

Chemical Interaction in Blends of Polyamide and Butyl Rubbers

J. D. (Jack) Van Dyke,¹ Marek Gnatowski,² Andy Koutsandreas,² Andrew Burczyk,³ Scott Duncan³

¹Trinity Western University, 7600 Glover Road, Langley, BC, V2Y 1Y1 Canada

²Polymer Engineering Co. Ltd., 110-3070 Norland Avenue, Burnaby, BC, V5B 3A6 Canada

³Defence Research Establishment Suffield, Box 4000, Medicine Hat, AB, T1A 8K6 Canada

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ABSTRACT: This study focuses on the interaction between polyamide and butyl or bromobutyl rubbers blended in a high shear environment. The fact that these two normally incompatible systems can be mixed is explained by a chemical reaction that occurs between polyamide and the butyl rubber components during processing. Measurement of the melt viscosity and differential scanning calorimetry of these blends, along with analysis of the extracted soluble butyl rubber component, supports the presence of small quantities of block or graft polymers in the system, signifi-

cant crosslinking during the blending process, and possibly other interactions between the blend components. The effect of electron beam radiation on interaction in these blends was briefly evaluated and was found to increase crosslinking in the blends, with some degradation of the polyamide component. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 89: 980–991, 2003

Key words: crosslinking; rubber; graft copolymers; blends; thermoplastics

INTRODUCTION

Thermoplastic elastomer (TPE) compositions in rubber applications are increasingly being made from blends of two or more homopolymers. Successful blends are dependent on the compatibility of the individual components, the use of compatibilizing agents, and the method and intensity of the mixing process used to prepare the blends.¹ When compatibilizing agents are present, otherwise incompatible polymers can be blended to yield polyblends or alloys with unique properties. Commonly, block and graft copolymers that possess segments with chemical structures or solubility parameters similar to those of the polymers being blended are effective compatibilizing agents. It is well known that these may be preformed and added to the mixture of polymers undergoing compatibilization, or they may be generated *in situ* by reaction between coreactive functional groups on the polymers or free-radical generation and recombination reactions.^{1–3}

The practical uses for blends of polymers are affected by the properties of the components used in the mixture. In our laboratories there has been a strong interest in TPE materials with barrier properties. These materials have a variety of uses, including applications as automotive parts, specialty hoses, ma-

chine parts, protective clothing for military applications (masks, gloves, and/or footwear), and protective films. It is usually understood that if the components of a blend have barrier characteristics, the blend will also have barrier properties.

Polyamides (PA) and butyl rubbers are two useful homopolymers that we have investigated as candidates for thermoplastic barrier compositions. Both are well known for their barrier characteristics as homopolymers.^{4,5} Polyamides, however, have limited impact resistance and a high flexural modulus,² whereas butyl rubbers display typical elastomeric characteristics.⁵ When butyl rubber (IIR) and its halogenated derivatives, such as bromobutyl rubber (BIIR), are blended with polyamides, the result is a softer material with a reduced flexural modulus. These two materials, however, have widely differing solubility parameters and surface energies. In the absence of compatibilizing agents, it is expected that blends of these two materials will exhibit large separation between the phases, and likely produce blends that are not very useful.

A literature search on blends of polyamides with various types of rubber shows activity on blends of this nature over the last 10–15 years.^{2,6–12} Dynamic vulcanization is one method described in the literature to prepare blends with useful properties. Blends have also been prepared using specific PA/IIR compatibilizers.¹³ PA can also be blended with maleic anhydride (MA)-containing polymers using the MA groups as coreactive functional groups with the amide groups of

Correspondence to: J. Van Dyke.

PA.^{3,14,15} Ionomers of polyacrylate have been used to compatibilize butyl rubber and PA.¹⁶ Several patents have already been filed on polyamide/butyl rubber blends, with applications in hose compositions impermeable to gasoline, fuel oils, kerosene, and fluorinated hydrocarbons, as well as gas barrier layers in pneumatic tires. Most of the literature focuses on the practical aspects of blending, such as material selection, mixing procedures, and mechanical properties. This study attempts to examine the interaction of butyl rubbers with polyamide during high shear mixing. Interaction between the two polymer systems is expected to include grafting and block formation between the two polymers, crosslinking in the butyl phase, polymer degradation, and crystallinity changes in the polyamide component.

In this investigation, blending experiments were undertaken to produce various compositions of polyamide (20–70%) with butyl or bromobutyl rubber. No vulcanization agents were added into the mix, and care was taken to avoid significant crosslinking of the components during blending. Evidence of compatibility between the phases was sought by examination of the phase microstructure using scanning electron microscopy (SEM), and a comparison of tensile properties, especially elongation. Reactivity differences between polyamide and either butyl or bromobutyl rubber in the blends were investigated through the melt viscosity of their blends, a determination of the amount of insoluble (crosslinked) material produced during blending, and the effect of blending on the melting temperature of the polyamide component. Finally, an attempt was made to observe the presence of graft polymer through infrared analysis and microanalysis of the soluble components in these blends.

Polyamide 12 was used in most experiments because this polyamide has often been used in previously published work, and has a relatively low melting temperature. A high shear environment was used to facilitate an expected *in situ* formation of compatibilizing agents by polymer chain rupture, radical formation, and recombination into small amounts of graft or block copolymers.^{17,18} In some cases maleic anhydride modified ethylene-propylene polymers were added as compatibilizing agents.

EXPERIMENTAL

Materials

Polyamide 12 (Grilamid L25) was obtained from EMS-American Grilon (Sumter, SC); butyl rubber (Polysar Butyl 402) and bromobutyl rubber (Polysar X2) from Bayer Chemical (Sarnia, Canada); and maleic anhydride grafted ethylene-propylene rubber (Fusabond MF-416D) from Dupont Canada (Mississauga, Canada).

Blending procedure

All blends were made in a 258-cm³ Brabender mixer at a target temperature of 190°C and a mixing speed of 15 rpm. Polyamide 12 was first added and mixed for 1–2 min until melted. When Fusabond modifiers were added as compatibilizing agents, they were premixed with the molten polyamide component for 7 min. This was followed by addition of the rubber component (bromobutyl or butyl rubber), and a further mix for about 8 min. If no Fusabond modifier was added, the rubber was added after the polyamide resin had melted, and the mixing process continued for 8 min. After mixing, the blend was removed from the mixer and spread onto a flat metal surface for cooling to avoid further reaction.

Testing

To determine the effect of heating time on the melt viscosity of a blend, the materials were tested for melt flow rate after selected heating time intervals. Melt flow rate, evaluated according to ASTM D1238 with conditions of 200°C and 5 kg weight, was taken as an indicator of melt viscosity. Materials were retained in the extrusion plastometer at 200°C for a prescribed heating interval, followed by measurement of melt flow rate, and the percentage change in melt flow rate was calculated relative to the initial melt flow rate obtained after 10 min of heating.

A number of samples prepared for laboratory analysis were exposed to electron beam radiation with doses of ≥ 100 kGy. Because each pass through the electron beam produced a 50-kGy dose, samples scheduled for doses > 50 kGy were passed through the beam several times to achieve their required dose. Between each 50-kGy dose the trays were allowed to cool before a subsequent pass through the electron beam.

For selective solvation on polyamide/rubber blends, a 0.25-g sample of the blend in the form of small cut pieces approximately 1 mm in size was suspended in reagent-grade hexane (Anachemia, Montreal, Canada) and shaken for 48 h, followed by centrifugation of the insoluble material. Portions of the soluble filtrate were deposited onto a NaCl disk for Fourier transform infrared (FTIR) analysis using a Mattson Galaxy Series FTIR 3000 instrument (Thermo Nicolet Corp., Madison, WI). A number of larger samples were extracted in a Soxhlet extractor using hexane as the solvent. After recovery and ashing of the extracted polymer, the total nitrogen content was determined by the Kjeldahl method.

Differential scanning calorimetry (DSC) was undertaken on the blends by ASTM D3417 using a Perkin-

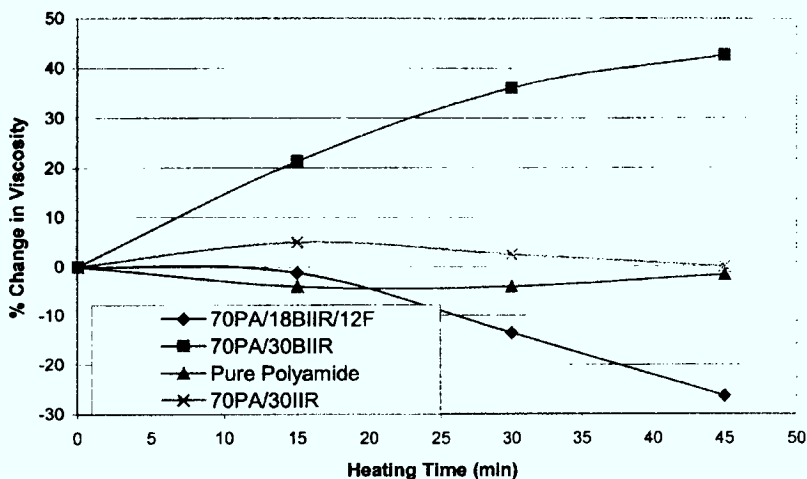


Figure 1 Change in melt viscosity with heating time (200°C, 5 kg).

Elmer DSC-7 instrument (Perkin Elmer Cetus Instruments, Norwalk, CT).

Scanning electron microscopy (SEM) was conducted using a variable pressure LEO 1455VP (Meridian Scientific Services, Stittsville, Canada). After cutting to size, the 40/60 polyamide 12/rubber blends were mounted on aluminum stubs with carbon paint, and examined without coating at low pressure using a Robinson backscatter detector.

Mechanical properties of blends were tested on specimens cut from compression-molded sheets or extruded films (dumbbells or strips), or prepared directly by injection molding. Compression-molded sheets of 2 mm thickness were prepared at 190°C for 10 min, and subsequently cooled and removed from the mold. Films of 1 mil thickness were made using a $\frac{3}{4}$ -in. Brabender mixing screw and a 4-in.-wide ribbon die heated to 190°C. Injection molding was done at high speed with the heating zones of the machine set at 180°C. Molded specimens were Type I according to ASTM D638M. Mold temperature was maintained near 20°C. An Instron 4400 universal testing instrument was used to determine stress/strain characteristics on compression-molded samples (ASTM D412), injection-molded samples (D638), and films (D882). Hardness values (Shore A and D) were determined by ASTM D2240.

RESULTS AND DISCUSSION

Melt viscosities

Figure 1 indicates the change in melt viscosity with time of several single or multicomponent polymer systems, consisting of polyamide homopolymer, two component blends of polyamide 12 with butyl or bro-

mobutyl rubber, and three component blends of polyamide, bromobutyl rubber, and Fusabond MF-416.

The melt viscosity of polyamide and polyamide/butyl rubber blends remains relatively constant over time, indicating relatively little chemical reaction or coalescence in these materials at melt temperatures. When bromobutyl rubber is used in the blend, there is a 43% increase in melt viscosity after 45 min of heating. The cause for this increase may be an increase in molecular weight, possibly through grafting and/or crosslinking that take place in these blends at this temperature. When Fusabond MF-416 is added to the mixture, the viscosity is reduced over time at these temperatures. (Fusabond may catalyze degradation in the polyamide phase, possibly resulting from hydrolysis in the maleic anhydride component and consequent increase in acidity.)

DSC data for polyamide and polyamide/rubber blends

Table I contains DSC data for polyamide 12 and for blends of polyamide/butyl and polyamide/bromobutyl rubber. Results are also listed for ternary blends that include Fusabond MF-416.

Compared to PA homopolymer, the polyamide component in PA/BIIR blends shows a slight melting point decrease of approximately 1°C, and a heat of fusion increase of approximately 1 J/g. The release of HBr from the bromobutyl rubber component during processing, with subsequent partial hydrolysis, molecular weight reduction, and increased crystallinity of the polyamide component, may contribute to these changes.¹⁹ Much larger changes are seen in blends treated under electron beam, where unzipping and molecular weight reduction (in addition to crosslink-

TABLE I
DSC Data for Polyamide and Polyamide/Rubber Blends^a

Composition	Material description	Main peak ^b (°C)	Heat of fusion corrected ^c (J/g)
Polyamide 12	Film	178.2	39.7
70PA/30IIR	Film	178.0	38.9
70PA/30BIIR	Film	177.4	40.7
70PA/30BIIR	Film (EB-treated 250 kGy)	175.0	44.6
70PA/18BIIR/12F	Film	178.2	42.1
70PA/18BIIR/12F	Film (EB-treated 250 kGy)	175.5	46.0
50PA/30BIIR/20F	Film	177.6	49.0

^a Based on samples tested in duplicate.

^b Expected error $\pm 0.15^\circ\text{C}$.

^c Enthalpy calculated based on weight of polyamide present in blend, expected error $\pm 1\%$.

ing) are known to take place, resulting in reduced melting points and increased crystallinity. The addition of Fusabond MF-416 to the blend may contribute to a reduction in the molecular weight of the polyamide component, as seen in increased values of heat of fusion for the PA component. In blends of PA and IIR no reduction in melting point or increase in crystallinity takes place, indicating limited interaction between IIR and PA.

Percentage (%) insolubles of blends

All nonprocessed butyl and bromobutyl rubber components used in these blends are soluble in hexane. Polyamide 12 homopolymer is insoluble in this solvent, and Fusabond is partially soluble. Several blends prepared from mixtures of PA with IIR, BIIR, and

BIIR/Fusabond mixtures were extracted with hexane, and the results are listed in Table II.

Mixtures of polyamide with either rubber component show significantly higher than expected values of % insolubles, based on the fraction of polyamide and Fusabond in the blends. In both mixtures the results suggest that there is some reaction and crosslinking within the rubber component during processing; however, this reaction is apparently more prominent in polyamide/bromobutyl rubber blends than in comparable butyl blends. When exposed to electron beam treatment, all blends show higher % insolubles, which likely results from the crosslinking reaction of butyl, bromobutyl, and polyamide components in the presence of electron beam radiation.

The presence of maleic anhydride grafted polymers increases the insoluble content and likely the

TABLE II
Percentage of Insolubles in Hexane-Extracted Samples of Polyamide/Butyl or Bromobutyl Blends

Sample	Expected % insolubles	% Insolubles after blending ^a	% Insolubles after blending/irradiation (250 kGy)
100PA	100	100	
70PA/30IIR	70	79	
70PA/30BIIR	70	97	
70PA/18BIIR/12F	77 ^b	99	
50PA/50BIIR	50	84	
50PA/30BIIR/20F	62 ^b	85	94
40PA/60IIR	40	42	
40PA/60BIIR	40	48	
30PA/70IIR	30	33	
30PA/70BIIR	30	35	
20PA/80IIR	20	21	
20PA/80BIIR	20	41	67
20PA/48BIIR/32F	39 ^b	52	86
100IIR	0	1	5
100BIIR	0	3	74
100F		60	

^a Based on samples tested in triplicate. Standard error of ± 1 .

^b Calculated on the basis that polyamide (PA) is 100% insoluble, and Fusabond (F) 60% insoluble.

TABLE III
Percentage Nitrogen in Extracted Samples of Pure Rubber and Blends

Sample	% Nitrogen ^a
40PA/60IIR, soluble portion	0.07
40PA/60BIIR, soluble portion	0.07
IIR	0.02
BIIR	0.02

^a Detection limit for this method is 0.01% nitrogen.

crosslinking in all blends. This additive also appears to enhance the crosslinking reaction under electron beam irradiation.

Microanalysis results of soluble material of blends

Hexane extraction of blended samples of PA/IIR and PA/BIIR produces a soluble component consisting mainly of butyl or bromobutyl rubber, respectively. If either of these rubbery components has undergone grafting or block formation with polyamide during the blending process, there should be a small nitrogen content in the soluble component, attributed to graft or block polyamide in the rubber component.

Several blends of butyl and bromobutyl rubber with polyamide were extracted with hexane and compared to pure samples of IIR and BIIR that had been put through a similar heat and shear treatment. Table III shows that the extract from blends of PA with IIR and BIIR was higher in nitrogen content than were the control samples of IIR and BIIR. Nitrogen enrichment in the extracted rubber portion of these blends, although relatively small, is consistent with a grafting or block reaction taking place between PA and these rubbers during processing. The relative difference between the butyl and bromobutyl rubber systems is not visible in numbers this small. Nitrogen detection in pure butyl or bromobutyl rubbers may be associated with the presence of an antioxidant.

Infrared spectra of soluble portion of blends

Figures 2 and 3 contain FTIR spectra of the hexane soluble portions of blends of polyamide 12 with butyl or bromobutyl rubber. If a grafting reaction has occurred, certain characteristic absorption bands attributed to polyamide should be present in these spectra. Figure 4 contains the FTIR spectra of the various component homopolymers in the blends. Because of its insolubility in hexane, the spectrum for polyamide 12 in Figure 4 was obtained from a chloroform solution, whereas all others were obtained from solutions in hexane.

Two absorption bands characteristic of polyamide polymer, 3292 cm^{-1} (N—H stretch) and 1640 cm^{-1} (C=O stretch), indicate the presence of a small yet

significant proportion of polyamide polymer in the soluble portion of the blends. Because polyamide 12 homopolymer is insoluble in hexane, this is most likely attributable to a bromobutyl-g-polyamide copolymer present in the mixture after blending of polyamide with pure bromobutyl rubber or bromobutyl/Fusabond blends. This component is detectable in all blends containing bromobutyl rubber (i.e., 70PA/30BIIR, 50PA/31BIIR/19F, 20PA/80BIIR, and 20PA/50BIIR/30F mixtures). It appears that the same reactions in bromobutyl rubber that cause increased crosslinking and higher % insolubles result in higher levels of grafting and/or block formation between polyamide and the rubber system.

Graft and/or block copolymer is also present in mixtures made with butyl rubber but to a lesser extent, observed by lower intensities in the polyamide absorption bands (1640 and 3292 cm^{-1}). This is attributed to the lower reactivity of butyl rubber compared to that of bromobutyl. These spectra also indicate that when Fusabond MF-416 is used in these blends, it can be seen in the soluble fraction of the mixtures. In most cases the soluble portions of these blends also show the probable presence of stabilizers added to the rubber, evidenced by peaks at 1740 and 1710 cm^{-1} .

The presence of graft polymer in the soluble component of 20PA/80BIIR blends exposed to electron beam radiation appears to be reduced. This is attributed to the relative ease with which polyamide 12 can participate in free-radical crosslinking reactions promoted by electron beam radiation, as a result of its relatively long hydrocarbon chains. A higher proportion of crosslinked polymer means that polyamide-containing graft polymers are less easily extracted by the hexane solvent.

It should be noted that the validity of these experiments to establish the presence of graft or block copolymer in blends of polyamide and butyl rubbers by solvent extraction is dependent on whether the homopolymeric component of polyamide is effectively excluded from the soluble portion of the blend. To establish this, an attempt was undertaken to dissolve a sample of polyamide homopolymer under extensive agitation in hexane. After a period of time, the liquid portion was either centrifuged or filtered through a $0.2\text{-}\mu\text{m}$ filter before preparation of the FTIR disk. No evidence of polyamide was seen in the solute after such treatments. Thus, the small peaks attributed to polyamide functional groups in the soluble portions of the blends are most likely the result of polyamide polymer that has chemically reacted with the butyl rubber matrix during melt processing.

SEM micrographs of blends

Figures 5 and 6 show the SEM images of bromobutyl/polyamide and butyl/polyamide blends, respectively.

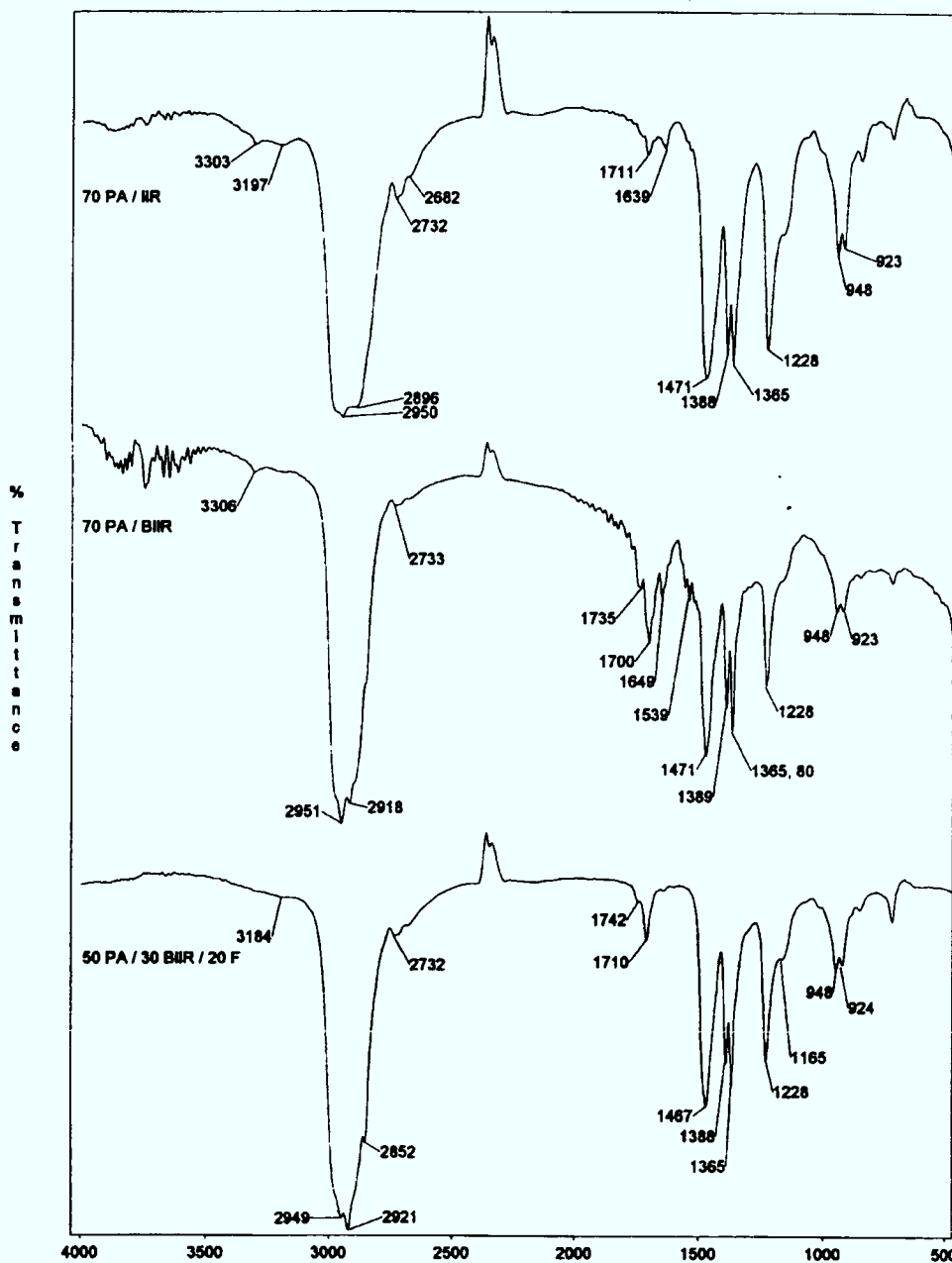


Figure 2 FTIR spectra of hexane-soluble portions of polyamide/butyl and bromobutyl blends containing high proportions of polyamide.

Blends of bromobutyl and polyamide with 60% rubber and 40% polyamide show the polyamide discrete phase to be about 10 μm , whereas comparable blends with butyl and polyamide have a discrete polyamide phase that varies from 10 to 50 μm . Thus, blends with bromobutyl rubber have a greater degree of compatibility, as evidenced by the size of individual phases in the blend. Greater compatibility is attributed to a

higher level of graft or block polymer in the blend after mixing.

Mechanical properties of blends

Typical mechanical properties values for PA/BIIR, PA/IR, and PA/BIIR/F blends are listed in Table IV, showing results for film, injection-molded, and com-

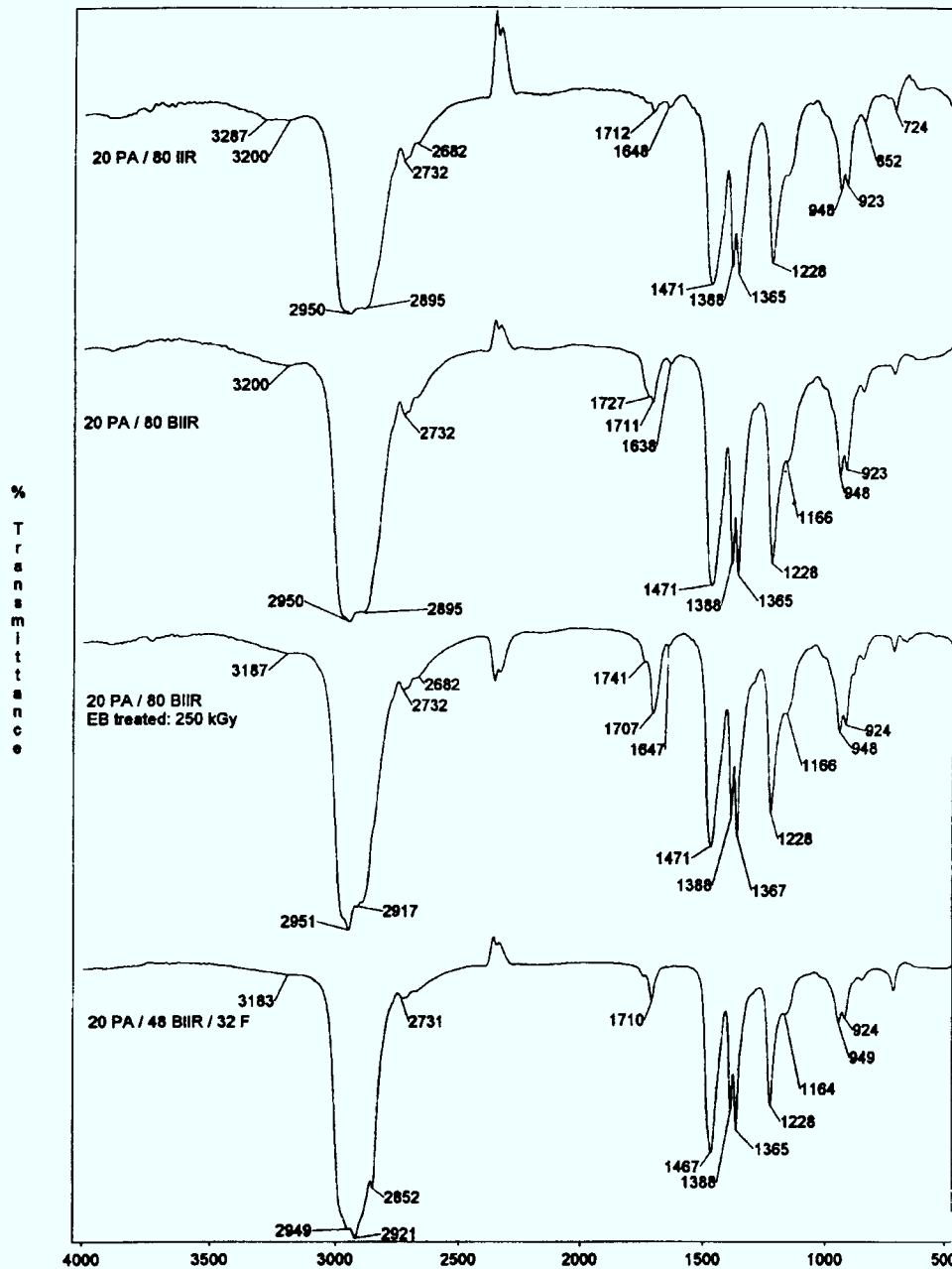


Figure 3 FTIR spectra of hexane-soluble portions of polyamide/butyl and bromobutyl blends containing high proportions of rubber.

pression-molded samples. Each set of properties reported is the result of five test specimens.

Although the tensile strength values of PA/BIIR blends are similar to those of PA/IIR blends, the elongation at break values for these blends are higher than those for IIR-containing blends, and exceed experimental error in each comparable case. Increases in this property for blends of similar composition are often associated with a higher degree of compatibility,²⁰

thus suggesting higher compatibility in BIIR-based blends as a result of higher reactivity of BIIR. When Fusabond is added to these blends, there is also an indication of increased elongation at break, given that maleic anhydride added to the rubber phase may enhance the coupling to the polyamide phase.

The ratio of PA to IIR or BIIR has the greatest effect on the overall properties of these blends. In blends containing more than 50% polyamide, the materials

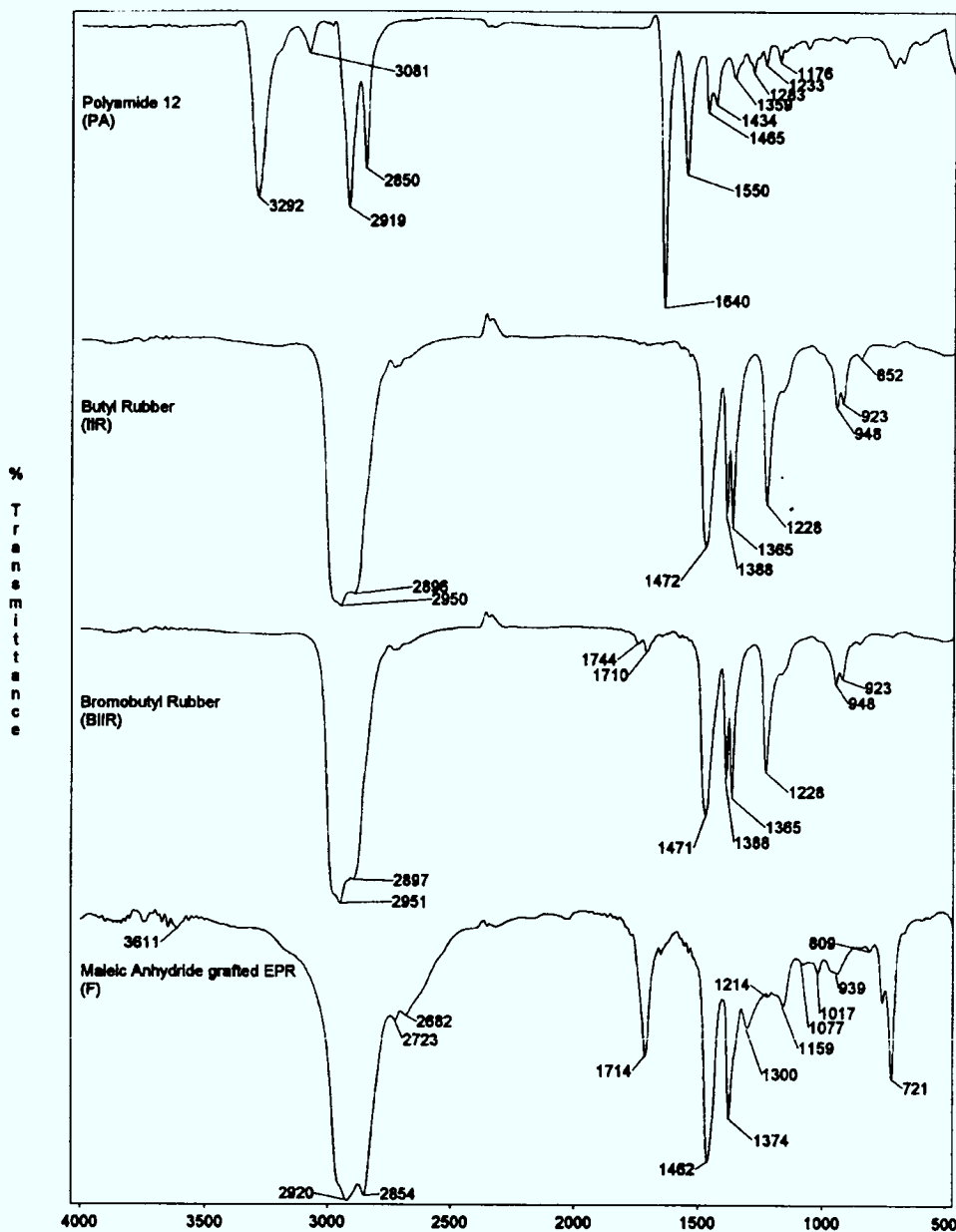
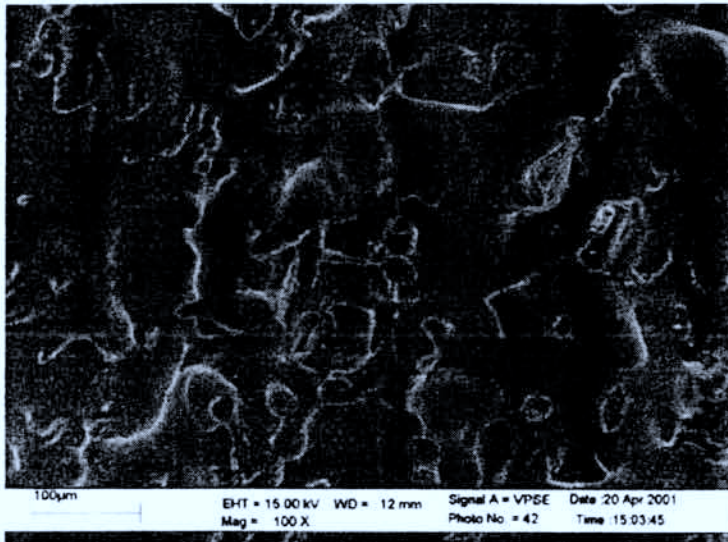


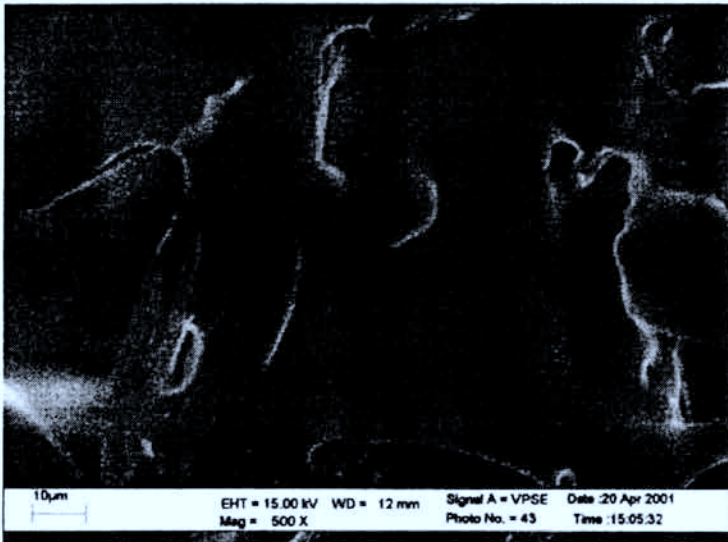
Figure 4 FTIR spectra of the homopolymeric components of blends for comparison to Figures 2 and 3.

have relatively high tensile strengths, and resemble the plastic properties of the polyamide component. In blends containing less than 50% polyamide, the materials have lower tensile strengths, characteristic of rubbery compositions. Injection- and compression-molded samples are compared to illustrate the differences in material properties produced by two different methods of sample preparation. The most probable cause for the poor match between the compression- and injection-molded samples of 20PA/50BIIR/30F is orientation, introduced into the samples during injection molding.

Graft or block copolymers that seem to be formed in the blending process improve compatibilization between the polyamide and butyl phases, which in turn allows blends of the two polymers to have mechanical properties intermediate between those of the two component homopolymers. This provides a range of uses for blends of these polymers as thermoplastic elastomeric materials, ranging from films in compositions containing higher levels of polyamide to rubbery materials at lower levels of this component. Although bromobutyl-containing blends appear to be the most



**40PA/60IIR
Mag. 100x**

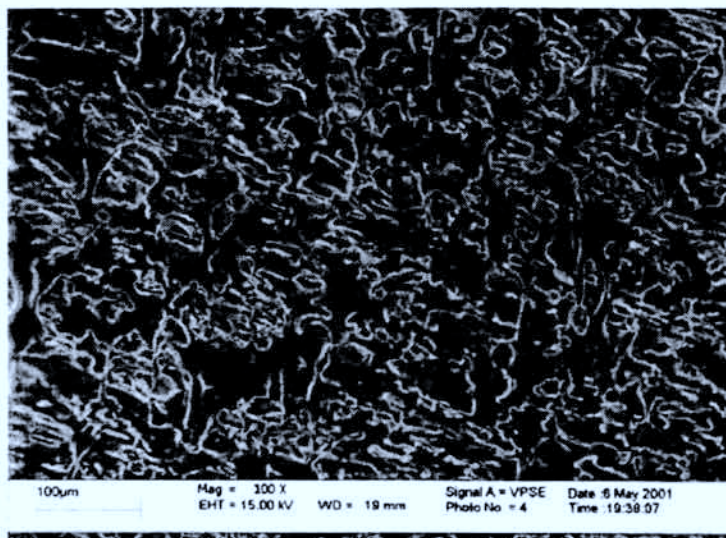


**40PA/60IIR
Mag. 500x**



**40PA/60IIR
Mag. 1000x**

Figure 5 SEM micrographs at different magnifications of 40/60 polyamide 12/butyl blends.



**40PA/60BIIR
Mag. 100x**



**40PA/60BIIR
Mag. 500x**



**40PA/60BIIR
Mag. 1000x**

Figure 6 SEM micrographs at different magnifications of 40/60 polyamide 12/bromobutyl blends.

TABLE IV
Properties of Blends Containing Polyamide 12 and Butyl or Bromobutyl Rubber^{a,b}

Sample	Ultimate strength (MPa) ^c	Modulus (MPa) ^d	Elongation at break (%) ^e	Hardness Shore A/D
Samples prepared as films				
100PA	100	952	376	
70PA/30IIR	69		139	
70PA/30BIIR	62	829	244	
70PA/18BIIR/12F	38	475	325	
Samples prepared as injection-molded specimens				
40 PA/60 IIR	10.0		81	41D
40 PA/60 BIIR	10.7		99	45D
30 PA/70 IIR	6.4		100	32D
30 PA/70 BIIR	7.0		122	34D
20PA/50BIIR/30F	4.4		219	77A
Samples prepared as compression-molded specimens				
20 PA/80 BIIR	0.57		377	54A
20 PA/50BIIR/30F	1.2		808	58A

^a All properties measured at room temperature.

^b PA, polyamide 12; IIR, butyl rubber; BIIR, bromobutyl rubber; F, Fusabond MF-416 (maleic anhydride grafted copolymer of EPR).

^c Standard error = ± 9 .

^d Standard error = ± 5 .

^e Standard error = ± 12 .

compatible, it has been shown that even in butyl-containing blends, the resulting material shows a level of compatibility. In blends where polyamide is the continuous phase, the rubber component is an effective plasticizer, and this is where butyl-containing blends have the greatest value.

Maleic anhydride grafted ethylene-propylene (EPR) polymers have been used in some blends, and they provide modest improvement in the mechanical properties of blends containing a low proportion of polyamide, with rubber as a continuous phase.

CONCLUSIONS

1. During blending a reaction appears to occur between polyamide 12 and butyl rubbers as a result of the *in situ* formation of compatibilizing agents, with bromobutyl rubber more reactive than butyl rubber.
2. Evidence of such a reaction, particularly in polyamide/bromobutyl blends, is observed by viscosity changes during processing, an increase in % insolubles in these blends, FTIR evidence of amide peaks in the butyl and bromobutyl portion, the relatively small domain size in SEM images of these blends, and intermediate mechanical properties in comparison to those of the component homopolymers.
3. Melt viscosity increases during heating of PA/BIIR blends, as a result of a chemical reaction taking place between the two components, particularly giving rise to increased crosslinking, and likely some grafting or block formation. This reaction is not nearly as evident in IIR-containing blends.
4. Solvent extraction of blends reveals an insoluble fraction that exceeds the proportion of rubber in the blend. This is attributed to crosslinking in the rubber phase, entrapment of the polyamide component, and possible grafting or block formation between the phases. Analysis of the soluble portion of these blends by FTIR and microanalysis indicates the presence of polyamide in the butyl rubber portion, which likely occurs by grafting or block formation. Compositions containing bromobutyl rubber show greater evidence of polyamide in the rubber portion than for butyl rubber. Evidence of crosslinking in the bromobutyl phase indicates that changes in the melt viscosity are most likely the result of crosslinking rather than of coalescence.
5. SEM images of blends of both bromobutyl and butyl rubbers with polyamide 12 show relatively high compatibility, with domain size in the range of 10 μm for bromobutyl and 10–50 μm for butyl rubber. The degree of compatibility is thus greater in the bromobutyl system.
6. The mechanical properties of a blend are affected by the proportion of rubber in the blend. Bromobutyl-containing blends have higher % elongation values than butyl-containing blends, indicating that bromobutyl is more compatible with polyamide.
7. Electron beam radiation, available as a post-blending cure process on molded samples, leads to increased crosslinking in the blends, with

some degradation in the polyamide component, as shown by DSC and reduced solubility in hexane. The ultimate strength of blends containing a butyl or bromobutyl rubber component is generally increased with exposure to the electron beam, whereas the elongation at break is relatively unaffected.

8. Maleic anhydride grafted ethylene-propylene copolymers enhance the compatibility of blends containing a low proportion of polyamide, as shown by improved tensile strength and elongation at break.

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