

# Mercapto-Modified Copolymers in Polymer Blends.

## II. The Compatibilization of NBR/EVA Blends

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**ABSTRACT:** Ethylene-vinyl acetate (EVA) copolymer functionalized with mercapto groups (EVALSH) has been used as compatibilizing agent in nitrile rubber/EVA blends. The tensile strength and elongation at break of the system were measured as a function of the EVALSH content and blend composition. The compatibilization affects the mechanical properties of these blends. The highest improvement of the tensile strength has been achieved in the composition range corresponding to the co-continuous phase morphology. The co-continuity of these blends has been studied by both dissolution studies and scanning electron microscopy. The addition of EVALSH as an interfacial modifier did not change the region of co-continuity but influences the percolation threshold for both dispersed nitrile rubber phase and dispersed EVA phase. From optical microscopy and differential scanning calorimetry analysis, it is possible to assume that the functionalized EVALSH copolymer affects the crystallization of the EVA phase. © 2000 John Wiley & Sons, Inc. *J Appl Polym Sci* 79: 193–202, 2001

**Key words:** elastomer blends; NBR; EVA; compatibilization; mercapto-modified copolymer

### INTRODUCTION

Blending of two or more polymers to produce new material systems with a combination of properties for specific uses has been extensively developed in several industries as a way to meet new market applications with minimum cost. The combination of thermoplastics with elastomers, for example, has given rise to a well known class of materials, the thermoplastic elastomers, which

can exhibit the good elastic properties of rubber and the processing ability of thermoplastics.<sup>1,2</sup> Several other important properties can be improved by using an appropriated blend composition. For example, outstanding impact performance is normally achieved by blending a small amount of rubber in a thermoplastic matrix of polystyrene,<sup>3</sup> nylon,<sup>4,5</sup> polyvinyl chloride,<sup>6</sup> polypropylene,<sup>7</sup> etc. The addition of a saturated thermoplastic such as polyethylene and ethylene-vinyl acetate (EVA) in an unsaturated elastomer matrix is also used to improve the aging resistance of the rubber.<sup>8–11</sup>

EVA copolymers are very interesting materials with excellent ozone and weather resistance, good toughness at low temperature, and good mechanical properties.<sup>12</sup> Specific other properties, such as crystallinity degree and flexibility, are also

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achieved by varying the amount of vinyl acetate in the copolymer. Several rubbers have been blended with EVA and include natural rubber,<sup>8,13</sup> polychloroprene rubber,<sup>14–17</sup> etc. Blends of nitrile rubber (NBR) with EVA also have been reported in literature.<sup>18,19</sup> Such blends can lead to an important class of materials with excellent oil resistance, abrasion resistance, and mechanical properties promoted by the NBR phase and excellent ozone and oxygen resistance because of the EVA phase. According to the morphological data reported in the literature,<sup>18</sup> these blends are immiscible. The immiscibility in polymer blends normally results in poor mechanical properties because of the phase separation and poor interfacial adhesion. This problem can be minimized by a proper control of phase morphology during processing and by the addition of compatibilizing agents.<sup>20</sup>

Recently, we have developed EVA modified with mercapto groups (EVALSH) to improve the interfacial adhesion between natural rubber and EVA.<sup>21–23</sup> The mercapto groups in the EVA backbone have reacted with the double bonds of the natural rubber giving rise to a strong anchorage between the phases. An improvement of mechanical properties and aging resistance was observed in blends with a high amount of natural rubber.<sup>10,21–23</sup>

The objective of this report is to study the influence of EVALSH on the compatibilization of NBR/EVA blends. The mechanical properties and morphological structure have been investigated as a function of the amount of EVALSH in the blend and the blend composition. Emphasis has been placed on the mechanical behavior at the composition corresponding to the co-continuous phase morphology.

## EXPERIMENTAL

### Materials

NBR (NBR615) (33 wt % of acrylonitrile; Mooney viscosity = 32) was kindly supplied by NITRIFLEX S.A., Rio de Janeiro, Brazil. EVA copolymer (18 wt % of vinyl acetate; MFI = 2.3 g/10 min at 120°C) was kindly supplied by Petroquímica Triunfo S.A., Rio Grande do Sul, Brazil. EVALSH was obtained in our laboratory by esterifying the hydrolyzed EVA copolymer with mercaptoacetic acid, according to the literature.<sup>24</sup> The amount of mercapto groups in the functionalized copolymer

corresponded to 62 mmol %, as determined by Fourier transform infrared spectroscopy and thermogravimetric analysis.<sup>24</sup>

### Blend Preparation and Characterization

The blends were prepared in a Berstoff two roll-mill at 110°C and 30 rpm. NBR was first masticated for 2 min, and then compounded with EVALSH and EVA in this order. The total mixing time was 6 min in all blends.

Tensile sheets of ca. 2-mm thickness were compression-molded in a hydraulic press at 160°C under 15-MPa pressure, using a residence time of 5 min. Dumbbell-shaped specimens (ASTM D638-77A) were punched out of the sheets and submitted to tensile testing with an Instron, model 4204 machine, at a cross head speed of 100 mm/min.

Differential scanning calorimetry (DSC) was performed in a Perkin-Elmer DSC7 equipment using a heating rate of 10°C/min and a cooling rate of 10°C/min, under nitrogen atmosphere.

### Morphological Studies

Molded samples were cryogenically fractured. The surface was immersed in methyl ethyl ketone (MEK) for about 24 h to preferentially extract the NBR phase. The samples were then dried in an air oven at 35°C for 24 h. The surface was covered with a thin layer of gold and analyzed in a JEOL 5300 scanning electronic microscope (SEM). Morphological studies were also performed with thin films of these blends using optical microscope Olympus BX50 at 100× magnification.

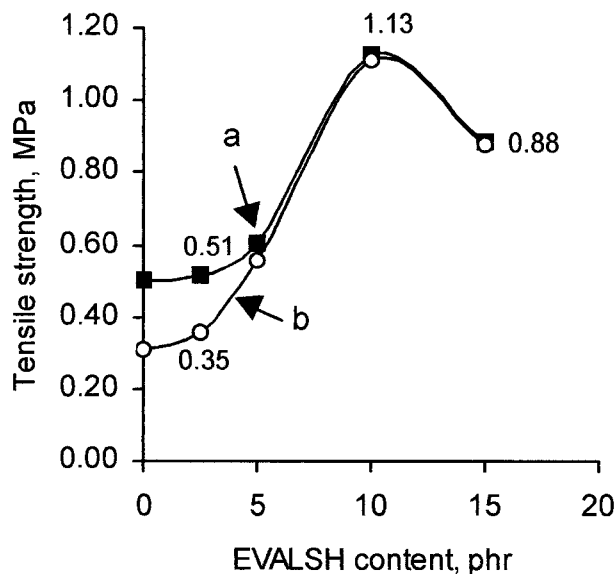
### Selective Extraction Experiments

The phase inversion composition was determined by submitting weighted specimens to selective extraction of NBR phase with MEK for 1 week, according to the procedure reported in the literature.<sup>25</sup> The solvent was changed every day. After the extraction, the greatest piece of each sample was withdrawn from the solvent, dried under vacuum, and weighed to determine the amount of nonextracted materials as a continuous phase.

## RESULTS AND DISCUSSION

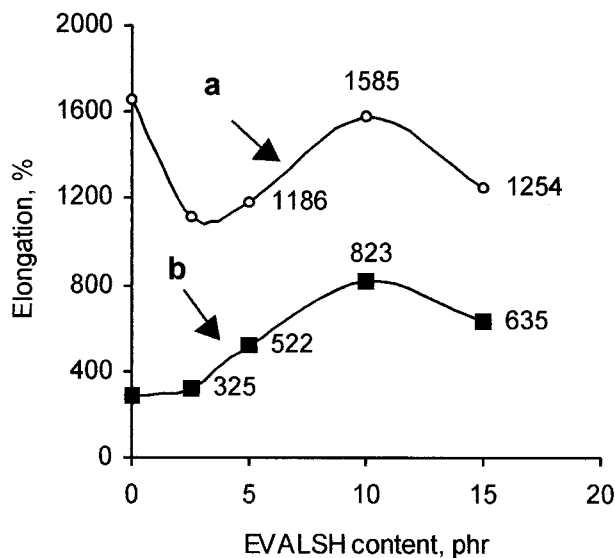
### Mechanical Properties

The effect of the EVALSH content on the tensile strength and elongation of NBR/EVA (80:20 wt %)

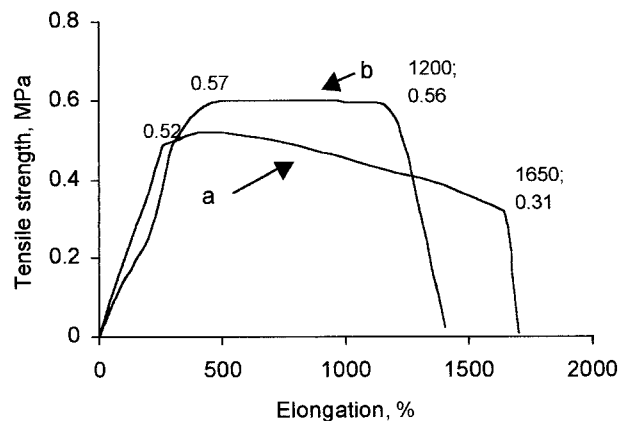


**Figure 1** Variation of tensile strength (a) at maximum load and (b) at break, for NBR/EVA blends (80:20 wt %) as a function of the EVALSH content.

blends is illustrated in Figures 1 and 2, respectively. The tensile values at yield point ( $\sigma_{\max}$ ) (yield stress) and at break ( $\sigma_B$ ) increase with the addition of the functionalized copolymer until 10 phr (part per hundred part of rubber) of EVALSH. Similar behavior was also observed for elongation at the yield point ( $\epsilon_{\max}$ ). Uncompatibilized NBR/EVA blend displays a value of  $\sigma_{\max}$  higher than



**Figure 2** Variation of elongation (a) at maximum load and (b) at break, for NBR/EVA blends (80:20 wt %) as a function of the EVALSH content.



**Figure 3** Stress-strain behavior of NBR/EVA blends (80:20 wt %) (a) without EVALSH, and (b) with 5 phr of EVALSH.

$\sigma_B$ . The stress-strain curves of compatibilized and uncompatibilized NBR/EVA blends are compared in Figure 3. In the case of uncompatibilized blend (curve a), the stress initially increases up to 325% elongation and then decreases until the failure occurs. This behavior is typical of uncrosslinked rubber systems and was also observed by Thomas et al.<sup>18,26,27</sup> in several elastomeric materials. It can be attributed to poor interfacial adhesion between the phases and absence of crosslinking in the system.

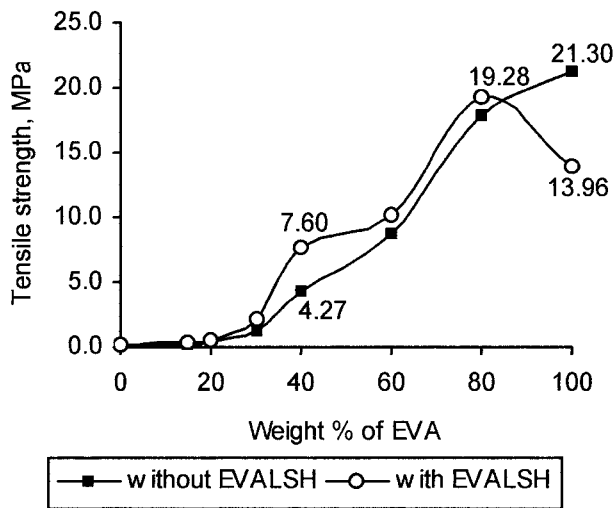
The presence of 5 phr of EVALSH in the NBR/EVA blend (Fig. 3, curve b) increases the tensile strength of the material. Indeed, both  $\sigma_{\max}$  and  $\sigma_B$  display similar values which are higher than uncompatibilized blend. This stress-strain curve indicates an improvement of the mechanical performance in compatibilized blend because of the interfacial adhesion between NBR and EVA phases, promoted by the mercapto-modified EVA. The compatibilizing effect increases with the amount of EVALSH until a maximum value of 10 phr. After this point, both tensile strength and elongation show a gradual decreasing, indicating a loss of the interfacial activity. Similar phenomenon also has been found in other compatibilized systems reported in literature,<sup>26,28</sup> which was attributed to the saturation of the interface.

The effect of EVALSH on the mechanical properties of NBR/EVA blends has been studied with different blend compositions and the results are summarized in Table I. An increasing of both tensile strength and elongation can be observed as the weight percentage of EVA increases. These properties are better illustrated in Figures 4 and

**Table I Mechanical Properties of NBR/EVA Blends as a Function of the EVALSH Addition and Blend Composition**

Blend Components (%)			Strength Max Load ( $\sigma_{max}$ ) (MPa)	Ultimate Tensile Strength ( $\sigma_B$ ) (MPa)	Elongation Max Load ( $\epsilon_{max}$ ) (%)	Elongation at Break ( $\epsilon_B$ ) (%)
NBR	EVA	EVALSH				
100	0	0	0.16	0.14	350	1090
100	0	5	0.22	0.21	450	1520
85	15	0	0.33	0.23	330	1200
85	15	5	0.38	0.36	480	1310
80	20	0	0.50	0.30	290	1600
80	20	5	0.60	0.56	520	1180
70	30	0	2.26	1.20	510	2200
70	30	5	2.85	2.20	540	1090
60	40	0	4.12	4.27	750	2500
60	40	5	7.62	7.60	700	1500
40	60	0	8.85	8.82	970	2950
40	60	5	9.35	10.10	850	2460
20	80	0	18.00	17.84	980	3570
20	80	5	20.10	19.30	880	3530
0	100	0	21.56	21.30	820	1420
0	100	5	14.36	14.00	690	1150

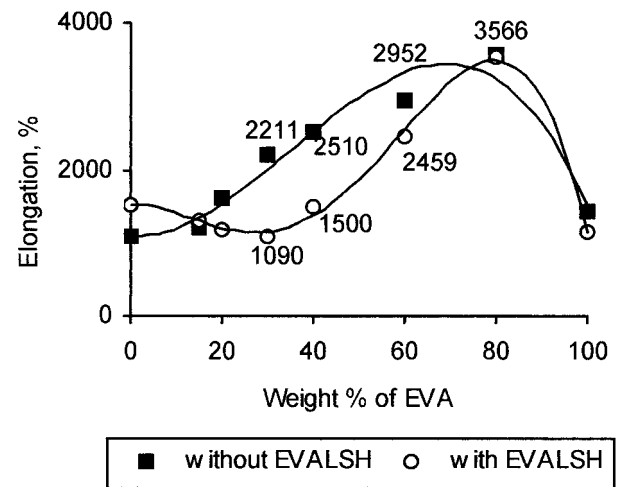
5, respectively. The addition of 5 phr of EVALSH resulted in an improvement of the tensile strength in all blend compositions except in pure EVA (Fig. 4). The highest difference between the values found in compatibilized and uncompatibilized blends can be observed in the range of 30 to 60 wt % of EVA. The addition of EVALSH in pure EVA decreases the ultimate tensile strength of the material. This behavior was also found in a



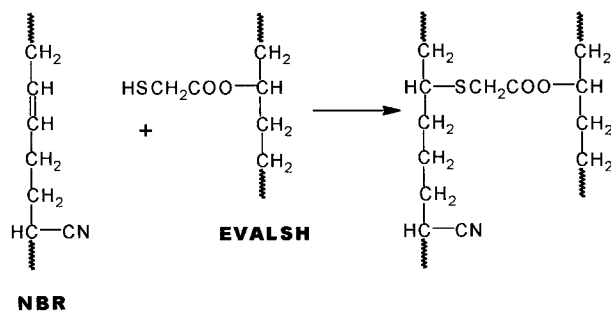
**Figure 4** Variation of ultimate tensile strength of NBR/EVA blends as a function of blend composition and compatibilization.

previous report concerning NR/EVA blends<sup>21</sup> and may be associated with the diluting effect of the EVALSH into the crystalline EVA phase.

The compatibilization decreases the elongation at break. The compositions in the range of 30 to 40 wt % of EVA present the highest differences in this property with the presence of EVALSH. This composition range may correspond to the co-continuous phase composition when a dual-continuous morphology is achieved and the blend be-



**Figure 5** Variation of elongation at break of NBR/EVA blends as a function of blend composition and compatibilization.



**Figure 6** Proposed reaction scheme between EVALSH and NBR.

has as a physical interpenetrating network. This network receives an additional reinforcement because of the strong interaction between the phases. The mercapto groups in the EVALSH chain of the compatibilizer react with the double bond of the NBR phase, according to the scheme presented in Figure 6. The morphological characteristic associated with interchain reactions contribute to a decreasing of the chains mobility and consequently, the elongation at break.

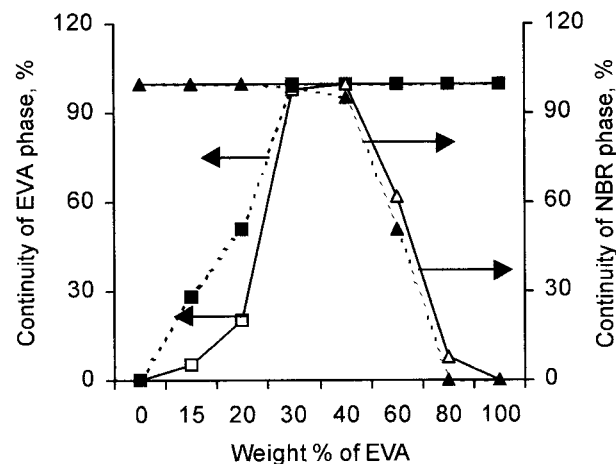
### Morphological Studies

The mechanical properties of polymer blends are directly related to the morphology. For the same processing conditions, the morphology is a function of the composition ratio and melt viscosities of each component in the blend.<sup>29</sup> The interfacial agents also exert a great influence on the morphology of the polymer blend. The presence of these compounds promotes a more uniform distribution of particle size and helps to stabilize the morphology during melt processing.<sup>30</sup> Blends involving a thermoplastic with an elastomer are phase-separated systems in which one phase is hard and solid whereas the other phase is rubbery at room temperature. The hard phase acts as pseudo-crosslinks. In NBR/EVA blends, the EVA phase contributes to the strength because of its crystallinity. In absence of this phase, the elastomeric compound starts to flow under stress. Therefore, the properties usually depend on the amount of the EVA phase present in this system. The tensile strength behavior presented in Figure 4 confirms this theory. The influence of the crystalline EVA phase on this property is expected to be higher when this phase is continuous. In the range of co-continuous phase composition, both phases are continuous and the material seems like a physical interpenetrating network.

To answer the questions originated from the mechanical behavior of compatibilized NBR/EVA blends, we decided to investigate the range of co-continuous phase by using both selective extraction experiments and SEM analysis.

For selective extraction experiments, compression-molded specimens of the blends were immersed into MEK, which is a selective solvent for NBR phase. The degree of continuity of each phase was determined according to the literature procedure.<sup>25</sup> The co-continuity is assumed when it is possible to extract all NBR phase without destroying the spatial shape of the sample initially immersed in the solvent. When EVA is dispersed in the NBR matrix, the shape of the specimen will be completely destroyed after the experiment. On the other hand, when EVA is the matrix, the solvent cannot diffuse inside the specimen and extract the NBR dispersed phase. In this case, the weight of the specimen before and after the immersion into the solvent should be the same.

Figure 7 shows the influence of the blend composition on the weight percentage of each component as a continuous phase. The values are also summarized in Table II. The range of phase inversion morphology in both uncompatibilized and compatibilized NBR/EVA blends is located around 30 and 40 wt % of EVA in the sample. Thomas et al.<sup>18</sup> have proposed a range between 40 and 60 wt % of EVA for a co-continuous morphology. The deviation between the range found by Thomas et al. and that one found in our studies may be related to the methodology for determina-



**Figure 7** Continuity of EVA and NBR phases as a function of the blend composition and compatibilization (dotted line corresponds to compatibilized blends).



**Table II** Continuity of EVA and NBR Phases as a Function of the Blend Composition and Compatibilization

Blend Composition (%)		Continuity of EVA Phase (%)		Continuity of NBR Phase (%)	
NBR	EVA	Without EVALSH	With 5-phr EBVALSH	Without EVALSH	With 5-phr EVALSH
85	15	5	28	100	100
80	20	20	51	100	100
70	30	98	100	100	99
60	40	100	100	100	95
40	60	100	100	58	54
20	80	100	100	5	0

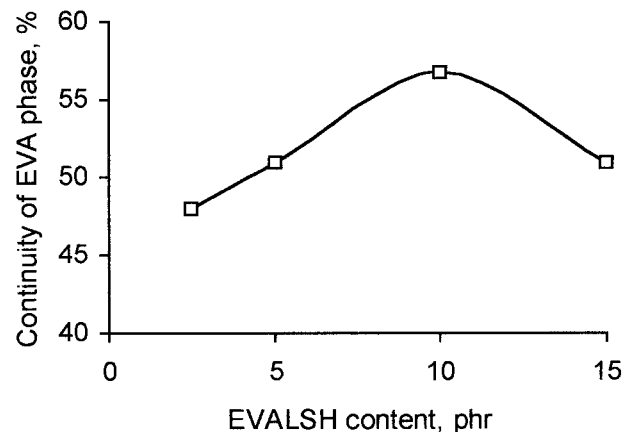
tion of the dual-phase continuity. In the Thomas' studies, the conclusions were based on SEM analysis. This technique is, of course, very helpful and common to analyze the phase inversion morphology. However, it may be nonrepresentative of the bulk material because it is taken from one specimen surface. Other techniques like selective extraction methodology, as used in our studies, should complement the information provided by SEM, especially in studies concerning co-continuous morphology.

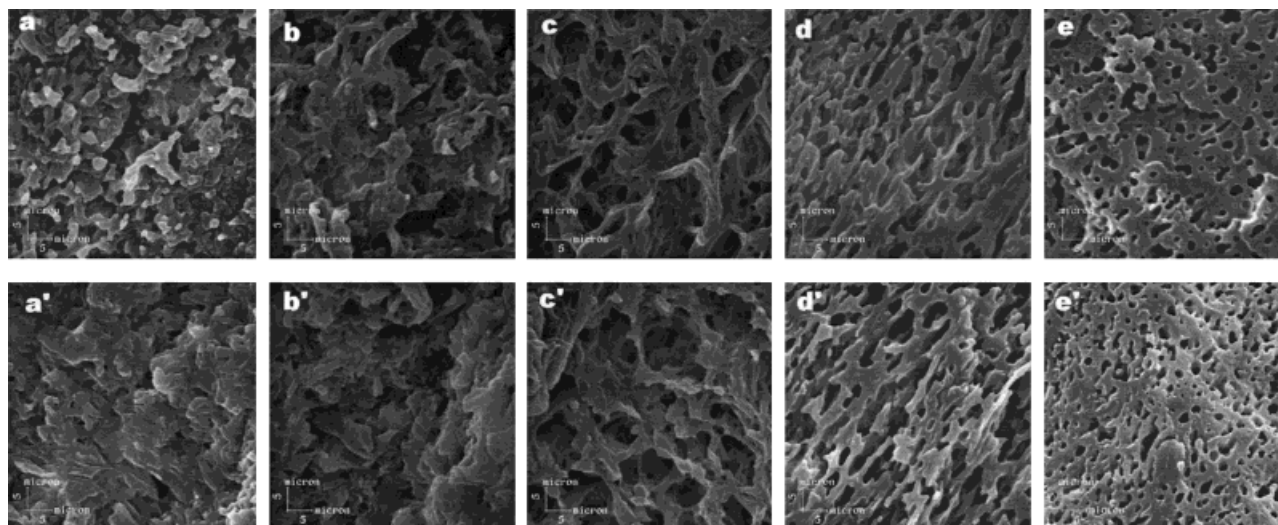
The continuity profiles observed in Figure 7 indicate that the compatibilization does not appear to displace the region of phase inversion but shifts the percolation points for both dispersed NBR and dispersed EVA. Indeed, a greater proportion of the EVA phase starts to be continuous at lower amount of EVA in the blend when 5 phr of EVALSH was added (see Fig. 7, dotted line). Considering for example, the NBR/EVA blends (80:20 wt %), the proportion of EVA phase with continuous morphology was found to be approximately 20 wt % in uncompatibilized blend. The presence of the mercapto-modified EVA increases the percentage of continuous EVA phase to around 51 wt %. The continuity degree of the EVA phase in NBR/EVA blends (80:20 wt %) also depends on the EVALSH concentration. As illustrated in Figure 8, the weight percentage of continuous EVA phase increases with the EVALSH content and reaches a maximum at 10 phr of the compatibilizer. Concerning the side of NBR dispersed phase in Figure 7, the addition of the compatibilizer shifts the percolation point for dispersed NBR to higher NBR composition.

According to several studies reported in literature,<sup>31-33</sup> the development of the morphology during the melt mixing of an heterogeneous polymer blend is related to several phenomena of drop

breakup and coalescence of the dispersed phase. When the dispersed phase has a high viscosity under shear conditions, droplets are stretched into elongated cylinders which can later break into a line of droplets.<sup>34</sup> The interfacial tension is the driving force to break up the threads, whereas the viscosity tends to retard the process. Droplets can also collide and recombine, depending on the concentration of the dispersed phase, the viscosity of the continuous phase, and the presence of compatibilizing agents. When the dispersed phase increases, the collision-coalescence phenomenon becomes important, giving rise to larger particles. These particles are not stable as droplets in the stress field and deform into fibers. The fiber content increases as the dispersed phase content increases, until phase inversion takes place.

The addition of compatibilizing agent in a heterogeneous polymer blend reduces the interfacial

**Figure 8** Continuity of the EVA phase in NBR/EVA blends (80:20 wt %) as a function of the EVALSH content.



**Figure 9** SEM of NBR/EVA blends with composition corresponding to (a) 20%, (b) 30%, (c) 40%, (d) 60%, and (e) 80% of EVA. The micrographs (a) to (e) denote the corresponding compatibilized blends, with 5 phr of EVALSH.

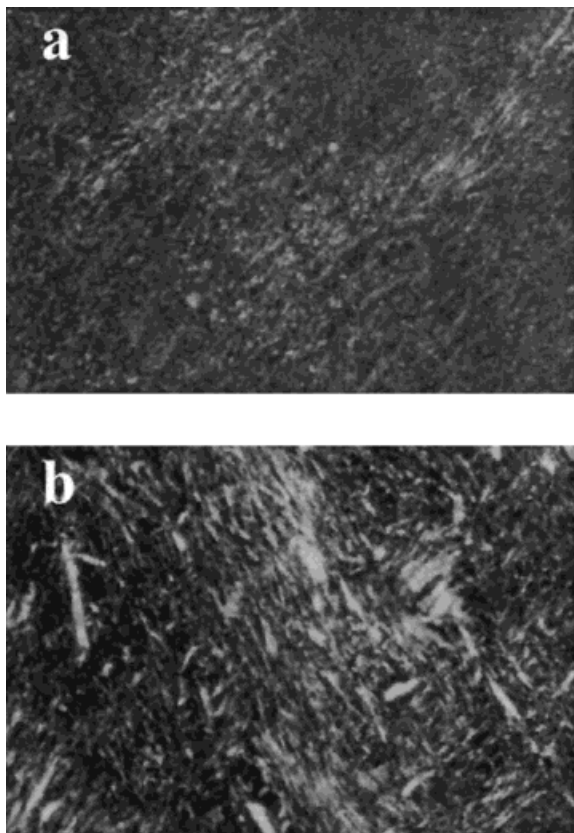
tension between the phases and permits a finer dispersion during mixing.<sup>35</sup> In addition, it can stabilize the morphology against coalescence and inhibit the formation of fibers.<sup>36</sup> As discussed in several articles, this phenomenon leads to phase inversion by droplet coalescence at higher concentration of the dispersed phase.<sup>36–39</sup>

Returning to the discussion of NBR/EVA systems studied in this work, one can observe similar behavior when NBR constitutes the dispersed phase, that is, the percolation point occurs at higher NBR concentration with the addition of the compatibilizer because the interface is supposed to be more stabilized against major phase coalescence. It is interesting to note, however, that the EVA dispersed phase displays an opposite phenomenon. The compatibilization shifts the percolation point toward lower EVA composition. In this case, the EVA droplets have time to elongate before the compatibilizer could reach the interface. In our blends, the EVALSH was preblended with NBR phase before the addition of the EVA component. This procedure was chosen to impart the chemical interaction between the mercapto groups of the compatibilizer and the unsaturated rubber. The grafted copolymer formed “*in situ*,” inside the NBR phase takes some time to diffuse to the interface and permits the dispersed phase to elongate. In addition, this “*in situ*” graft copolymer may be increasing the viscosity of the NBR phase. According to Elmendorf,<sup>34</sup> the breakup time of the cylindrical do-

main can vary from several seconds to several hours. When this time exceeds the droplet deformation time, cylindrical bodies will be formed. This phenomenon is especially true for systems with low interfacial tension and high viscosities. Concerning the NBR/EVA blends with higher amount of NBR, the displacement of the percolation threshold toward lower amount of EVA by the addition of EVALSH may be related to the phenomenon discussed by Elmendorf.<sup>34</sup> The presence of EVALSH helps to form a stable co-continuous network.

The SEM micrographs of NBR/EVA blends give additional information concerning morphology. Figure 9 presents the micrographs of these blends in which NBR phase was etched from the sample surface with MEK to provide a better insight into the blend morphology. The surface of uncompatibilized NBR/EVA (80:20 wt %) blend was destroyed after etching because NBR constitutes the matrix [Fig. 9(a)]. The compatibilization with EVALSH increases the proportion of EVA as continuous phase, as discussed previously. Therefore, the sample is less affected by etching and the morphology is very similar to those observed in the phase inversion region [see Fig. 9(a)]. These results confirm the stabilization of the co-continuous structure by the compatibilization.

Despite the great differences in mechanical properties, there is no substantial morphological difference between compatibilized and uncompatibilized NBR/EVA blends in the range be-



**Figure 10** Optical microscopy of NBR/EVA blend (80:20 wt %) (a) without EVALSH and (b) with 5 phr of EVALSH.

tween 70:30 to 40:60 wt % which corresponds to the co-continuity composition. For NBR/EVA blends (20:80 wt %), the presence of EVALSH promotes a more uniform distribution of the NBR dispersed phase [see Fig. 9(e)]. These morphologies suggest a reduction of interfacial tension with the compatibilization. The morphological differences in these blends are not very strong and agree with the similar mechanical behavior of these blends at this composition (see Figs. 4 and 5).

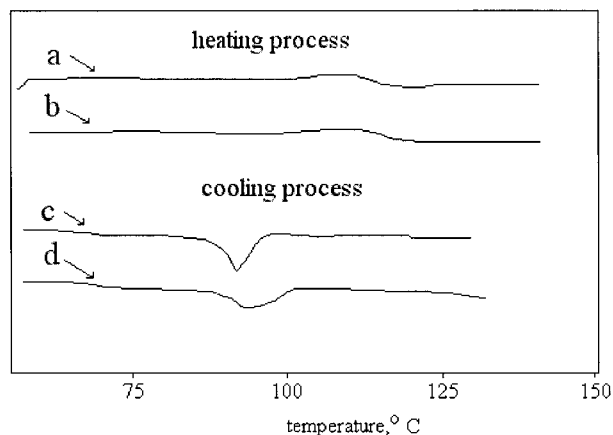
The optical microscopy taken with polarized light illustrated the morphological variations with the compatibilization. This technique is able to detect the effect of the EVALSH on the crystalline phase of the EVA component. As observed in Figure 10, the EVA crystals of uncompatibilized blends with low amount of EVA (20 wt %) are uniformly distributed whereas in compatibilized blends, larger crystals have been formed during processing. This behavior is also an indication of an increasing of the EVA as continuous phase.

### DSC Analysis

The intriguing results observed in optical microscopy analysis, prompted us to investigate these blends by DSC. Figure 11 displays the melting and crystallization curves of NBR/EVA 80:20 wt % blends taken during the heating and cooling process. The melting behavior of the EVA phase in both blends is not affected by the compatibilization (see curves a and b). However, the presence of EVALSH influences significantly the crystallization of the EVA phase. As observed in curve d (Fig. 11), the crystallization peak of EVA phase for compatibilized blend begins at higher temperature and is broader than that one observed for uncompatibilized blend, indicating the formation of more irregular crystals in the EVA phase. This phenomenon associated with the morphological observation obtained from SEM and optical microscopy suggests that the compatibilizing agent interacts with the EVA phase and changes the morphological situation of the blend.

### CONCLUSIONS

The addition of mercapto-modified EVA (EVALSH) in NBR/EVA blends exerts a substantial influence on the mechanical properties. Increasing the amount of the functionalized EVALSH copolymer also increases the tensile strength and elongation at break for NBR/EVA blends (80:20 wt %). Studies related to the blend



**Figure 11** Melting and crystallization curves of NBR/EVA blends (80:20 wt %) (a) without EVALSH and (b) with 5 phr of EVALSH (during the heating process); (c) without EVALSH and (d) with 5 phr of EVALSH (during the cooling process).



composition revealed that the compatibilization increases significantly the tensile strength but decreases the elongation at break in the phase inversion range.

The region of dual-phase continuity has been determined for NBR/EVA blends by using selective extraction experiments. The addition of the compatibilizing agent has not changed the region of phase inversion but influences the onset of percolation threshold for both dispersed NBR and dispersed EVA. In the case of dispersed NBR phase, the percolation was shifted to higher NBR concentration. These results are in agreement with several other results in the literature and can be explained by the interfacial effect of the functionalized copolymer reducing the NBR particle elongation and coalescence of the droplets. In the case of EVA dispersed phase, the percolation point is shifted toward lower EVA concentration. In compatibilized blends, the EVA particles elongate and form fibers but the formation of larger droplets by coalescence is prevented. From these results, it is possible to suggest that the compatibilization stabilize the co-continuous morphology for NBR-richer blends. In addition to phase inversion morphology, the presence of the interfacial modifier also affects the crystallization of the EVA phase. The results obtained from selective extraction experiments, SEM analysis, optical microscopy, and DSC experiments must be related to rheological behavior. In NBR-richer blends, the presence of the functionalized EVALSH copolymer may be influencing the viscosity of the NBR phase because of the formation of "in situ" graft copolymer, contributing to a higher ability of the EVA droplets elongation. The cylindrical bodies are, however, stabilized by the interfacial agent and decrease the ability of droplet breakup. The rheological studies associated with dynamical mechanical properties of these blends will be considered in future works from this laboratory.

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