Utilization of Nano-Black Sand as Filler in Styrene Butadiene Rubber Composites

S. N. Lawandy¹, R. Sayed², B. K. Saleh*¹, S. F. Halim¹

¹ Polymer Metrology and Technology Lab., National Institute of Standards, Egypt.
² Nanotechnology and Nanometrology Lab., National Institute of Standards, Egypt.

Introduction

The preparation of polymeric nanocomposites filled with inorganic nanoparticles has been of great interest for both academic researches and industrial applications. A wide variety of inorganic materials has been used in polymer nanocomposites [1]. Among them, silica is the most studied material. The hybrid structures of silica/polymer have excellent physical reinforcement, high thermal resistance, high flexibility, high gas permeability and low surface energy [2, 3].

Egyptian black sand (BS) deposits occur mainly in four localities namely, the beach areas of Rosetta, Damietta, north Sinai, and the coastal sand dunes of El Burullus-Baltim [4]. In literature, the Egyptian black sand (BS) deposits were subjected to extensive research which dealt mainly with the mineralogy of these black sands and discussion of their economics [5]. Many studies stated that the Egyptian black sand deposits comprise huge reserves of certain economic mineral, for example, zircon (ZrSiO4) [6], rutile (TiO2) [7], ilmenite (Fe2+TiO3) [8], and garnet (X3Y2Si3O12) where X-Mg, Fe, Ca and Y-Al, Fe, Cr) [9]. However, the average content of total economic minerals differs from place to other along the northern coast of Egypt [10].

The objective of the present study is to take advantage of the sand nature collected from the beach area of Rosetta area and use it, for the first time, as a filler to tailor styrene-butadiene polymeric nanocomposites. Ball mill was utilized to process BS particles to the nanoscale range, because it is an effective technique to produce nanoparticles [12]. Also, it has been shown to modify the filler morphology, which enhances the compatibility of fillers with polymer blends [13].
**Experimental**

**Materials and Methods**

BS samples were collected, using a plastic cup, from the beach area of Rosetta, Egypt's Beheira Governorate. They were selected from the upper 10 cm layer of the sea shore and then stored in plastic bags. The collected BS samples were sieved to remove coarse particles then dried in an air vacuum oven. A laboratory ball milling machine used to reduce the sand particle size to the nanoscale. Styrene butadiene rubber (SBR), trade name- Buna SE 1502 L supplied by ARLANXEO Deutschland GmbH, Business Unit Tire & Specialty. Zinc oxide and stearic acid were added to the SBR gum at first as plasticizers. Curing system comprises from N-cyclohexyl benzthiazyl sulfonamide (CBS), diphenyl guanidine (DPG) and sulfur (S) was added. Diphenyl guanidine (DPG) accelerator was used to accelerate curing process. 2-Mercaptobenzimidazole (MB) was used as antioxidant. To evaluate the role of BS-NPs as a filler in the SBR composites, SBR compounds containing high abrasion furnace black (HAF-N330), with mean particle size 32 nm and nanosilica (NS) with mean particle size 12 nm were prepared. The formulations of the different SBR composites are given in Table (1).

**Preparation of BS-NPs/SBR nanocomposites**

The black sand nanoparticles/SBR rubber (BS-NPs/SBR) nanocomposites were compounded using laboratory two-roll mill (152.4 x 330.2 mm) at a gear ratio 1:1.4. The nip gap, roll speed ratio, and number of passes were kept the same for all of the mixes. Firstly, the SBR matrix was masticated on a two-roll mill. Then different types of fillers along with different concentrations of BS-NPs were added to the SBR matrix followed by other ingredients according to ASTM D-3182-16. Different mix formulations are given in table 1.

**Cure characteristics and samples preparation**

The rheometeric properties were determined with an oscillating disk rheometer (Alpha Technologies MDR 2000) working at 170°C for 30 minute. The cure characteristics of the different mixes maximum torque (MH), minimum torque (ML), scorch time ts (a measure of premature vulcanization of rubber) and the optimum cure time (Tc90) time required to reach 90% crosslinking) were recorded. Delta torque (∆M = M_H - M_L) and Cure rate index (CRI = 100/ tc90 - ts) were calculated. Sheet samples for testing were compression molded in a laboratory hydraulic press (Mackey Bowley, C1136199) at 170°C at a pressure of 13.5 MPa according to ASTM D2084-17. The prepared nanocomposites were molded to the optimum cure, using molding conditions that were previously determined from rheometer data. Dumbbell shape samples of thickness 2.5 mm were cut from the compressed sheets using standard cutter according to ASTM D 412-16.

**Characteristics**

**X-Ray Diffraction**

X-Ray Diffractometry (XRD) analysis for powder of milled BS was carried out using BRUKER- D8 Advance X-ray diffractometer with Cu-Kα (λ=1.54Å) as a radiation source, operating voltage at 45 kV and current 40 mA. The pattern is recorded in the wide angle range from 4° to 80° with step 0.02°.

---

<table>
<thead>
<tr>
<th>Ingredients, phr</th>
<th>Bo</th>
<th>B1</th>
<th>B2</th>
<th>B3</th>
<th>B4</th>
<th>B5</th>
</tr>
</thead>
<tbody>
<tr>
<td>SBR</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Processing Oil</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Paraffin Wax</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Zinc Oxide</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Stearic Acid</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>MB</td>
<td>1.5</td>
<td>1.5</td>
<td>1.5</td>
<td>1.5</td>
<td>1.5</td>
<td>1.5</td>
</tr>
<tr>
<td>CBS</td>
<td>0.75</td>
<td>0.75</td>
<td>0.75</td>
<td>0.75</td>
<td>0.75</td>
<td>0.75</td>
</tr>
<tr>
<td>DPG</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>Sulfur</td>
<td>2.5</td>
<td>2.5</td>
<td>2.5</td>
<td>2.5</td>
<td>2.5</td>
<td>2.5</td>
</tr>
<tr>
<td>HAF-N 330</td>
<td>--</td>
<td>5</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Nanosilica (NS)</td>
<td>--</td>
<td>--</td>
<td>5</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Black Sand (BS-NPs)</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>5</td>
<td>10</td>
<td>15</td>
</tr>
</tbody>
</table>

*Egypt. J. Chem. 63, No. 6 (2020)*
**FT-IR Spectroscopy**

A Nicolet 380 thermo Fourier transform infrared (FITR) spectrometer with an attenuated total reflection (ATR) accessory and a zinc selenide crystal was used to analyze the BS-NPs sample. Scans were obtained with a resolution of 4 cm⁻¹.

**HR-TEM studies**

To estimate the particle size of BS-NPs, a sample was imaged by high resolution transmission electron microscope (HR-TEM, Jeol JEM 2100, Japan) operated at 200 Kev. The sample was prepared by dispersing it in methane. This dispersion is then dropped on carbon copper grids.

**Mechanical properties**

For measuring the mechanical properties of the rubber vulcanizates, sheets of dimensions 230 mm × 230 mm × 2.8 mm were prepared using a hydraulic press under a pressure of 150 kg/cm². Dumbbell shaped samples were cut from the molded sheets. Modulus, tensile strength and elongation at break were determined using a Zwick (Germany) tensile testing machine (Model Z010) at a temperature of 23 ± 2°C and a crosshead speed of 500 mm/min according to ASTM D412-16.

**Compression measurements**

The compression-recovery measurements were used to determine the compression and recovery percentage from which the crosslink density was evaluated.

**Compression-recovery measurements**

Cylindrical samples of dimension 22 mm diameter and 10 mm thickness were molded in the hydraulic press at the same conditions of temperature and time recorded before by the rheometer. A compression set device was used to accommodate all samples at the same time. The device consists of four steel plates between which the samples are compressed. The plates are held together by three axial bolts. Ring-shaped spacers of different thickness, 6.00, 7.614 and 9.228 mm, are placed around three axial bolts [14]. The different thicknesses (ls) of the spacers help to control the degree of compression. After placing the samples in between the steel plates, the bolts were tightened firmly over the samples. The compression set, including the samples, was accommodated in an air circulating oven at 70°C for 48 hours. After that, it is removed from the oven and allowed the whole set to cool. Then, the samples were released and allowed to recover for half an hour before measuring the final thickness accurately. The compression and recovery percentage was calculated using the following relations,

\[ \text{Compression} \% = \frac{l_0 - l_s}{l_0} \times 100 \]  
\[ \text{Recovery} \% = \frac{l_0 - l_r}{l_0 - l_s} \times 100 \]

where, \( l_0 \) is the sample initial thickness, \( l_r \) is the sample thickness after recovery and \( l_s \) is the spacer thickness.

**Crosslink density determinations**

The physical crosslink density of various styrene butadiene and nitrile rubber compounds was estimated using the Kinetic Theory of Elasticity [15, 16]. The theory relates the force \( F \) applied per unit area \( A \) required to strain a perfectly elastic network at a small extension ratio, \( \lambda \), by the following equations:

\[ \frac{F}{A_0} = \frac{\rho RT}{M_c} (\lambda - \lambda^{-2}) \quad \text{dyne.cm}^{-2} \]

\[ M_c = \frac{\rho RT A_0}{F} (\lambda - \lambda^{-2}) \quad \text{g/mole}^{-1} \]

where, \( \rho \) is the density of rubber, \( T \) is the absolute temperature, \( R \) is Boltzman’s constant, and \( M_c \) is molecular weight between two crosslinks. The crosslink density was expressed in terms of \( M_c \) as follows:

\[ \nu = \frac{1}{2M_c} \quad \text{g}^{-1}.\text{mol} \]

In the stress strain measurements, the extension ratio \( \lambda \) is defined as \( l/l_0 \), where \( l \) and \( l_0 \) are the specimen thickness before and after strain respectively. In this study the force applied on the sample \( F/A_0 \) was compression stress and was calculated as follows:

\[ \frac{F}{A_0} = \left( \text{compression} \% \times 100 \times 9.81 \right)/(\pi r^2) \]

where, \( r \) is the radius of the compressed sample. The values of \( F/A_0 \) obtained from the Equation 4, for each sample, were plotted versus the values of \( \lambda - \lambda^{-2} \), where \( \lambda \) here is defined as \( l_0/l \) instead of \( l/l_0 \). Results of these calculations are illustrated in Figure (1) where the slope of the straight line gives the value of \( \frac{\rho RT}{M_c} \). Thus the \( M_c \) values of each sample were calculated and subsequently the crosslink density by using Equation (5). It is to be noted here that there is a limit of 30% compression. For compression greater than 30% the...
values were far from linearity; in other words do not obey the statistical theory of elasticity.

**Hardness**

Hardness testing measurements were carried out according to ASTM D 2240-15. Hardness determination was made using a Zwick Hardness Tester 3150, Germany. Test specimens used were cylindrical in shape with thickness and diameter 6.00 ± 0.2 mm and 14.00 ± 0.2 mm respectively.

### Results and Discussions

**BS-NPs Characterization**

The size of the BS-NPs particles was examined by HR-TEM. Figure (2) shows that the BS-NPs particles are not uniform with agglomeration among them. Nanoparticles agglomeration usually happens to be due to the extreme small dimensions and high surface energy [17, 18].

![Image](image_url)

**Fig. 1. Relationship between force applied on SBR/filler nanocomposites versus λ-λ^2.**

Figure (3) represents particle size histogram, the nano-crystalline particles size of BS-NPs ranged from 6 to 122 nm and the mean diameter is about 31 nm.

Structural determination and estimation of crystallite size of the BS-NPs were characterized by XRD. The diffraction pattern is recorded in the wide angle range from 4° to 80° with step 0.02°, as shown in Figure (4). The XRD patterns were analyzed and indexed using ICDD (International Center for Diffraction Data) database. These data are given in Table (2). XRD examination demonstrates that the BS-NPs contain quartz (SiO\(_2\)), Albite (NaAlSi\(_3\)O\(_8\)) and baddeleyite (ZrO\(_2\)) in the ratio 17.7, 63.8 and 18.5 by weight respectively. Each of these minerals has its own crystalline structure. The variety in

*Egypt. J. Chem. 63, No. 6 (2020)*
the crystal structure is suggested to be a privilege for the BS-NPs to act as filler in the polymeric composites. This can be attributed to the fact that albite is a superior substitute for alumina and SiC is a reinforcing material in metal matrix composites [19, 20]. Moreover the monoclinic crystal lattice structure of baddeleyite has small grain size and non-spherical crystal shape yield high surface to volume ratios [21].

The FTIR measurements support the XRD results that demonstrate the existence of different minerals in BS-NPs. The FTIR spectrum is shown in Figure (5). The characteristic feature of quartz is the doublet appearing at 777 cm\(^{-1}\) and 795 cm\(^{-1}\) due to Si-O symmetrical stretching vibration. Moreover, the peaks at wavelength 459.6 cm\(^{-1}\) (Si-O asymmetrical bending vibrations), 694.4 cm\(^{-1}\) (Si-O symmetrical bending vibrations) are ascribed to existence of quartz in the samples [22]. The broad band at 3852.9 cm\(^{-1}\) and the peak placed at 1619.4 cm\(^{-1}\) referred to the H-O-H stretching and bending modes of the adsorbed water respectively, related to the silanol OH groups (20). The deformation vibrations of Al-O-Si and Si-O-Si are allocated in the region 530 cm\(^{-1}\) and 483 cm\(^{-1}\), respectively [23]. The broad band at 1080 cm\(^{-1}\) is probably due to Si-O-Si symmetrical vibration or to the Zirconyl bonds Zr = O (11). The observed absorption peak at about 470 cm\(^{-1}\) region is due to the Zr–O vibration, which confirms the formation of ZrO\(_2\) structure. However, weak peaks in the range 460 cm\(^{-1}\) to 690 cm\(^{-1}\) corresponding to monoclinic ZrO\(_2\) are observed. A similar observation was reported by Chen et al. [24] for zirconia nanoparticles.

**BS-NPs/ SBR Composites Properties**

The present study comprises of two criteria a) compare the effect of BS-NPs with other fillers, with different structure and particle sizes, on the SBR properties; b) study of the effect of different concentrations of BS-NPs on the SBR/ BS-NPs nanocomposites.

**Rheometric properties**

In order to study the influence of BS-NPs, as filler, on the vulcanization reaction of SBR
Fig. 3. Histogram of the particle size counts of BS-NPs.

Fig. 4. X-ray diffraction patterns of black sand nanoparticles (BS-NPs).

**TABLE 2. Composition of BS-NPs collected from XRD examination.**

<table>
<thead>
<tr>
<th>Mineral name</th>
<th>Chemical structure</th>
<th>Crystallographic phase</th>
<th>Wt, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Albite (sodium aluminum silicate)</td>
<td>NaAlSi$_3$O$_8$</td>
<td>Triclinic</td>
<td>63.8</td>
</tr>
<tr>
<td>Quartz (silicon dioxide)</td>
<td>SiO$_2$</td>
<td>Hexagonal</td>
<td>17.7</td>
</tr>
<tr>
<td>Baddeleyite (Zirconium oxide)</td>
<td>ZrO$_2$</td>
<td>Monoclinic</td>
<td>18.5</td>
</tr>
</tbody>
</table>

_Egypt. J. Chem._ 63, No. 6 (2020)
compounds, it was interesting to compare the effect of other fillers such as high abrasion furnace black (HAF) and nanosilica (NS) on the SBR composites. The vulcanization curves of samples obtained from oscillating disc rheometer (ODR) were analysed. The rheometric properties of the SBR/nanofillers nanocomposites are given in Table [3]. The results show a decrease of the scorch time ($t_s$) value of BR/BS-NPs nanocomposites relative to that of SBR/HAF and SBR/NS nanocomposites. Further decrease in the $t_s$ values is observed as the BS-NPs loading increase in the SBR matrix (B4&B5) which indicate a lower scorch safety. The optimum cure time ($t_c$) defined as the time required to reach 90% of maximum cure. Values of SBR/BS-NPs composites are less than that of SBR/NS and SBR/HAF, at the same filler loadings. As the BS-NPs loadings increase in the composites to 10 and 15 phr, the $t_c$ values decrease indicating that BS-NPs are able to accelerate the vulcanization process of SBR composites. This can be referred to the existence of metal ions such as Zr, Na, Al and Si in the BS-NPs powder which accelerates the curing reaction. The cure rate index (CRI) is a parameter indicates the speed of the curing reaction [16]. At equivalent concentrations of HAF, NS and BS-NPs, samples B1, B2 and B3, the SBR/BS-NPs manifest the highest CRI value. As the BS-NPs loading in the SBR composites increase to 10 and 15 phr, samples B4 and B5, the CRI values increase. This behaviour was reported before to fillers with small surface area that usually speed up the rate of rubber composites curing reaction. It is worthy to mention that Hosseini et al. in a similar study, studied the effect of adding different NSPs loading on SBR nanocomposites in presence of silane coupling agent. Rheological parameters reported by him were much low compared to those obtained in our study specilaly the values of $M_t$ and DM [25]. to These results ensure the outfindings of the morphological studies mentioned previously in section 3.1. The minimum torque ($M_t$) value represents an index of the material viscosity [26]. From Table [3] we can notice that at equivalent filler concentration of
fillers, the $M_c$ value of SBR/BS-NPs is lower than that of SBR/HAF and SBR/NS. This decrease in the ML value means a decrease in the composites viscosity and in turns an increase in the molecular mobility [26]. This can explain the low $T_{c90}$ and CRI values of the SBR/BS-NPs composites. The extent of cure (given by $\Delta M$ values) decreases in the order B1>B2>B3 containing 5 phr of HAF, NS and BS-NPs respectively. Lower $\Delta M$ values was expected for B2 and B3 due to the interaction of the silanol group (–OH), on the silica particles surface, with the accelerator molecules [27]. This deactivating effect of the –OH group leads to a lower $\Delta M$ values in comparison to carbon black filled composites (B1). However the reduction of $\Delta M$ values of SBR/BS-NPs composites is not dramatic and can be overcome by treating the BS-NPs surface in our future work. 

Analysis of the mechanical properties

Table (4) lists the mechanical properties of the SBR nanocomposites. The results of the tensile tests are expressed in terms of the elastic modulus, maximum tensile strength (T.S) and elongation at break percentage ($E\%$). Firstly, we will discuss the effect of 5 phr concentration of HAF, NS and BS-NPs as represented by B1, B2 and B3 respectively. From Table (4) it is clear that B1 showed the highest T.S and elastic modulus values. This result was expected as it was previously reported that nanosilica causes a reduction in the tensile properties when compared to carbon black fillers [28]. This was attributed to the presence of agglomerated nanoparticles that has the tendency for splitting [29]. It is worthy to mention that T.S of SBR/BS-NPs nanocomposite is higher than that of SBR/NS. This result indicates that the degree of dispersion, as well as good interaction in the SBR matrix, is higher for BS-NPs than NS. Another interesting observation is that the lower elastic modulus value of B3 which indicating lower stiffness than B1 and B2 nanocomposites. Meanwhile, B3 possess the highest elongation value among B1 and B2. This can be attributed to the reactivity of the BS-NPs due to the existence of quartz ($\text{SiO}_2$), Albite ($\text{NaAlSi}_3\text{O}_8$) and Baddeleyite ($\text{ZrO}_2$) nanoparticles in it, which can lead to more rubber-filler interaction than filler-filler interaction. Thus the BS-NPs agglomerations do not impede the higher relative deformation of the rubbery matrix. This postulate is evidenced by the highest hardness value exhibited by B3, as hardness value evaluate the resistance of a rubber surface against penetration by an indenter, and relates to the deformation of the rubber surface [30].

The crosslink density

In previous studies, Lawandy et al. [14,15] had successfully used the stress-strain measurements, compression percentage % measurements and ultrasonic measurements to evaluate the crosslink density of rubber compounds. These evaluations were based on the statistical theory of rubber like elasticity[11, 23]. The theory relates the force applied (F) per unit area ($A_u$) required to strain a perfectly elastic network at a small extension ratio. The compression stress is a reversible process to that of elongation strain. The force applied expressed as $F/A_u$ for each sample was calculated using Equation (5). Then the calculated $F/A_u$ was plotted versus $\lambda-\lambda^2$. Consistent linear relations were obtained as shown in Figure (1). The slopes of these straight lines represent $K$ value of the samples. Substituting by the values of $M_c$ in Equation (5), the crosslink density of the SBR nanocomposites were obtained, as shown in Figure (6). The experimental procedure and the crosslink density calculations are fully explained above in section 2.4.7. Figure (6) illustrates the crosslink density of the samples containing 5, 10 and 15 phr of BS-NPs together with the control sample and the sample containing 5 phr NS. A dramatic decrease in the crosslink density of rubber vulcanizates is noticed upon adding the nanofillers. Similar behaviour was interpreted before on the basis of the relation between the bound rubber and the crosslink density within the rubber vulcanizates [25]. Bound rubber in rubber vulcanizates is a reinforcement level which is formed by physical bonding between filler and rubber molecules; whereas crosslink density is a level of reinforcement formed during vulcanization between rubber backbone and sulfur, as vulcanizing agent [31,32]. Thus, the decrease in the crosslink density of the SBR rubber composites along with enhancement of mechanical properties, especially elongation at break, indicates a predomination of bound rubber portion. In other words, the active sites on the filler nanoparticles surface compete with the vulcanizing system bonding ability. Another interesting observation, from Figure (6), is that the crosslink density of SBR composites containing 5phr of NS and BS-NPs, samples B3 and B4, are very close to each other. Meanwhile, as the BS-NPs concentration increases in the SBR compounds, samples B4 and B5, the crosslink density slightly decreases.

Conclusion

Black sand (BS) samples were collected from
TABLE 3. Vulcanization characteristics obtained from the rheometer.

<table>
<thead>
<tr>
<th>Property</th>
<th>Sample</th>
<th>Bo</th>
<th>B1</th>
<th>B2</th>
<th>B3</th>
<th>B4</th>
<th>B5</th>
</tr>
</thead>
<tbody>
<tr>
<td>M_1, Kg.cm</td>
<td>SBR/HAF</td>
<td>0.37</td>
<td>0.56</td>
<td>0.59</td>
<td>0.52</td>
<td>0.43</td>
<td>0.44</td>
</tr>
<tr>
<td>M_2, Kg.cm</td>
<td>SBR/NS</td>
<td>5.69</td>
<td>7.91</td>
<td>7.13</td>
<td>6.80</td>
<td>7.06</td>
<td>7.14</td>
</tr>
<tr>
<td>D M, Kg.m.cm</td>
<td>SBR/BS-NPs</td>
<td>5.32</td>
<td>7.35</td>
<td>6.54</td>
<td>6.28</td>
<td>6.63</td>
<td>6.70</td>
</tr>
<tr>
<td>Scorch time Tc*, min</td>
<td>SBR/BS-NPs</td>
<td>2.20</td>
<td>3.37</td>
<td>3.06</td>
<td>1.52</td>
<td>1.48</td>
<td>1.29</td>
</tr>
<tr>
<td>Cure time Tc90, min</td>
<td>SBR/BS-NPs</td>
<td>8.52</td>
<td>9.39</td>
<td>10.18</td>
<td>6.37</td>
<td>5.26</td>
<td>5.09</td>
</tr>
<tr>
<td>Cure Rate Index, min^{-1}</td>
<td>SBR/BS-NPs</td>
<td>15.82</td>
<td>16.61</td>
<td>14.04</td>
<td>20.62</td>
<td>26.46</td>
<td>26.32</td>
</tr>
</tbody>
</table>

TABLE 4. Mechanical Properties of the different SBR/filler nanocomposites.

<table>
<thead>
<tr>
<th>Nanocomposite</th>
<th>Tensile Strength, MPa</th>
<th>Modulus, MPa</th>
<th>Elongation at break, %</th>
<th>Hardness, Shore A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bo SBR</td>
<td>2.04</td>
<td>2.03</td>
<td>335</td>
<td>45.7</td>
</tr>
<tr>
<td>B1 SBR/HAF (5phr)</td>
<td>2.74</td>
<td>2.28</td>
<td>416</td>
<td>44.6</td>
</tr>
<tr>
<td>B2 SBR/NS(5phr)</td>
<td>2.32</td>
<td>2.18</td>
<td>421</td>
<td>45.3</td>
</tr>
<tr>
<td>B3 SBR/ BS-NPs (5phr)</td>
<td>2.57</td>
<td>2</td>
<td>426</td>
<td>45.7</td>
</tr>
<tr>
<td>B4 SBR/ BS-NPs (10phr)</td>
<td>1.95</td>
<td>1.95</td>
<td>454</td>
<td>44.4</td>
</tr>
<tr>
<td>B5 SBR/ BS-NPs (15phr)</td>
<td>1.84</td>
<td>1.97</td>
<td>377</td>
<td>44.3</td>
</tr>
</tbody>
</table>

Fig. 6. The crosslink density of SBR control sample, SBR/NS and SBR/BS-NPs nanocomposites containing 5, 10 and 15 phr BS-NPs.
the beach area of Rosetta in Egypt and grinded in a ball milling machine, an energy saving and echo-friendly technique, to obtain black sand nanoparticles (BS-NPs). The TEM examination revealed that the milled BS has an average particle size 31nm. The FTIR and XRD examination showed that Black Sand nanoparticles BS-NPs contain quartz (SiO$_2$), Albite (NaAlSi$_3$O$_8$) and baddeleyite (ZrO$_2$) in the ratio 17.7, 63.8 and 18.5 by weight respectively. Results evidenced that the incorporation of BS-NPs in the rubber compounds accelerate the vulcanization reaction as revealed by the lower cure time and higher CRI values relative to that of SBR/NS and SBR/HAF. Also SBR/BS-NPs nanocomposites, even at high concentration, possess lower E% values along with higher E% values. The T.S of SBR/5phr BS-NPs was higher than that of SBR/NS nanocomposites. The study of the crosslink density values of SBR/NS and SBR/BS-NPs nanocomposites shed light on the type of bonds in the SBR/BS-NPs. The decrease in the crosslink density values of the SBR/BS-NPs, relative to that of SBR/HAF and SBR/NS nanocomposites, without sacrificing E% values evidenced a higher adhesion of the rubber matrix to the BS-NPs surface.

References


الاستقادة من جزيئات الرمل الأسود النانومترية كمادة مالئة لمتراكبات مطاط إستيرين

سيم نجيب لاوندي، رانيا سيد أحمد، سمير نجيب لاوندي

يتواجد الرمل الأسود بكثرة على شواطئ الروزيتا في جمهورية مصر العربية، وقد تم الحصول عليه من هذه الشواطئ وطحنها للحصول على جزيئات الرمل الأسود النانومترية. 

إن هذه الجزيئات تتكون أساساً من الأبيت (سيليكات الألومنيوم) وتوصيفها بالأحدث تقنيات مثل XRD، TEM. أظهرت نتائج XRD أن نسبة جزيئات الرمل النانومترية تتراوح بين 6% إلى 31%، بينما تصل نسبة ثاني أكسيد الأمونيوم وأكسيد الزركونيوم إلى 18،7% من الترتيب.

وارتفع حجم جزيئات الرمل الأسود النانومترية إلى 17،7 نانومتر. وقد تم إضافة 1.25% من الغازات إلى خلطات مطاط إستيرين البيوتيلين ودرجة الخواص الروبوتية والميكانيكية للمتراكبات الناتجة. وقد أوضحت النتائج تقارب نتائج القياسات الميكانيكية للمتراكبات الرمل الأسود النانومترية مثيلاتها المحضر وسابل الكربون والسيليكا النانومترية. وقد أوضحت النتائج تقارب نتائج القياسات الميكانيكية للمتراكبات الرمل الأسود النانومترية.

س. ن. لواندي et al.

Academiae Patavini

Egypt. J. Chem. 63, No. 6 (2020)