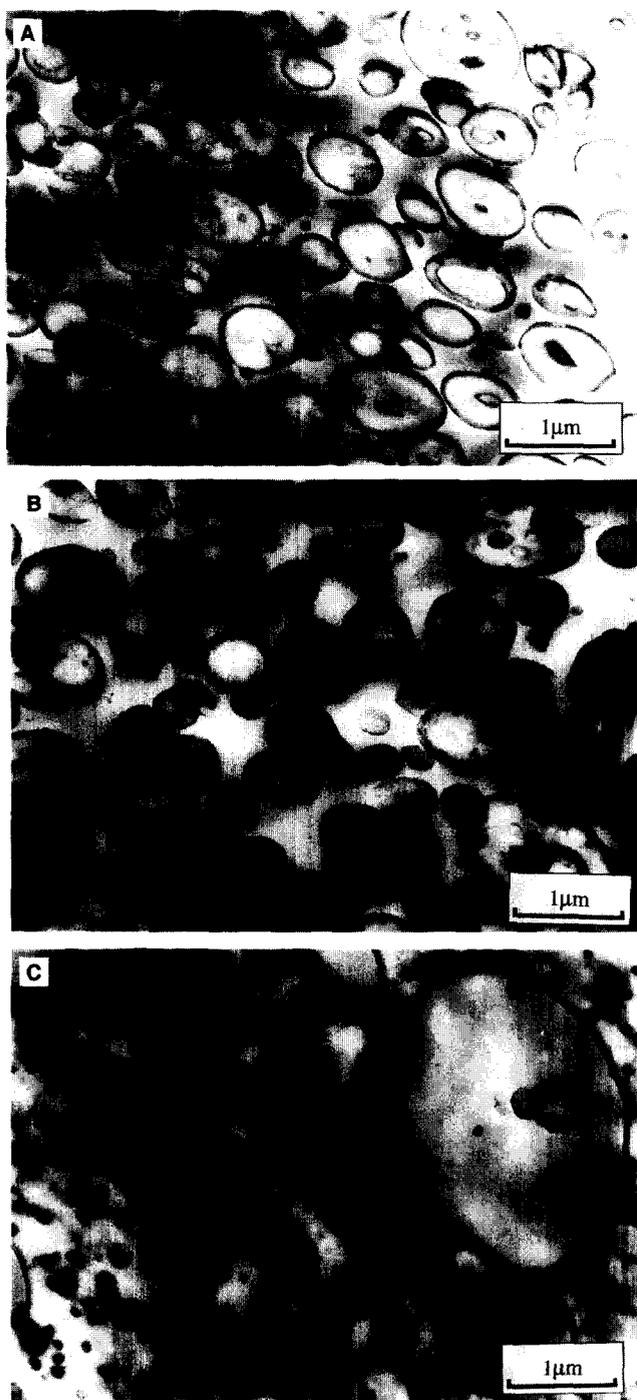


Figure 1 The morphology of blends prepared from equal amounts of PP and PA6 plus 5% by weight SEBS-MA using mixing procedures A (a), B (b) and C (c), as revealed by transmission electron microscopy of thin sections taken at right-angles to the flow direction and stained by ruthenium tetroxide



Figure 2 The morphology of the blend prepared with mixing procedure A, as observed by transmission electron microscopy of a thin section taken along the flow direction and stained by ruthenium tetroxide



Figure 3 A close-up of the morphology of blend A (see *Figure 1a*) showing the structure of the interphase surrounding inclusions of PP in dispersed PA6 phase domains

PA6. The dark spots in these areas correspond to polystyrene microdomains, which are responsible for the self-assembling properties of the compatibilizer.

Blends A and B seem to have rather similar morphologies, differing mainly with respect to the size of the PA6 domains. The thin sections cut at right-angles to the flow were examined and the average widths of the PA6 domains was estimated to be $0.36 \mu\text{m}$ in A and $0.85 \mu\text{m}$ in B. In both cases the dispersed PA6 domains contain inclusions of PP surrounded by a layer of the compatibilizing phase. The presence of a structured interphase around the PP inclusions in blend A is clearly seen at the higher magnification; see *Figure 3*.

The morphology of the blend obtained by mixing procedure C differs considerably from that of the other blends. In this case, the continuous phase contained dispersed phase domains of two different sized populations; see *Figure 1c*. One of these consists of large PA6 phase domains, most of which were free of inclusions. These domains are much larger in size than the PA6 domains in blends A and B. There is also a large population of small particles with sizes down to about $0.1 \mu\text{m}$. A close inspection of the micrograph indicates that most of the small particles have a core of PA6. Although a separate core

dispersed phase domains, seen in *Figure 1*, represent cross-sections through a mixture of elongated (length/diameter ratio up to about 10) large volume phase domains and small volume phase domains of near spherical shape; see *Figure 2*. The contrast derives from staining with RuO_4 , which in this system primarily reacts with and stains the polystyrene end blocks of SEBS-MA. On the basis of arguments outlined elsewhere¹¹, the continuous phase can be identified as PP and the dispersed phase as PA6. The dark spotted areas separating the continuous and dispersed phases represent the separate compatibilizing phase of SEBS-MA grafted by

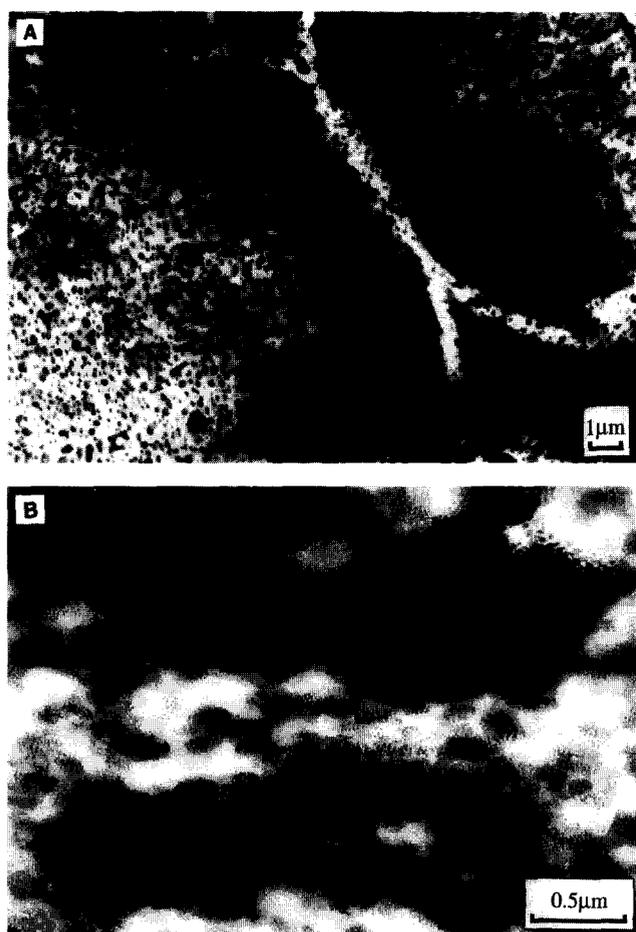


Figure 4 The morphology of preblends obtained by extrusion mixing of (a) PP with SEBS-MA, and (b) PA6 with SEBS-MA

cannot be seen in the smallest particles, these might contain PA6 solubilized in the structure by association with the PA6 grafts on the grafted SEBS-MA. The presence in the PP phase of small dispersed particles surrounded by grafted SEBS-MA born in a PA6 environment proves that the compatibilizer has a relatively high affinity for PP.

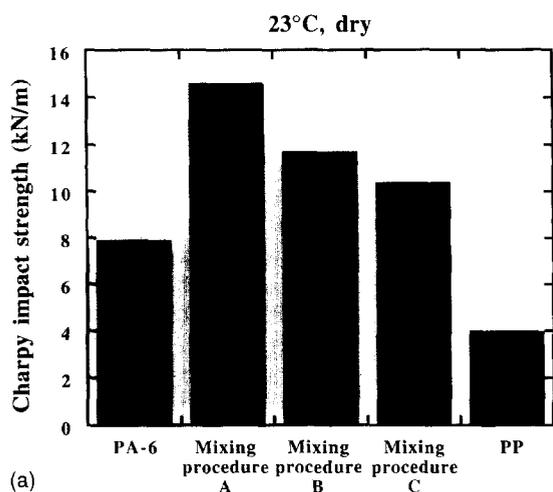
The preparation of the present blends involves a chemical reaction (grafting of SEBS by PA6) and several physical processes. These include complex transport processes, melting of PP and PA6, self-assembling of ungrafted and grafted SEBS-MA and adsorption of polyamide-grafted SEBS-MA at the PP/PA6 interface. In mixing procedure A, all of these processes occur under conditions determined by the temperature and shear rate profiles along the extruder and on a time scale determined by the feed rate to the extruder. By using a two-step mixing procedure, the various processes are to a certain extent separated in time and space. This, of course, is the reason for the observed differences in morphology.

The solidified premix of SEBS-MA and PP obtained in step one of procedure B consists of a dispersion in PP of small, well separated particles of SEBS-MA; see *Figure 4a*. The average size of these particles is about $0.1 \mu\text{m}$. The melt viscosity of the preblend was slightly lower than that of its pure components¹¹, indicating that the melt also contained dispersed aggregates of SEBS-MA and that these are flexible¹². Aggregates of SEBS-MA similar to those observed in the preblend were not found in the final blends from either procedure A or B. The morphology of

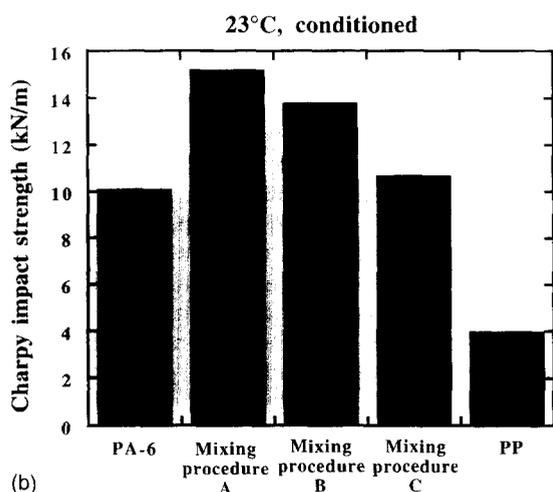
blend B demonstrates that small particles of SEBS-MA dispersed in PP, see *Figure 4a*, undergo efficient grafting at the PP/PA6 interface and that the graft copolymer formed remains at the interface. The PA6 domain size distributions of the final blends (see above) indicate that the total surface area of the PA6 domains, i.e. the total PP/PA6 interface minus that of the PP inclusions in the PA6 domains, was much smaller in blend B than in A. In terms of maximizing the total surface area of the PA6 domains, procedure B does not seem to offer any advantages over the simple one-step mixing procedure A.

The morphology of the preblend of SEBS-MA and PA6 obtained from the first step of mixing procedure C is illustrated in *Figure 4b*. This micrograph shows a large number of slightly dark spotted structures, having a characteristic size of about $0.2\text{--}0.4 \mu\text{m}$ and, in the upper right corner, a considerably larger and more heavily stained particle. Structures of the latter type infrequently appeared in micrographs from this preblend, and can be identified as unreacted SEBS-MA. The numerous spotted structures correspond to agglomerates of SEBS-MA, grafted by PA6, possibly containing some solubilized unreacted polyamide. The polystyrene microdomains in the odd large particle are considerably larger than those in the agglomerates of grafted SEBS-MA. This shows that the grafting induced a change in the polystyrene microdomain structure of the thermoplastic elastomer. The micrograph suggests that the individual agglomerates of grafted SEBS-MA are associated and tend to form a network. Associated agglomerates of grafted SEBS-MA were most likely also present in the melt state. This suggestion is strongly supported by results from a parallel study of the viscosity of a preblend of polyamide 66 and SEBS-MA, containing the same relative amounts of SEBS-MA and polyamide¹¹. In this study, the preblend was found to have a melt viscosity (at 270°C and 100 s^{-1}) about 15 times higher than that of the pure polyamide and more than twice that of the pure SEBS-MA. The tendency of the agglomerates to associate in the melt implies that grafting was not sufficiently extensive for the agglomerates to become sterically stabilized in the polyamide melt, to prevent bridging through common polystyrene microdomains.

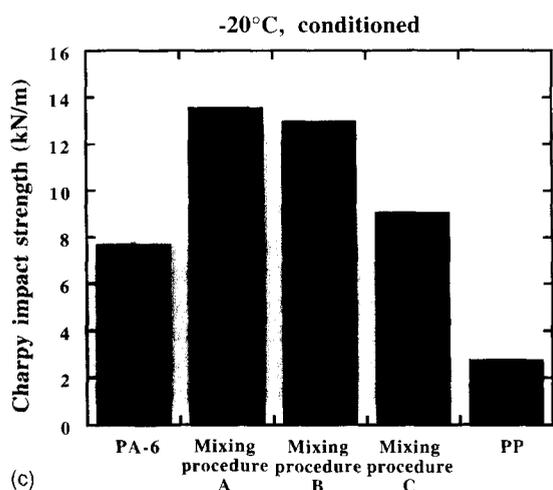
The main morphological features of the final blend C, as compared to those of blends A and B, namely the large PA6 domains with infrequent inclusions and also the large number of small particles dispersed in the continuous phase, may be explained in the following way. During mixing step 2, the molten preblend interacts nearly simultaneously with two melt phases PP and PA6. Instead, because of its relatively high affinity for the PP phase, agglomerates or associated agglomerates of grafted SEBS-MA are transferred to the PP phase. This process involves rearrangement of the agglomerates in order to favour contacts between PP and ungrafted mid-block segments of SEBS-MA; this forces the polyamide grafts and unreacted PA6 associated with the agglomerates towards the core of the particles. This explains the presence of small and well dispersed particles in the PP phase of the final blend. The population of large PA6 phase domains mainly originates from the quantity of PA6 added in the second step. The formation of these domains would most likely involve the dilution of molten preblend by newly added PA6, dispersion of this part of the melt in the PP matrix, and stabilization of the PP/PA6 interface by adsorption of grafted SEBS-MA from the PA6 phase. The size of the resulting PA6 domains would be determined by the amount of compatibilizer available, i.e.



(a)



(b)



(c)

Figure 5 Charpy impact strength of blends prepared by mixing procedures A, B and C and for the pure main blend components (PA6 and PP), as determined on (a) dried samples at 23°C, (b) conditioned samples at 23°C, and (c) conditioned samples at -20°C

the original amount of compatibilizer in the preblend minus the amount of compatibilizer present in the small phase domains dispersed in the PP matrix.

Mechanical properties

All of the blends showed a higher impact strength than either the pure PP or pure PA6 at temperatures above and below the glass transition temperature of the continuous PP phase; see *Figure 5*. This behaviour confirms previously

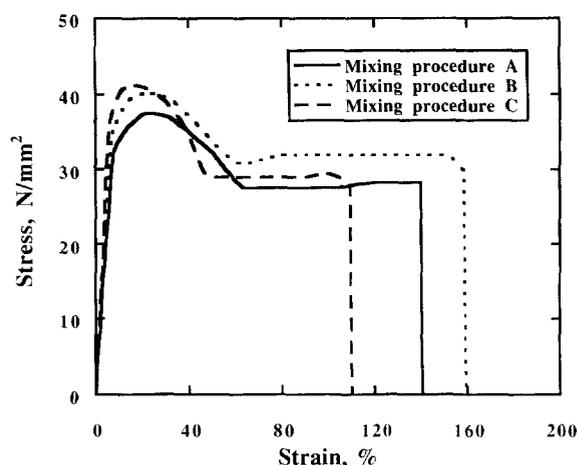


Figure 6 Tensile stress-strain curves at room temperature, as obtained by measurements on blends prepared by mixing procedures A, B and C

published results demonstrating that SEBS-MA is an efficient compatibilizer precursor for PP/PA6 blends^{11,13}. The good impact strength of the blends indicates that the compatibilizing interphase between PP and PA6 (*Figure 1*) adheres strongly to both phases, is sufficiently thick and of adequate toughness¹⁴. The ability of the interphase to adhere to PP, as well as to PA6, is a consequence of the amphiphilic nature of the grafted thermoplastic elastomer. This polymer probably interacts with the PP phase through highly flexible, nonpolar poly(ethylene-co-butylene) segments and with PA6 through its polyamide grafts. The polystyrene micro-phase domains would confer cohesive strength to the interphase polymer.

The impact strength of the blends varied considerably depending on the mixing procedure used (*Figure 5*). For all the experimental conditions used, the impact strength was found to decrease in the blend order A > B > C. These differences can be related to differences in morphology, in particular to differences in the size distribution of the dispersed phase and the thickness of the interphase layer. The average dispersed phase domain size obtained by blending procedure A appears to compare favourably with reported values of the optimum size of the dispersed phase in rubber-modified polypropylene¹⁵. In blend C, the small SEBS-MA-rich particles dispersed in the PP phase are probably too small to influence the impact strength of the material¹⁵. The comparatively low impact strength of this blend would therefore be related to the relatively large size of the PA6 domains originating from the amount of PA6 added in the second mixing step. In addition, the compatibilizing layer surrounding these domains could be thinner than the corresponding layers in blends A and B. This is because, in blend C, a considerable fraction of the compatibilizer is bound to ineffective small phase domains.

The effect of the mixing procedure was also reflected in the stress-strain behaviour of the blends. This is illustrated by the stress-strain curves in *Figure 6* and by the data for elongation and stress at yield and break in *Table 1*. For all of the blends the ultimate failure was preceded by necking and cold drawing, indicating a high degree of ductility. The results show, however, that the different mixing procedures produced large differences in elongation at break and also significant differences in initial stiffness. Elongation at break increased in the order C < B, whereas initial stiffness increased in the order A < C. It is not obvious how the observed behaviour can be related to the blend morphology.

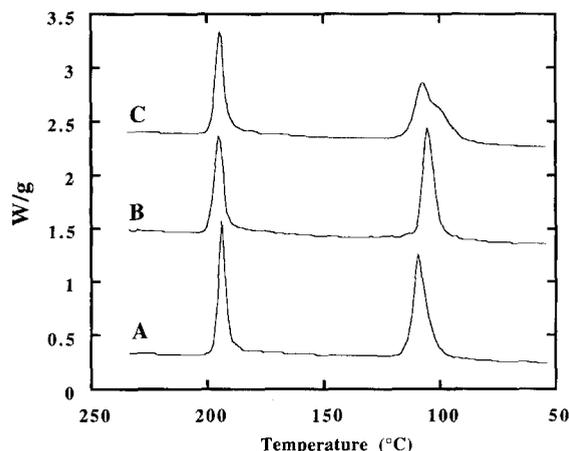
Table 1 Data for stress (σ) and elongation (ϵ), at yield (index y) and break (index b), as obtained from tensile stress-strain measurements on the blends and the pure main blend components (PP and PA6) (the error limits correspond to the standard deviation from measurements of ten samples)

Material	σ_y (N mm ⁻²)	ϵ_y (%)	σ_b (N mm ⁻²)	ϵ_b (%)
PP	43.7 ± 1.1	20.0 ± 0.5	26.2 ± 0.4	560 ± 10
Procedure A	38.1 ± 1.2	26.1 ± 0.0	28.9 ± 1.1	140 ± 5
Procedure B	39.2 ± 0.5	23.5 ± 0.0	30.5 ± 0.2	159 ± 6
Procedure C	40.0 ± 0.3	17.6 ± 0.4	28.7 ± 0.2	111 ± 4
PA	64.9 ± 1.2	23.0 ± 0.6	77.0 ± 0.9	403 ± 15

Table 2 Average cross-sectioned size of polyamide domains in blends A, B and C, and the apparent heat of crystallization (ΔH_c) for PP and PA6 in the blends^a

Mixing procedure	Diameter of PA6 domains (μm)	ΔH_c for the PP phase (J g ⁻¹)	ΔH_c for the PA6 phase (J g ⁻¹)
A	0.36	42.9	35.7
B	0.85	41.4	34.0
C	0.14 and 1.20	46.9	31.8

^aThe PA6 domain sizes were calculated from TEM images of thin sections taken at right-angles to the flow direction, and the heat of crystallization from the low (PP) and high (PA6) temperature crystallization exotherms of d.s.c. cooling curves (Figure 7)

**Figure 7** D.s.c. cooling curves showing the crystallization behaviour of blends prepared using different mixing procedures

It can be conjectured, however, that, all other parameters being equal, the elongation at break would increase with an increase in the thickness of the interphase layer. Considering the fact that a considerable amount of the compatibilizing phase in blend C is not located at the interface between the large PA6 domains and PP, the thickness of the interphase layer might well be expected to vary in the same order as the elongation at break.

From the observations described above it can be concluded that, in the present system, the use of a preformed compatibilizer is not advantageous.

Thermal properties

A comparison of d.s.c. cooling curves for the three blends is presented in Figure 7. The exotherm peaks for blends A and B at about 190 and 110°C correspond to crystallization of PA6 and PP respectively. With blend C, the low temperature exotherm seems to be composed of two overlapping crystallization peaks. The values for the heat of crystallization corresponding to the area below the different exotherm peaks in Figure 7 are presented in Table 2. The data show that, for blend C, the high temperature exotherm is smaller and the low temperature exotherm larger than the corresponding ones for blends A and B. The likely explanation of this is that, in blend C, the polyamide nucleates and crystallizes in two temperature regimes. The crystallization peak at about 190°C would correspond to crystallization of the polyamide present in the large PA6 domains, and the crystallization peak overlapping the crystallization of PP to crystallization of polyamide in some or all of the small particles dispersed in the continuous phase of blend C; see Figure 1c. This interpretation is in agreement with observations made in a recent study of PP/PA6 and PP/PA66 blends stabilized by different amounts of SEBS-MA¹¹. In this study a blend containing PA6 domains

of average size 0.11 μm was found to produce a d.s.c. crystallization peak at about 80°C, whereas no d.s.c. crystallization peak was observed for a blend containing PA66 domains with an average size of 0.23 μm .

The amount of slowly crystallizing PA6 in blend C can be roughly estimated by comparing the apparent heats of crystallization for the blends; see Table 2. Using the averaged value for blends A and B as the reference, the apparent excess heat of crystallization of PP and the apparent deficit in heat of crystallization of PA6 correspond to 44% and 29% respectively of the amount of PA6 used in the first step of mixing procedure C. The comparison suggests that a considerable portion of the PA6, from the first mixing step, is present in PA6 domains with a diameter probably not exceeding 0.2–0.3 μm . On the average, these small particles would contain PA6 and SEBS-MA in the same proportion as the preblend, which implies that in blend C probably more than 30% of the compatibilizer is associated with PA6 domains, too small to affect the impact strength of the material.

CONCLUSIONS

The effective use of SEBS-MA as a compatibilizer precursor for PP/PA6 blends prepared by extrusion mixing requires that the grafting reaction occurs at the PP/PA6 interface and not in the PA6 phase.

Interfacially grafted SEBS-MA (blends A and B) remains at and stabilizes the interface at which it is formed, whereas grafted SEBS-MA formed by mixing SEBS-MA with PA6 (blend C) stabilizes the blend only if adsorbed at a newly created PP/PA6 interface. In preparing the final blend C, preformed aggregates of grafted SEBS-MA from the preblend were either adsorbed at the PP/PA6 interface or passed into the PP phase, where they rearranged and formed small, well dispersed particles with a high compatibilizer/PA6 weight ratio. Therefore a large proportion of the compatibilizer was used to stabilize PA6 domains too small to contribute to the impact strength of the blend, the rest to stabilize PA6 domains of fairly large size. The crystallization behaviour of PA6 indicated that more than 30% of the compatibilizer was present in the small phase domains.

The polystyrene microdomains in the compatibilizing phase are smaller in size than those in the original ungrafted SEBS-MA. This is an indication that grafting, possibly in combination with solubilization of unreacted polyamide, is accompanied by a restructuring of the thermoplastic elastomer phase.

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