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THERMAL AND MECHANICAL CHARACTERIZATION OF PP/NBR BLENDS

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ABSTRACT

Thermal and mechanical behavior of i-PP and NBR blends were studied, with special reference to the effect of dynamic vulcanization and compatibilization. DSC and TGA characterization techniques were employed. Tensile properties and impact strength were also evaluated. Results indicate that NBR content decreased PP's crystallinity degree, increased thermal stability and slightly increased impact strength. Dynamic vulcanization did not show significant changes on mechanical or thermal properties. The addition of a compatibilizer seemed to lower interfacial tension of the dynamically vulcanized blend, although this decrease was not enough to increase tensile properties.
Keywords: PP, NBR, thermal properties, mechanical properties, dynamic vulcanization, compatibilizer.

CARACTERIZACIÓN TÉRMICA Y MECÁNICA DE MEZCLAS DE PP/NBR

RESUMEN

Se estudió el comportamiento térmico y mecánico de mezclas de Polipropileno (i-PP) y caucho nitrilo (NBR), haciendo énfasis en el efecto de la vulcanización dinámica y la presencia de un compatibilizante. Se emplearon las técnicas de caracterización por DSC y TGA. Las propiedades de tensión y de resistencia al impacto también fueron evaluadas. Los resultados obtenidos indican que el contenido de NBR disminuye la cristalinidad del PP, incrementa la estabilidad térmica y aumenta ligeramente la resistencia al impacto. La vulcanización dinámica no arrojó cambios significativos en las propiedades mecánicas o térmicas. Finalmente, el uso de un agente compatibilizante pareciera disminuir la tensión interfacial de la mezcla vulcanizada dinámicamente, a pesar de que esta disminución no fue suficiente para mejorar las propiedades de tensión.
Palabras claves: PP, NBR, propiedades térmicas, propiedades mecánicas, vulcanización dinámica, compatibilizante.

INTRODUCTION

The use of elastomer-thermoplastic blends has become increasingly important because the resulting systems have many of the properties of elastomers and can be processed like thermoplastics. The method called dynamic vulcanization, where the elastomer vulcanizes during its melt mixing with the molten plastic, is the best way to produce these types of compounds (George *et al.*, 1999, 2000; Jain *et al.*, 2000). Examples of these blends are those comprising PP and NBR, where the excellent processing characteristics and tensile properties of PP are combined with the oil resistance and flexibility of NBR. However, these blends are incompatible and require compatibilization for better properties (Coran *et al.*, 1983). In order to overcome the gross mutual incompatibility of olefin polymers and nitrile rubber such that compounds having improved ultimate properties can be obtained, the addition to the blend of a compatibilizing agent comprising a block copolymer having an olefin polymer compatibilizing segment chemically linked to a nitrile rubber-compatibilizing segment is proposed by Coran *et al.* (1983). These compatibilizers are found to modify the morphology of the blends and also to improve the viscoelastic, thermal and mechanical properties (George *et al.*, 1995, 1996, 2000).

Previously, the authors of this investigation studied the rheological behavior of PP/NBR blends obtaining that the addition of up to 20% of rubber did not affect significantly the rheological properties of these blends (Hernández *et al.*, 2003). In spite of these results, in this paper we have studied the thermal and mechanical properties of PP/NBR blends and the PP/NBR (70/30) compatibilized and dynamically vulcanized.

EXPERIMENTAL

Propilven S.A supplied Isotactic Polypropylene (PP) J-600 with a melt flow index (MFI) of 7 g/10 min (230 °C, 2.16 Kg). Acrylonitrile-butadiene rubber (NBR) Arnipol BJLT having an acrylonitrile content of 30.5-34.5 % was obtained from Industrias PASA S.A. Maleic anhydride modified PP (MA-PP) Polybond 3150 supplied by Uniroyal Chemical was used as a compatibilizing agent.

NBR particles obtained from the bale were introduced in liquid nitrogen and immediately fed into a cutting mill in order to reduce its size. The unvulcanized PP/NBR blends (PP_0 (without NBR), PP_{10} (with 10 wt% NBR), PP_{20} (with 20 wt% NBR), PP_{30} (with 30 wt% NBR)) were prepared by melt mixing using a Werner and Pfleiderer ZSK-30 twin-screw extruder at a temperature of 210 °C and a screw speed of 60 rpm. Dynamically vulcanized blend ($PP_{30\text{-}v}$) with a composition of 30 wt% NBR was prepared from the formulation shown in Table 1. All compound ingredients were physically blended and then fed into the twin-screw extruder for mixing and vulcanizing at the same conditions as the unvulcanized mixture. Rheometric curves were obtained using a Zwick oscillating disk rheometer at 210 °C, oscillation arc of 5° and torque range of 0.5 Kp.m. The 90% cure time value was set as the minimum residence time needed in the extruder so the dynamic vulcanization reaction could take place. Both 70/30 PP/NBR blends, unvulcanized and dynamically vulcanized, were compatibilized with 5 wt% MA-PP. These blends were denoted as $PP_{30\text{-}c}$ and $PP_{30\text{-}v,c}$, respectively. It should be mentioned that the composition with 30 wt% NBR was selected since it showed the better thermal stability. According to literature (George *et al.*, 1995, 1996, 2000) at higher NBR contents there seems to start a coalescence process, considerably decreasing the interfacial area and hence the interaction between phases.

Thermal behavior was studied using a Mettler Toledo DSC821 thermal analyzer, performed under nitrogen with a heating rate of 10 °C min^{-1}. Samples were then cooled to room temperature at a constant cooling rate of 10 °C min^{-1} with identical settings of the instrument for all the systems studied. Samples were reheated to measure the crystallinity degree. Crystallinity degree of PP in the blends was evaluated from the second melt by comparing the enthalpy change of the PP content in the blend to that of fully crystalline PP (ΔH_{PP} = 209 J/g was used for calculations). Thermogravimetric analysis was carried out in a Mettler Toledo TGA/ST DA851 equipment. Samples were scanned from 30 to 600 °C at a heating rate of 5 °C min^{-1} in a nitrogen atmosphere. Tensile properties were measured with an Instron Machine model 1125. Dumb-bell test specimens were injection molded at 200 °C. Tests were done using a stretching rate of 50 mm min^{-1} according to ASTM D638 procedure. Impact strength was analyzed using a Zwick Impactometer according to ASTM D256 procedure. Morphology of the samples was analyzed using a scanning electron microscope Hitachi S-24000. Specimens were obtained from the extruded blends, cryogenically fractured in liquid nitrogen and then coated with platinum/palladium. An optical microscope Zeiss was also used for morphology studies. In this case, samples were heated to 200 °C, kept at that temperature for 5 minutes and then cooled to 135°C following isothermic conditions. Gel content of dynamically vulcanized blends compatibilized and non-compatibilized was determined, wherein about 1.0 g of sample was submitted to continuous extraction in boiling o-xylene for 7 days at 140 °C.

Table 1. Formulation of dynamically vulcanized PP/NBR blends.

Ingredients	phr
NBR	100
ZnO	5
Stearic acid	2
CBS (1)	2
TMTD (2)	2.5
Sulphur	0.2

(1) n-cyclohexyl benzothiazyl sulfenamide

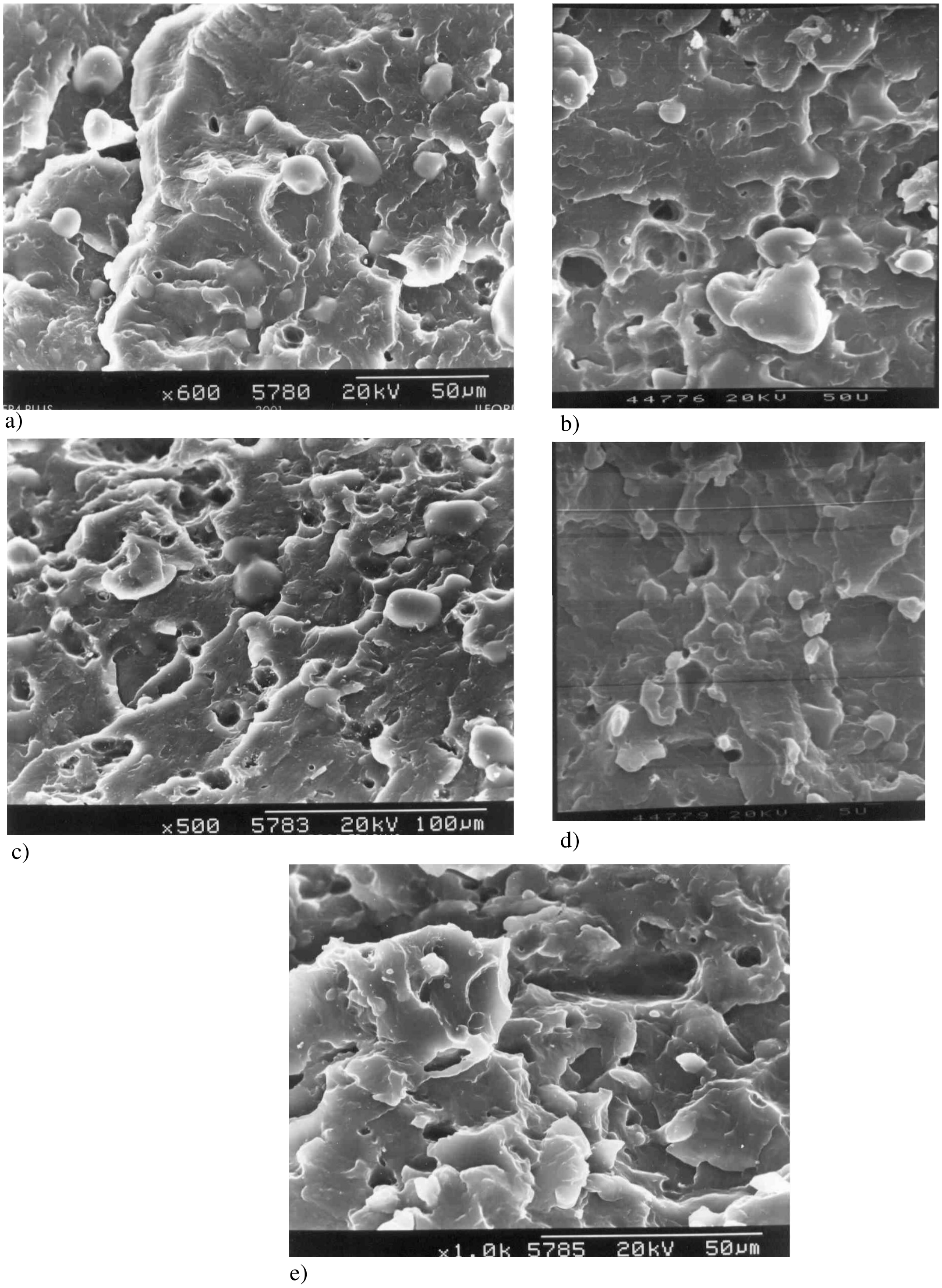
(2) Tetra methyl thiuram disulfide

RESULTS AND DISCUSSION

Thermal Properties

In polymer blends with a crystallisable component, the final properties are determined by: 1) mode and state of dispersion of rubbery domains in the crystalline matrix, 2) texture, dimensions and size distribution of spherulites of the matrix, 3) inner structure of spherulites, 4) physical structure of inter spherulitic boundary regions and amorphous inter lamellar regions, and 5) adhesion between the rubbery domains and the crystalline matrix (George *et al.*, 2000).

Thermal behavior of blends under investigation is affected by the size of the dispersed phase. As it is shown in Figure 1 and in Table 2, there is an increase on the average particle



a) b) c) d) e)

Figure 1. SEM micrographs of PP/NBR blends: a) PP_{20}; b) PP_{30}; c) $PP_{30\text{-c}}$; d) $PP_{30\text{-v}}$; e) $PP_{30\text{-v, c.}}$

size when NBR content increases. Different particle sizes are also present, obtaining particles as big as 50 µm with 30 wt% NBR. When the blend is compatibilized ($PP_{30\text{-c}}$) there is a decrease in the average particle size if compared to the non-compatibilized peer (PP_{30}). This effect is even more notorious when the blend is vulcanized ($PP_{30\text{-v}}$) and compatibilized ($PP_{30\text{-v, c}}$). This drastic reduction in particle size presumably affects the thermal behavior of the blends under study.

Melting behavior of PP/NBR blends was analyzed using DSC. It is known that unmodified PP has a regular structure with a spherulitic arrangement relatively ordered. Figure 2a) shows the photomicrographs of pure PP. However, the addition of a rubbery component or an impact modifier decreases this regularity. Not only the spherulitic structure of PP, but also their size is highly altered with 10% of a rubber phase incorporation (Figure 2b)) and even more changed for contents of up to 20% rubber (Figure 2c)).

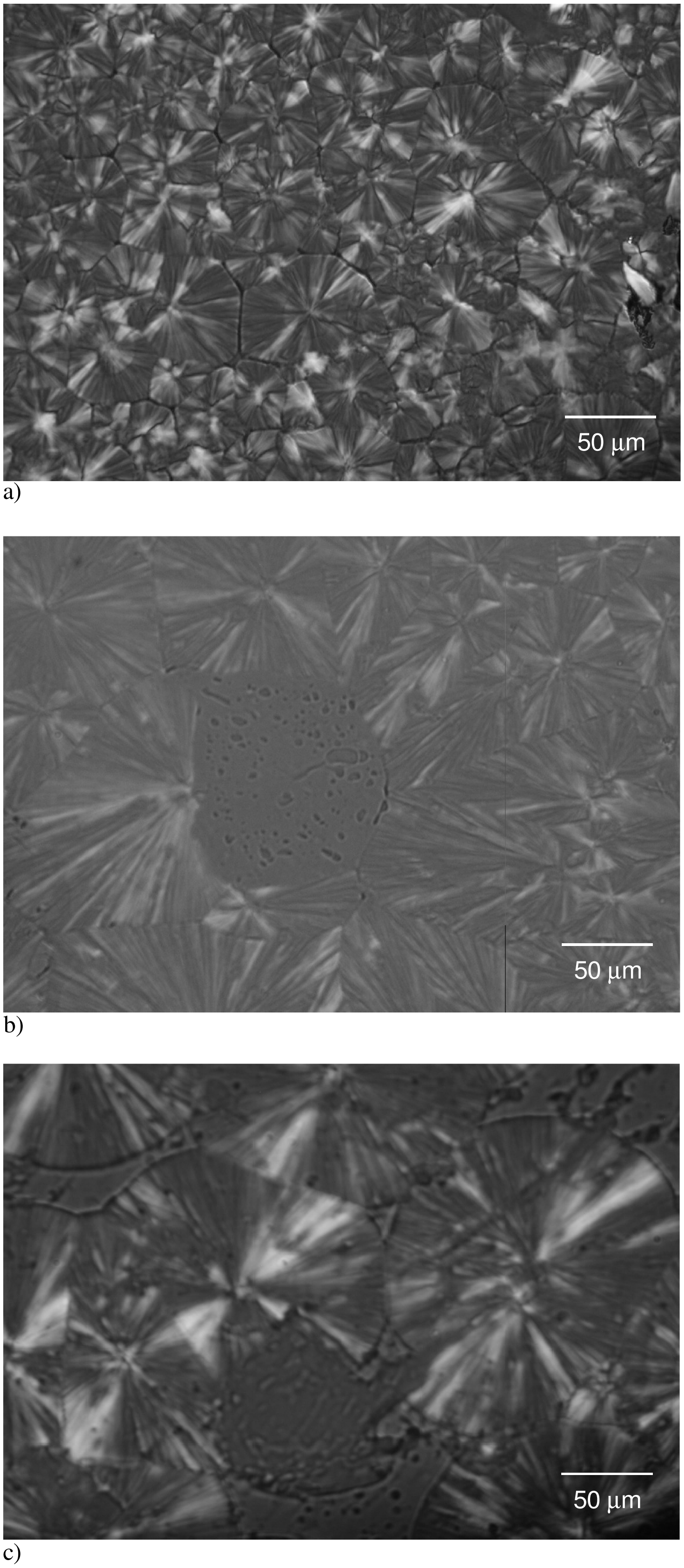


Figure 2. Photomicrographs: a) pure PP; b) PP_{10}; c)PP_{20}.

Table 2. Particle size of PP/NBR blends.

Blend	Average particle size (µm)	Variation range (µm)
PP$_{20}$	8.6	7.14 - 17.14
PP$_{30}$	16.09	6.25 - 50.00
PP$_{30\text{-c}}$	11.74	6.66 - 23.33
PP$_{30\text{-v}}$	5.17	3.13 - 7.50
PP$_{30\text{c,v}}$	5.23	1.66 - 6.66

The incorporation of the elastomer alters the superstructure of PP matrix changing the average size and number of spherulites and this change in the superstructure is very important to interpret the function of impact modification of the elastomer with the PP matrix.

Crystallization exotherms of each blend were found during the cooling cycle executed by the DSC equipment. Blend characterization was carried out by the exotherm peak temperature (crystallization temperature T_c). Table 3 shows T_c values for each of the PP/NBR blends studied. The observed decrease on T_c with the addition of NBR is due to the fact that the system is affected by the presence of NBR, which hinders the growth of PP´s crystals. An increase on T_c can be seen for the blends when the rubbery phase is vulcanized (PP$_{30\text{-v}}$ and PP$_{30\text{-v,c}}$). Previous work concerning blend morphology (Hernández *et al.*, 2001) indicates that rubber particle size decreases when it was dynamically vulcanized. When the dispersed phase is reduced to very small particle sizes, it could be acting like a nucleation agent. The same behavior, but less pronounced, is observed for the compatibilized blend (PP$_{30\text{-c}}$). When the compatibilizer is added to the blend, it acts by locating itself at the interface; thereby, reducing the interfacial tension between the phases and permitting a finer dispersion during mixing (Figure 1).

Also, in compatible blends (PP$_{30\text{-c}}$, PP$_{30\text{-v,c}}$), the increase on T_c can also be related to the extent of interaction between the components (George *et al.*, 2000). Moreover, an increase of 2-4 °C in crystallization temperature for the compatibilized and vulcanized blends is highly profitable from a processing point of view, especially when the material is injection molded, since the overall cooling cycle can diminish considerably.

Melting temperature (T_m), melting enthalpy (ΔH_m) and crystallinity degree (X_c) values obtained from thermograms are also shown in Table 3. The melting point does not vary with the addition of NBR to PP. A decrease of about 20% on melting enthalpy (ΔH_m) is adverted when incorporating the rubbery phase (30%) to pure PP (George *et al.*, 2000). Kumar *et al.* (1996) reported similar behavior for Nylon/NBR blends. In the case of incompatible blends, the crystallinity slightly decreases since the non crystalline component retards the crystal growth which leads to imperfect crystals (Stolp *et al.*, 1996).

With respect to compatibilized and/or vulcanized blends, an increase in melting enthalpy is adverted, which implies a rise on PP's crystallinity degree. This could be due to the drastic decrease on the size of the dispersed phase, which could be acting as a nucleation agent. This effect is more pronounced for PP$_{30\text{-v}}$ and PP$_{30\text{-v,c}}$ blends. Similar results were found by Chang *et al.* (1986) for PP/EPDM blends.

Major changes are observed when studying the crystallinity degree. A decrease in X_c can be adverted in all cases when incorporating rubber to the PP matrix. Several researchers indicate that this decrease could be a consequence of an increase in lamellar thickness when rubber content is higher. This characteristic, widely reported for PP/EPDM and PP/NBR blends (George *et al.*, 1996; Jang *et al.*, 1985; Choudhary *et al.*, 1991) is due to the fact that the presence of rubber particles in the thermoplastic matrix hinders the spherulitic growth in the zones near each rubber particle.

According to Martuscelli *et al.* (1982) and Bartezak *et al.* (1984), the rubber particles are present in inter and intra spherulitic regions of the crystalline plastic phase. WAXS studies carried out by George *et al.* (2000) with PP/NBR blends confirm this assumption, which also implies a decrease of ΔH_m values and crystallinity, due to the fact that the formation of crystallites in the blend was affected by the presence of Nitrile rubber.

Table 3. Thermal Properties
of pure PP (PP$_0$) and PP/NBR blends.

Blend	T_c (ºC)	T_m (ºC)	ΔH_m (J/g)	X_c (%)	T_{id} (ºC)	E_a (kJ/mol)
PP$_0$	119	163	121	58	330	164
PP$_{10}$	112	162	108	51	360	196
PP$_{20}$	112	162	105	50	374	199
PP$_{30}$	111	162	92	44	399	207
PP$_{30\text{-c}}$	113	162	101	48	418	228
PP$_{30\text{-v}}$	114	162	109	52	423	230
PP$_{30\text{-v,c}}$	115	162	117	55	432	245

Thermal stability of blends was determined using TGA technique. Table 3 shows the initial decomposition temperature (T_{id}) obtained from the corresponding thermograms and activation energy (E_a) calculated according to Dharwadkar & Karkhanavala (1969) (D-K) method of all PP/NBR blends studied.

Initial decomposition temperatures of PP and NBR are 330 ºC and 480 ºC respectively, so unvulcanized PP/NBR blends show intermediate values of the T_{id}.

The incorporation of NBR into PP was found to increase the thermal stability of PP; an increase of approximately 70 °C on T_{id} is adverted when PP is mixed with a rubbery component as NBR. In the case of polymer blends, thermal degradation depends on morphology (size of dispersed phase) and extent of interaction between the phases. This increase in initial degradation may arise from the interaction of radicals formed during degradation of PP with NBR. George *et al.* (2000) obtained a similar behavior for 70/30 PP/NBR blends due to the presence of a co-continuous morphology. Also, from the data shown in Table 3, it can be seen that the initial decomposition temperature is shifted to higher temperatures upon vulcanization. Vulcanization of rubbers generally enhances the initial degradation temperature (399 vs. 423 ºC) since more energy (207 vs. 230 kJ/mol) is required to break the bonds formed during crosslinking. In addition, it should be taken into account that the curing system employed in this research corresponds to an accelerated type with very low content of sulphur, where the final vulcanizates exhibit low modulus, very low elongation at break, but high resistance at elevated temperatures (Kempermann, 1986); thus a higher T_{id} should be expected.

Concerning the action of compatibilization, an increase of T_{id} is adverted. This improvement in degradation temperature may arise from the better interaction between PP and NBR. Moreover, the effect is more pronounced for the vulcanized and compatibilized blends ($PP_{30\text{-}v,c}$), where the size of the dispersed NBR domains decreased with the addition of maleic anhydride modified PP (Figure 1e).

The activation energy for the homopolymer and blends is also present in Table 3. The tendencies obtained are in accordance with decomposition temperatures. Among all results, PP shows the lowest activation energy, since this polymer is more susceptible to degradation than NBR upon increasing temperature. It can be noticed that the addition of NBR whether unvulcanized, dynamically vulcanized or compatibilized increases this kinetic parameter, due to the presence of slight interactions in the interface between PP and NBR phases.

If the effect of the compatibilizer is studied separately, one can see that the slight interaction in the interface due to the presence of a compatibilizer increases the activation energy for the compatibilized blends ($PP_{30\text{-}c}$ and $PP_{30\text{-}v,c}$), when compared to PP_{30}.

Finally, concerning the effect of vulcanization, the behavior obtained is in accordance with the behavior of the initial decomposition temperatures, where a vulcanized system is more stable than the corresponding unvulcanized one.

MECHANICAL PROPERTIES

In this paper, different factors affecting mechanical properties of PP/NBR blends were investigated by the stress-strain behavior determination of each blend and by impact strength calculation. Table 4 shows mechanical properties values for the PP/NBR unvulcanized blends and for pure PP. It can be seen that with the addition of NBR, Young's Modulus decreases. The tensile strength of PP/NBR blends depends on the strength of the PP phase, which in turns depends on the extent of crystallinity. As it was discussed in the previous section, the crystallinity degree of PP was decreased by the incorporation of NBR (Table 3). Hence, the observed decrease is due to the presence of the soft rubber phase and fall in crystallinity of the PP phase (George *et al.*, 1995).

Table 4. Mechanical properties of pure PP (PP_0) and PP/NBR blends.

Blend	Property			
	Young's Modulus (MPa)	Tensile strength (MPa)	Elongation at break (%)	Impact strength (J/m)
PP_0	1087 ± 14	18.4 ± 0.9	192.9 ± 44.3	20 ± 3
PP_{10}	973 ± 39	22.9 ± 0.9	17.5 ± 3.4	25 ± 1
PP_{20}	826 ± 33	19.4 ± 0.8	22.8 ± 3.9	26 ± 2
PP_{30}	684 ± 9	17.1 ± 0.2	24.5 ± 2.0	23 ± 2
$PP_{30\text{-}c}$	671 ± 11	16.5 ± 0.7	25.5 ± 3.9	20 ± 1
$PP_{30\text{-}v}$	672 ± 12	18.6 ± 0.5	21.6 ± 5.6	22 ± 3
$PP_{30\text{-}v,c}$	764 ± 19	18.0 ± 0.1	19.5 ± 2.4	19 ± 1

Table 4 also compares values for the 70/30 PP/NBR unvulcanized with dynamically vulcanized ($PP_{30\text{-}v}$) blends. Results obtained show no significant changes on mechanical properties, although a higher Young's Modulus for the dynamically vulcanized blend should be expected. However, the relatively low gel content obtained (14%) and the vulcanization system employed confirm the results presented.

Concerning elongation at break, the low values for all blends with respect to pure PP can be explained on the basis of the poor adhesion between the two phases. This poor interfacial adhesion causes premature failure as a result of a crack opening mechanism (Kumar *et al*., 1996). As the rubber concentration increases, the interfacial area increases, then, the probability to initiate the cracks increases (Cai *et al*., 1993). Further, the decrease in elongation for cured PP/NBR blends may be due to the compatibility of the two components being sacrificed by the vulcanization of the rubbery phase. The interface between the two phases readily becomes the place where cracks are initiated. As previously noted, the vulcanization process reduces even more this value, since the crosslinking system employed in this study (2.5 phr TMTD, 0.5 phr CBS and 0.2 S) corresponds to a typical formulation for compounds with very low elongation at break (Kempermann, 1986).

In addition, Table 4 shows the influence of a compatibilizer on the mechanical properties of the unvulcanized and dynamically vulcanized PP/NBR compounds. Different behaviors can be observed. While the compatibilizer showed no significant effect on the unvulcanized blend ($PP_{30\text{-c}}$), there is a rise on elasticity Modulus when MA-PP is present in the vulcanized blends. In the case of compatibilized PP/NBR blends, the compatibilizing action of MA-PP is due to the dipolar interaction between the maleic anhydride group of MA-PP and NBR. This causes a reduction in interfacial tension, which reduces the domain size of the dispersed phase. As a result, there is an effective stress transfer between the dispersed phase and the continuous phase and an increase in interfacial adhesion. This contributes to the reduction in interlayer slippage and therefore an increase in viscosity, with the corresponding increase in Young's Modulus (Hernández *et al*., 2003; George *et al*., 1999).

The ideal function of a compatibilizer on the tensile properties of a plastic-rubber compound is to improve tensile strength and elongation at break. The compatibilizer, when increasing the interfacial adhesion, increases the material strength and decreases the rubber particles size avoiding its premature fracture; however, results displayed in Table 4 suggest that the effect of the compatibilizer was not completely effective. When analyzing the morphology of blends $PP_{30\text{-v}}$, $PP_{30\text{-c}}$ and $PP_{30\text{-c,v}}$, (Figures 1c), d) e)), a slight reduction in rubber particle size occurs when compatibilizer is added to the unvulcanized blend, while the particle size decreases significantly for the vulcanized blend, meaning that the compatibilizer increased the interfacial interaction between PP and NBR phases and lowered the interfacial tension of the blend (Hernández *et al*., 2001; 2003). Nonetheless, results obtained for mechanical properties indicate that this decrease of interfacial tension was not enough to increase those properties. Similar results were obtained by Sereda *et al*. (1997).

Concerning impact strength, unvulcanized blends show a slight increase when compared to pure PP, except the blend with 30 wt% NBR, due to the bigger particle size of the rubber dispersed in the polymeric matrix. The energy absorbed on impact is the sum of the energy to fracture the glassy matrix and the work to break the rubber particles. Block and graft copolymers formed during the production of those blends tend to bridge the two phases together, thus contributing to improve the impact strength of the blends (Norzalia *et al*., 1994). However, when analyzing the effect of a compatibilizer on the impact strength of unvulcanized and dynamically vulcanized blends, a decrease on such property is found. This result suggests once again, that the compatibilizer, due to its characteristics, increases the stiffness of the system and affects the elastic properties of the NBR, thus reducing its ability to improve PP´s impact strength. Similar behavior was obtained by George *et al*. (1995) when 15% Ph-PP was used in PP/NBR blends.

CONCLUSIONS

Mechanical and thermal behavior of isotactic polypropylene (PP) and nitrile rubber (NBR) blends has been investigated. Concerning thermal behavior, there occurred a slight increase in PP's crystallization temperature when in blends vulcanized and/or compatibilized ($PP_{30\text{-c}}$, $PP_{30\text{-v,c}}$) when compared with PP_{30}. With respect to thermal stability, it increased with the addition of NBR and even more with vulcanization and compatibilization. However, crystallinity degree of PP decreased with the addition of NBR, even though it increased slightly with the vulcanization and with the presence of a compatibilizer. Young's Modulus and elongation at break diminished when introducing rubber into the PP matrix. Impact strength of unvulcanized and dynamically vulcanized blends showed a limited increase when compared to pure PP. The addition of a compatibilizer lowered the interfacial tension of the dynamically vulcanized blend reducing particle size, although this decrease was not enough to increase tensile properties (tensile strength and elongation at break).

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