

# Chemical Sensing Materials. I. Electrically Conductive SEBS Copolymer Systems

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**ABSTRACT:** Chemical sensing materials based on conductive carbon black (CB) filled [styrene-ethylene butylene-styrene] triblock-copolymers (SEBS) were investigated. Several types of SEBS copolymers were studied, differing in composition and melt viscosity. The sensing is based on electrical conductivity changes upon solvent sorption/desorption. Compression molding SEBS composites containing various amounts of CB were prepared. Their electrical conductivity was measured and samples containing CB, preferentially located in the continuous ethylene/butylene (EB) phase, at a level near the corresponding percolation threshold were used for the sensing experiments. The conductivity was measured during several exposure/drying cycles. Structure characterization included scanning electron microscopy (SEM), dynamic mechanical analysis (DMA), and calorime-

try (DSC). The SEBS composites exhibit large reversible changes in conductivity upon exposure to a limited number of solvents, e.g., acetone, *n*-heptane, and air drying cycles. This behavior was related to the sorption kinetics, affected by the solvent characteristics (solubility parameter, polarity, molecular volume and vapor pressure). The samples' resistance tended to return to their initial value upon short drying of acetone, and longer drying of other studied solvents. The nature of the SEBS, the CB content, and mixing temperature are all significant parameters, determining the sample's structure and the resultant sensing property. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 113: 3322–3329, 2009

**Key words:** chemical sensor; SEBS; thermoplastic elastomer; carbon black; conductivity

## INTRODUCTION

Chemical sensors based on electrically conductive polymers have been widely studied and developed in recent years.<sup>1–4</sup> A common method to produce a conductive polymer composite is by mixing insulating polymers and conducting fillers, e.g., carbon black (CB), metal particles, etc.<sup>5,6</sup> Upon exposure to organic chemicals compatible with the polymer matrix, the composite resistance increases drastically.<sup>7</sup> It is generally believed that the matrix swelling, increasing the inter-filler particles distance, accounts for the sensing ability.<sup>2</sup> The different effects for various solvents have been explained in terms of the corresponding solubility parameters of the matrix and the analytes. Through using different polymer matrices, a certain degree of selectivity to different analytes can be achieved.<sup>8</sup>

CB-filled polyethylene composites were discovered to have some chemical sensing capabilities.<sup>7</sup> The feasibility of the concept of polymer/CB compounds as chemical sensing materials has also been demonstrated for other polymers i.e., poly(tetrafluoroethylene), polyvinyl chloride, polybutadiene, PMMA, and polyurethane.<sup>2,4,6,9</sup>

Recently, few preliminary studies on sensing materials based on conductive CB-containing miscible polymer blends have been reported. Miscible blends of poly(vinyl acetate) and poly(methyl methacrylate) have been used to produce electrically conducting CB-containing compounds. They exhibit electrical resistance sensitive to the nature and concentration of an analyte in the vapor phase. It was proposed that the utilization of miscible binary polymers blends as sensors for various solvent vapors offers the opportunity to develop a diversity of polymer compounds detector arrays in comparison to those fabricated from single polymers.<sup>10</sup>

Narkis and coworkers<sup>3,11,12</sup> have studied liquid sensing materials based on electrically conductive CB-containing immiscible polymer blends. The multiphase nature of these systems provides an opportunity for the CB particles to distribute nonuniformly within the phases, due to the different properties of the blend components. The conductivity enhancement in these blends is due preferential localization of the CB particles in one of the continuous phases or at their interface, giving rise to conductive networks at low CB content.<sup>13</sup>

Styrene-ethylene/butylene-styrene (SEBS) molecules are block copolymers characterized as thermoplastic elastomers. Since the S and the ethylene/butylene (EB) blocks are not compatible, they form polystyrene domains locking both ends of the EB

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TABLE I  
Properties of Various SEBS Used

	G1650E	G1651E	G1652	G1657	FG1901X
Styrene/ Rubber Ratio	30/70	33/67	30/70	13/87	30/70
Brookfield Viscosity <sup>a</sup> (Toluene solution), Pa·s at 25°C	8	>50	1.8	4.2	5
Melt Index <sup>b</sup> (g/10 min)	<1	<1	5	22	22
Specific gravity (g/cm <sup>3</sup> )	0.91	0.91	0.91	0.9	0.91
Comments	–	–	–	–	1.7% bound functionality (Maleic Anhydride)

<sup>a</sup> Neat polymer concentration, 25 wt %.

<sup>b</sup> 230°C, 5 kg.

block into a physically crosslinked network structure. The physical crosslinking of SEBS is a reversible process, an important characteristic of styrenic block copolymers.<sup>14–16</sup> Different from immiscible blends, the two phases in SEBS are chemically bonded and the average size of the dispersed PS domains is on the order of 10 to 30 nm, the same order of magnitude of the CB particles size.<sup>17</sup>

The main objectives of this research were to develop and characterize sensing materials for the detection of chemical compounds, with high sensitivity and reliability, based on conductive CB filled SEBS, taking advantage of its rubbery and glassy bonded phases at ambient temperature and its availability in different compositions and molecular weights.

## EXPERIMENTAL

Polymers used for the preparation of compression molded CB-containing samples were thermoplastic SEBS tri-block-copolymers (SEBS), Kraton G Polymers, (Kraton Polymers, UK). Five types of Kraton G were used, G1650E, G1651E, G1652, G1657, and FG1901X, differing in S/EB ratio, solution viscosity, and group functionality (see Table I). A highly structured electrically conductive CB, Ketjenblack EC-600

(Akzo-Nobel, Netherlands), was used as the conductive filler (density of 1.8 g/cm<sup>3</sup>, BET of 1250 m<sup>2</sup>/g). CB content within the polymer is referred in the text in phr (parts per hundred, by weight).

SEBS/CB blends were prepared by a standard procedure of melt mixing in a Brabender Plastograph equipped with a 30 cm<sup>3</sup> cell. The components were compounded at 260°C for approximately 15 min. The resulting blends were cut to small pieces and then compression molded at 125–170°C, depending on the type of polymer used in the blend.

The volume electrical resistivity was measured using the 2-point method. The resistance between two silver marks painted on the edges of compression-molded samples ( $\sim 10 \times 20 \times 23$  mm<sup>3</sup> in sizes) was measured (DIN-53596) using a Fluke 8840A multimeter. Silver paint was applied to minimize contact resistance between the sample and the electrodes [Fig. 1(a)].

For sensor analysis,  $\sim 3 \times 3 \times 23$  mm<sup>3</sup> compression molded samples were used. Two small amounts of silver paint were applied on the surface of the samples, 10 mm apart [Fig. 1(b)]. Copper electric wires were connected to the sample at the marked areas. Liquid sensing experiments were performed by immersion/drying cycles at  $\sim 25^\circ\text{C}$  along with

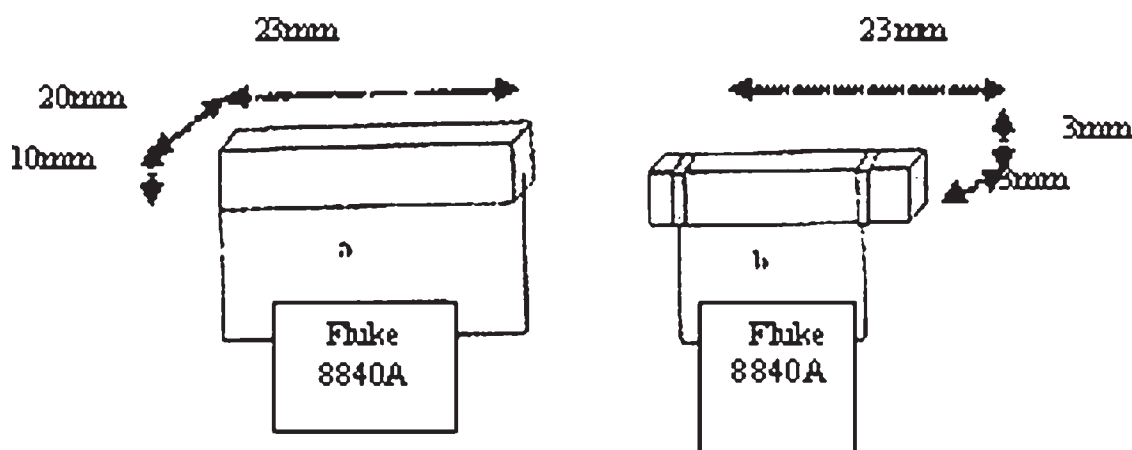


Figure 1 Two-point methods of DC resistivity measurements: (a) edge connected–volume resistivity; (b) surface connected–surface resistivity.

continuous monitoring of the changing resistance. All data are presented as relative resistance,  $R_t/R_0$ , where  $R_0$  is the initial resistance of the sensor and  $R_t$  is the measured resistance at time  $t$ . The solvents used in this study include acetone and *n*-heptane.

Scanning electron microscopy (SEM) of freeze-structures surfaces was performed using a Zeiss EVO-50, at an accelerating voltage of 15 kV. Samples were gold sputtered prior to observation. Differential scanning calorimetry (DCSQ10) was carried out at heating and cooling rates of 10°C/min in a nitrogen atmosphere. Samples ~ 5 mg in weight were heated to a temperature above their  $T_g$ , cooled at the same rate, and subsequently reheated.

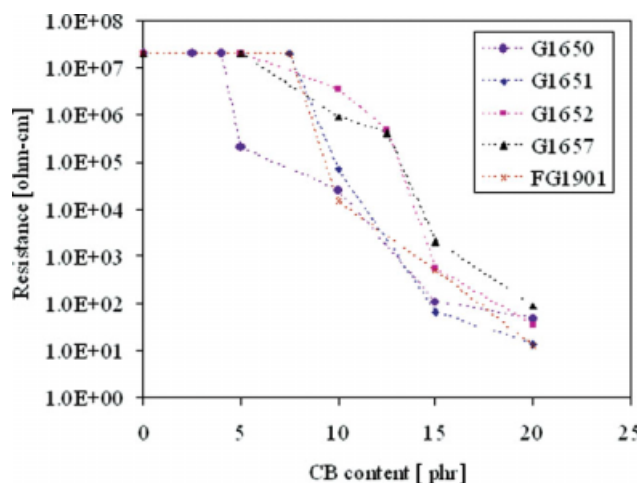
Sorption of solvents was studied using an immersion/weight gain method at room temperature. Compression molded samples, ~ 3 × 3 × 23 mm<sup>3</sup> in sizes, were immersed in plastic bottles containing the respective solvents. At various time intervals, the specimens were removed from the solvent, wiped off by a filter paper, and weighed. Solvent content in the samples at time  $t$  was calculated as:

$$\% \text{ Sorption} = (\text{Weight at time } t - \text{Initial weight}) \times 100 / \text{Initial weight}$$

## RESULTS AND DISCUSSION

### Resistivity–composition relationships

The volume resistivity of SEBS as function of CB content is depicted in Figure 2. The percolation threshold for G1650 is the lowest of the studied SEBS copolymers, obtained at approximately 5 phr CB. G1651, G1657, and FG1901 percolate at approxi-

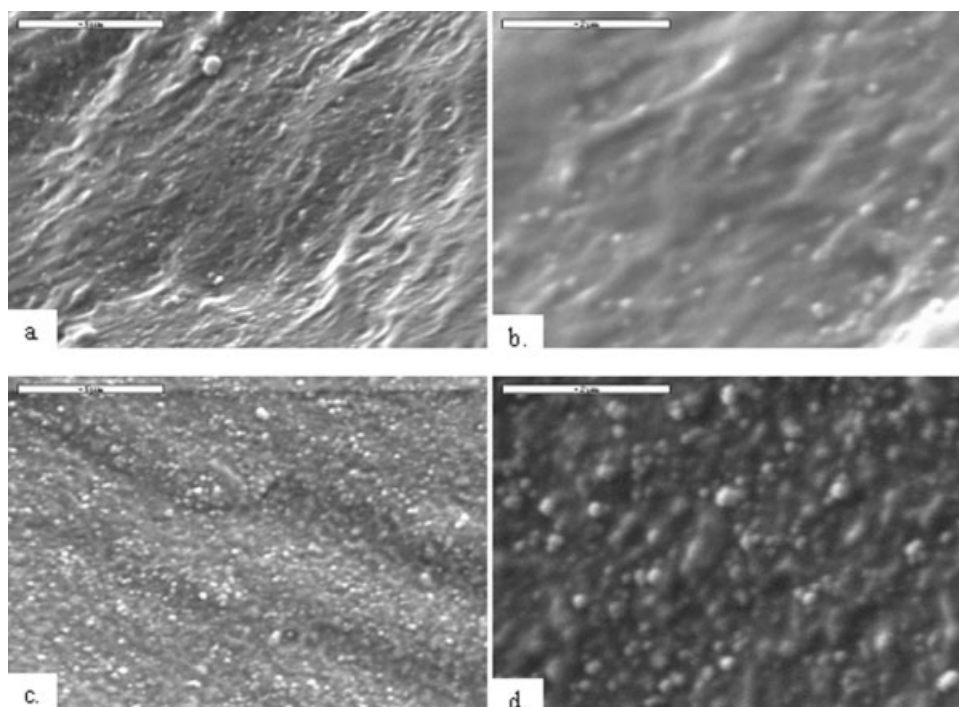


**Figure 2** Volume Resistivity vs. CB content for variety of SEBS. Specimens size ~ 10 × 20 × 23 mm<sup>3</sup>. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

mately 10 phr CB, whereas G1652 at 15 phr. These results are opposed to what would be expected based on literature. G1652 has the lowest viscosity (1.8 Pa s) among the studied SEBS polymers, thus expected to exhibit the lowest percolation threshold, in contrary to the depicted in Figure 2.<sup>18</sup> In contrast, G1651 has the highest viscosity (>50 Pa s) among the studied SEBS polymers, thus, expected to exhibit the highest percolation threshold, different from the depicted in Figure 2.<sup>18</sup> Melt viscosity affects both the CB deagglomeration and the ability to form continuous networks. It should be noted that all systems were blended at the same temperature, while the molding temperature was adjusted according to the melt viscosity.

A gradual resistivity decrease with increasing CB content characterizes the SEBS/CB system, as opposed to a sharp percolative behavior observed for non-polar matrices.<sup>19</sup> This behavior was previously reported for several thermoplastic elastomer/CB systems i.e., styrene-isoprene-styrene copolymer and polyester-type TPU.<sup>20,21</sup> The gradual resistivity drop may be attributed to matrices of relatively high surface tension (e.g., ethylene-vinyl acetate copolymers, TPU), which tend to interact with the dispersed CB, resulting in homogeneous distribution of CB particles, delaying their ability to cluster and form conductive networks.<sup>22</sup> Accordingly, the SEBS/CB system percolates at a relatively high CB content (10–15 phr), similar to other polar and amorphous matrices. For example, in an amorphous and polar co-polyamide, percolation has not been realized up to CB content of 14 wt %, owing to the formation of uniform particle distribution.<sup>23</sup> A percolation threshold of approximately 10 phr is obtained in Styrene-Butadiene rubber (SBR) with Ketjenblack EC CB.<sup>24</sup> Carmona depicts PS as demanding a higher CB content than polyethylene or polypropylene in order to percolate since PS has a higher surface tension than either PP or PE (~ 41, 30, 36 dyne/cm at 20°C, respectively)<sup>18</sup>.

It can be noticed that from above percolation threshold, for a given CB content, the value of resistivity is higher for SEBS with higher content of the rubbery EB phase (G1657 with 87% EB compared to other SEBS with ~ 70% EB). The main reason for this behavior is that CB distributed mainly in the rubbery EB phase. The average size of the PS domains is on the order of 10–30 nm, the same order of magnitude of the CB particles.<sup>17</sup> Therefore, for a given nominal CB content, the actual CB content in the EB phase is lower in the copolymer consisting of higher amount of EB phase. It should be recalled that the EB phase is the continuous one in the studied SEBS tri-block-copolymers, enabling the formation of continuous CB networks through out the entire material.



**Figure 3** SEM micrographs of G1650 freeze fractured blends with different CB content: (a–b) 2.5 phr and (c–d) 10 phr. 5 m bar (a, c); 2 m bar (b, d).

The distribution of CB within neat SEBS is shown in Figure 3. In the 2.5-phr CB sample [Fig. 3(a,b)] CB particles are randomly dispersed in the matrix; however, too apart for creating a conductive network. In the higher CB content sample, 10-phr [Fig. 3(c,d)], although large number of CB particles seem to be quite uniformly but more densely dispersed in the SEBS, some particle chains can be observed, thus, forming electrically conductive networks.

#### Resistivity–mixing temperature relationships

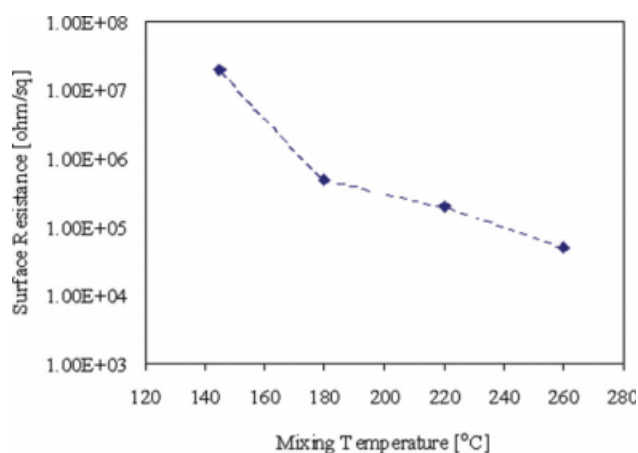
Figure 4 depicts the effect of blending temperature on the resistivity of the G1657/15phr CB blends. At this composition, above the percolation threshold, the CB particles form conductive networks within the continuous EB phase. The surface resistivity decreases by more than two orders of magnitude upon increasing the mixing temperature from 140 to 260°C (interval was determined by viscosity). The increase in mixing temperature reduces the viscosity of the matrix in the internal mixer, decreasing the efficiency of CB agglomerates breakdown, however, increasing the efficiency of CB networks formation.<sup>25</sup> Therefore, 260°C was selected for all blendings.

#### Thermal behavior

The two-phase system in SEBS polymers results in two distinct glass transition temperatures corresponding to poly(ethylene/butylene) at  $T_{g1} \sim (-55^\circ\text{C})$  and

to polystyrene at  $T_{g2} \sim 100^\circ\text{C}$  in agreement with literature.<sup>14</sup> Table II summarizes the  $T_g$  values of the “soft” and “hard” domains of neat SEBS and CB containing SEBS, as was measured by DSC.<sup>14</sup> It should be mentioned that it was quite difficult to determine the  $T_g$  values due to the shape and width of the transition region in the thermograms.

The data in Table II shows that the addition of 20 phr CB results in, unexpectedly, a decrease in the  $T_g$  of the rubbery phase ( $T_{g1}$ ), presently unexplained. However, lower CB contents, as studied for G1651,



**Figure 4** Surface resistivity vs. Brabender mixing temperature for G1657/15phr CB blends. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

**TABLE II**  
Glass Transition Temperatures of Neat SEBS and CB  
Containing SEBS as Determined by DSC

SEBS Type	Styrene part in SEBS	EB part in SEBS	CB content [phr]	$T_{g1}$ [°C]	$T_{g2}$ [°C]
G1650	30	70	–	–55	93
G1650	30	70	20	–59	111
G1651	33	67	–	–57	110
G1651	33	67	5	–60	101
G1651	33	67	7.5	–57	102
G1651	33	67	10	–59	100
G1651	33	67	15	–59	106
G1651	33	67	20	63	101
G1652	30	70	–	–57	100
G1652	30	70	20	–67	117
G1657	13	87	–	–59	106
G1657	13	87	20	–61	97
FG 1901	30	70	–	–53	85
FG 1901	30	70	20	–59	105

do not seem to affect the  $T_{g1}$  value. For the same systems, there is no clear tendency of the effect of CB on the glass transition temperature of the hard phase ( $T_{g2}$ ). It was quite difficult to determine accurately the  $T_{g2}$  value, probably due to the low content of the hard PS phase.

### Sensing properties

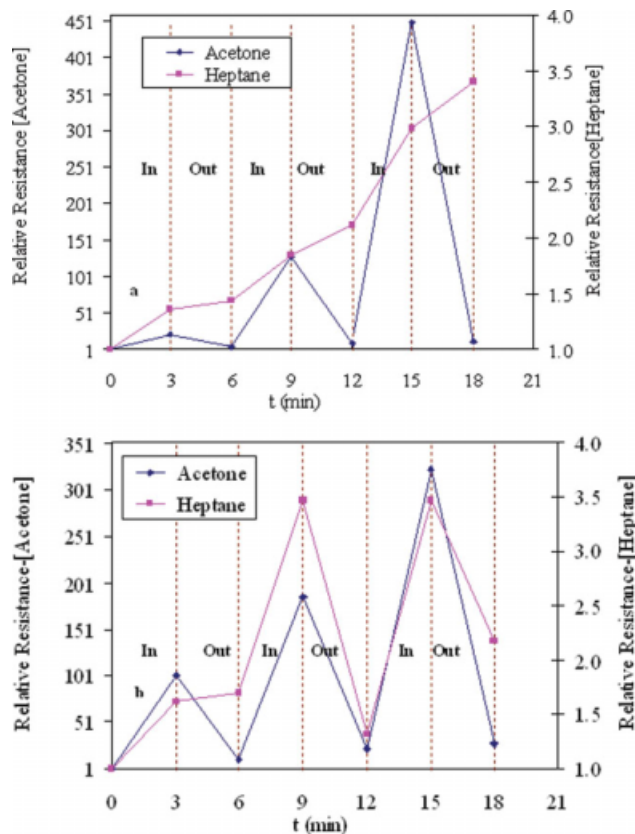
Selected SEBS/CB compression-molded samples were studied as sensing materials for various liquids. The compression molded samples containing 15 or 20phr CB (just above the percolation threshold), exhibited similar resistance changes upon exposure to the studied liquids. Figure 5 depicts the relative resistance,  $R_r = R_t/R_0$  of G1651 [Fig. 5(a)] and G1652 [Fig. 5(b)] blends with 15 phr CB, as a function of cycled exposure time to acetone and heptane. In a single cycle, a sample was immersed for 3 min in the studied solvents and then allowed to dry in air for 3 min. Three sequential cycles were performed for each specimen. SEBS G1651 and G1652 have similar PS/EB phase content of 33/67 and 30/70, respectively. The main difference between these polymers is in their molecular weight. G1651 has the highest solution viscosity of more than 50 Pa s and G1652 has the lowest solution viscosity of 1.8 Pa s (The viscosity values mentioned are of a 25% solution of the polymer in toluene at 25°C), among the studied SEBS polymers.

The G1651/15 phr CB and G1652/15 phr CB samples displayed an increasing resistance upon exposure to heptane and acetone. This behavior can be interpreted in terms of a percolation-type theory. During exposure, the polymeric matrix swells owing to solvent sorption, decreasing the effective volume fraction of CB particles and, thus, causing resistance increase in proximity to the percolation threshold.<sup>8</sup>

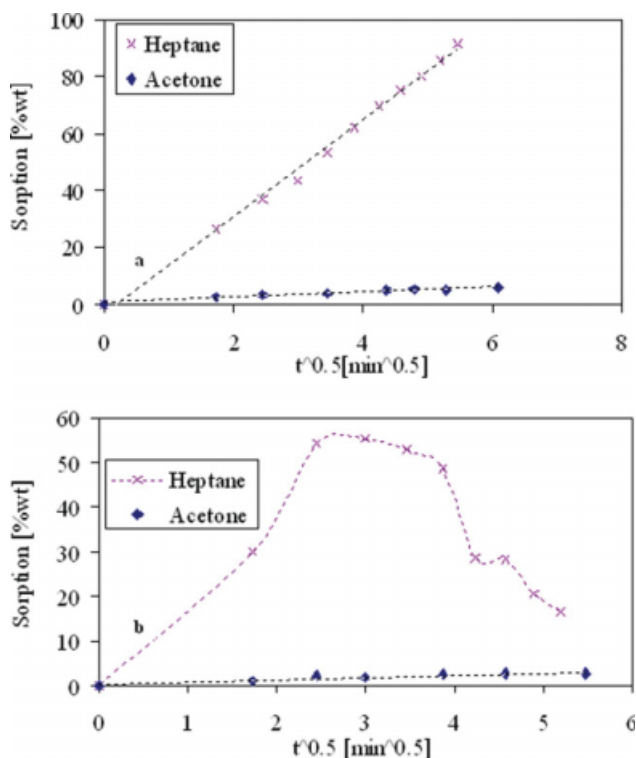
G1651/15 phr CB [Fig. 5(a)] and G1652/15 phr CB [Fig. 5(b)] have similar reversible behavior in acetone. G1651/15 phr CB samples exposed to acetone exhibit high resistance increase while the maximal resistance increases with the cycle number (up to a relative resistance of 450 in the 3rd cycle). Differently, G1651/15 phr CB samples exposed to heptane show permanent low increase in resistance (up to a relative resistance of 3.5 in the 3rd cycle) G1652/15 phr CB; however, exhibits reversible behavior. The different behavior of G1651/15 phr CB [Fig. 5(a)] and G1652/15 phr CB [Fig. 5(b)] in heptane can be explained by the difference in the polymers molecular weight, expressed in different sorption behavior in heptane, as discussed below [Fig. 6(a,b)].

Table III presents the solubility parameters ( $\delta$ ) and boiling temperatures ( $T_b$ ) of the solvents presently used for this study. These parameters are crucial to understand the different behavior of SEBS in these solvents. The better the solubility the higher the swelling. The lower the boiling point of the solvent (high vapor pressure), the faster it can desorb out from the sample during the drying cycle.<sup>26–28</sup>

Figure 6 depicts the sorption of acetone and heptane in G1651/15 phr CB [Fig. 6(a)] and G1652/15



**Figure 5** Relative resistance vs. exposure time to different solvents for (a) G1651/15 phr CB and (b) G1652/15 phr CB. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]



**Figure 6** Mass change vs. square (root of time) for (a) G1651/15 phr CB and (b) G1652/15 phr CB in various solvents. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

phr CB [Fig. 6(b)] as a function of square root of exposure time. For G1651/15 phr CB [Fig. 6(a)] the linear relation between the fraction of solvent absorbed and  $t^{1/2}$  indicates that Fick's law for diffusion is applicable. For G1652/15 phr CB [Fig. 6(b)] the sorption of acetone is similar to that in the G1651/15 phr CB sample, following Fick's law. However, the sorption of heptane first increases with  $t^{1/2}$  followed by a steep decrease. The decrease in weight implies that the G1652/15 phr CB sample is dissolving in heptane. Since G1652 has the lowest low-molecular weight, it can be dissolved more easily in heptane, which is a good solvent for the EB phase. Differently, G1651, having the highest molecular weight, is not soluble in heptane.

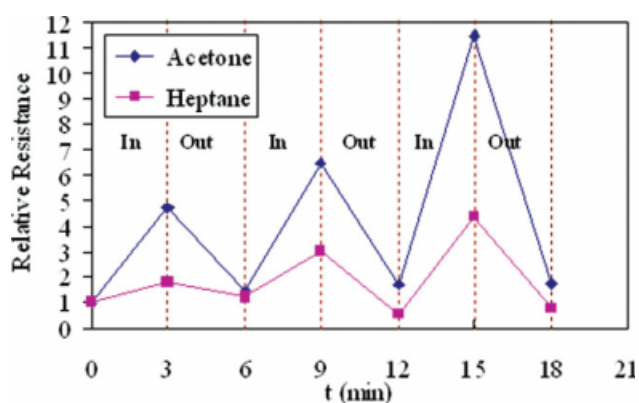
The different sensing behavior in different solvents can be explained by the difference in the solubility parameters,  $\delta$ . The solubility parameter of SEBS (G1651 and G1652) was estimated, using the group contribution method,<sup>26</sup> to be  $\sim 8.3$  (cal cm<sup>-3</sup>)<sup>1/2</sup>.

**TABLE III**  
Solubility Parameters and Boiling Points of the Various Solvents Used in this Study

	$T_b$ (°C)	$\delta$ (cal cm <sup>-3</sup> ) <sup>1/2</sup>
Acetone	$\sim 56$	9.9
<i>n</i> -Heptane	$\sim 98$	7.45

The sorption of acetone ( $\delta = 9.9$  (cal cm<sup>-3</sup>)<sup>1/2</sup>) is rather low, less than 7 wt %, owing to the relative large difference between the solubility parameter of SEBS and acetone. Thus, it can be concluded that when SEBS/CB samples are exposed to acetone, the acetone molecules are being absorbed mainly by the surface of the sample. When the sample is allowed to dry in air, the acetone molecules diffuse out of the sample quite quickly, since the acetone boiling point ( $\sim 56^\circ\text{C}$ ) is quite low, resulting in the observed reversible behavior. In contrast, in heptane ( $\delta = 7.45$ ), a good solvent for the continuous mid-block EB phase,<sup>27-29</sup> the solvent molecules diffuse into the bulk. They can't diffuse quickly out during the air drying due to their high boiling point ( $\sim 98^\circ\text{C}$ ). Thus, permanent low increase in resistance is observed for the G1651/15 phr CB sample [Fig. 5(a)]. The reversible behavior of G1652/15 phr CB [Fig. 5(b)] sample observed in heptane, in contrary to G1651/15 phr CB sample, is actually due to dissolution of the G1652 matrix in heptane, resulting in a higher nominal CB content within the sample (No CB was observed in the solvent).

Figure 7 depicts the relative resistance,  $R_r$ , of G1657 with 15 phr CB as a function of exposure time to acetone and heptane for three sequential cycles of 3 min. The samples exhibited similar reversible behavior both in acetone and heptane. A relatively small reversible increase was observed for the G1657/15 phr CB samples compared with the G1651/15 phr CB [Fig. 5(a)] samples and G1652/15 phr CB [Fig. 5(b)] samples in acetone. G1657 has the lowest PS phase content (13% wt) among the studied SEBS copolymer. Also, G1657 has relatively low solution viscosity (4.2 Pa s) compared to G1651 ( $>50$  Pa s). Since the G1657 copolymer consists of 87% EB phase, compared to G1651 (67%) and G1652 (70%) and since CB distributes mainly in the EB phase, at a nominal CB content the effective volume fraction



**Figure 7** Relative resistance vs. exposure time to different solvents for G1657/15 phr CB. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

of CB is lowest in the G1657 copolymer. Hence, the G1657/15 phr CB sample is the least sensitive to solvents, exhibiting small increases in resistance (Maximal  $R_r$  for G1657 is 12, while for the other studied copolymers G1651 and G1652 it is 450 and 320, respectively). Thus, the composition of the SEBS copolymers is an important parameter in determining their sensitivity.

The sorption of acetone and heptane by the G1657/15phr CB blend (not shown) is similar to the behavior observed for G1652/15phr CB samples [Fig. 6(b)]. The behavior of the G1657/15phr CB sample in acetone is similar to that of G1651/15phr CB and G1652/15phr CB samples in acetone, but exhibiting the lowest relative resistance increase. Since G1657 has relatively low-molecular weight, it can be dissolved more easily in heptane, resulting in a higher nominal CB content within the G1657/15phr CB sample, allowing the sample to return to its initial or even lower resistance when dried in air.

Three consecutive immersion/drying cycles were performed in order to characterize the samples' resistance reproducibility. Significant resistance changes are observed between the subsequent immersion/drying cycles. In most cases, the sensitivity of the SEBS/CB samples significantly increased in the second and third cycle. A similar behavior has been already observed for ethylene vinyl acetate/CB filaments exposed to non-polar solvents, i.e., benzene and *n*-heptane and thermoplastic polyurethane/CB filaments exposed to methanol.<sup>13</sup> These changes in resistance may be accounted for by the occurrence of structural changes in the samples (occurring mainly in the outer skin regions) due to solvent sorption/desorption during the immersion/drying cycles, respectively. These structural changes may also activate mechanisms of solvent penetration into the compound through capillarity and transport through microcracks. Thus, enhanced solvent permeation (in and out) can occur through small flaws in the outer skin regions and presumably also by some debonding of CB particles from the matrix, followed by solvent diffusion from the interface into the matrix.

## CONCLUSIONS

The two-phase microstructure of SEBS, a soft ethylene/butylene phase and hard domains of polystyrene, results in two distinct glass transition temperatures corresponding to poly(ethylene/butylene) and polystyrene. The CB particles are preferentially located in the continuous EB phase. The SEBS/CB system percolates at a relatively high CB content, attributed to matrices of higher surface tension, which tend to interact with the dispersed CB, delaying its ability to cluster and form conductive CB networks. Similar resistance values are obtained for the

SEBS/CB systems with different SEBS copolymers studied of different molecular weight but similar PS/EB ratio. Above the percolation threshold for a given CB content, the value of resistivity is higher for SEBS with higher content of the rubbery EB phase due to the preferred location of the CB in that phase of the copolymer.

All CB-containing compression molded samples displayed an increase in resistance upon exposure to acetone and *n*-heptane. Samples exposed to acetone exhibited the highest resistance increase, i.e., highest sensitivity. The SEBS/CB systems were limited to acetone and partially to heptane due to their solubility in other common solvents. The system with SEBS copolymer having the highest EB phase content was the least sensitive among the studied SEBS/CB systems. The relative rapid recovery of SEBS/CB samples' initial values for acetone, suggests that morphological changes, owing to solvent sorption, mainly occur in the outer skin regions of the samples, whereas the core region remains essentially intact. The *n*-heptane is absorbed in the sample's bulk and its desorption is slow owing to its relative low vapor pressure.

The SEBS composition, molecular weight, mixing temperature, and CB content determine the blend structure and the resultant sensing properties. The main sensing phase is the rubbery EB while the rigid styrene domains contribute to the materials mechanical stability.

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