

Mechanical and electrical properties of acrylonitrile-butadiene rubber filled with crosslinked polystyrene prepared by emulsion polymerization

Nehad N. Rozik^{1, *}, Sabrinal H. El-Hamouly²⁾, Emad S. Shafik¹⁾, Adel F. Younan¹⁾, Salwa L. Abd-El Messieh³⁾

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Abstract: In the present work, we have studied the effects of mixed emulsifier systems, sodium dodecyl sulphate (SDS — as anionic surfactant) and poly(oxyethylene) nonylphenyl ether (NP-30 — as non-ionic surfactant), on the emulsion polymerization of styrene. The crosslinked, monodispersed polystyrene (PS) particles were synthesized with a variable divinylbenzene (DVB) concentration as cross-linking agent. The crosslinked polystyrene was incorporated into acrylonitrile-butadiene rubber (NBR). The mechanical and electrical properties were determined using carbon black as a conductive filler and silica as non conductive filler.

Keywords: acrylonitrile-butadiene rubber, emulsion polymerization, crosslinked polystyrene, mechanical and electrical properties NBR composites.

Mechaniczne i elektryczne właściwości kauczuku akrylonitrylo-butadienowego napełnionego usieciowanym polistyrenem otrzymanym w procesie polimeryzacji emulsyjnej

Streszczenie: Badano wpływ anionowych (SDS) i niejonowych (NP-30) środków powierzchniowo czynnych na przebieg polimeryzacji emulsyjnej styrenu. Syntezowany monodispersyjny polistyren (PS) sieciowano przy użyciu różnej ilości diwinylobenzenu (DVB). Cząstki usieciowanego PS wprowadzono do kauczuku akrylonitrylo-butadienowego (NBR). Wyznaczano właściwości mechaniczne wytworzonych kompozytów, a także porównywano właściwości elektryczne kompozytów zawierających dodatkowo napełniacz przewodzący w postaci cząstek sadzy lub — nieprzewodzący — w postaci cząstek krzemionki.

Słowa kluczowe: kauczuk akrylonitrylo-butadienowy, polimeryzacja emulsyjna, usieciowany polistyren, mechaniczne i elektryczne właściwości kompozytów NBR.

Polymer nanomaterials have gained attention in many applications such as adsorbents [1–3], fillers in polymers [4], calibration standards [5], diagnostics [6], entrapment and delivery of drugs and genes [7–10] and reaction catalysis [11]. Due to their simplicity and the wide range of monomers that can be successfully polymerized, the common mechanism for preparing these particles is emulsion polymerization [12–15].

In emulsion polymerization, the emulsifier plays two important roles in the formation and stabilization of the polymer particles. Anionic emulsifiers, such as sodium dodecyl sulfate (SDS), are widely used in industrial emulsion polymerization. However, polymer latex produced by emulsion polymerization with anionic emulsifiers are not stable enough against the addition of pigments. To increase the colloidal stability of the latex, a mixture of non-ionic emulsifiers, which stabilize the polymer particles by thermodynamically favored steric repulsions, and anionic emulsifiers, which stabilize particles through electrostatic repulsions, are often used in emulsion polymerization [16].

Polystyrene (PS) is one of the most widely used polymers and its synthesis in the form of nanoparticles is considered a model system for developing polymer nanotechnology. Monodispersed PS nanoparticles can be achieved by emulsion polymerization. An elastomer is a polymer with viscoelasticity, generally having low

¹⁾ Polymers and Pigments Department National Research Center, Dokki, Cairo, Egypt.

²⁾ Menoufia University, Faculty of Science, Department of Polymer Chemistry, Egypt.

³⁾ National Research Center, Microwave Physics and Dielectrics Department, Al-Bohoos St., Dokki Post Number 12 622, Dokki-Giza, Egypt.

^{*} Author for correspondence; e-mail: nrozeik@yahoo.com

Young' modulus and high strain compared with other materials.

In most applications of elastomers, the elastomeric matrix requires reinforcement before utilization because of its weakness in modulus and strength. As this case stands, great numbers of rubber products are actually composites made of an elastomeric matrix, fillers and some other curatives as well. And the filler, especially the reinforcing filler, plays an important role in determining the ultimate performance of the filled rubber compounds.

The so-called reinforced elastomers exhibit specific properties such as precocious, non-linear elastic response or strong restrictions to swelling. The numerous studies devoted to filled elastomers have shown that their behavior is essentially controlled by two structural parameters (i) the particle/matrix interface and (ii) the dispersion state of the fillers [17, 18].

Polymeric fillers may open the door to the development of some novel elastomer reinforcing agents. The polymeric fillers can be designed and synthesized as the designer's intention [19].

Our work was carried out in an attempt to provide a commercially viable method to prepare nanofillers for elastomers to improve their mechanical and electrical properties. Styrene was chosen as the monomer due to its' easy access. In this work, the preparation of crosslinked, mono-dispersed polystyrene (PS) particles was carried out under the variation of divinylbenzene (DVB) concentration as crosslinking agent. Carbon black and silica are used in this paper for their mechanical and electrical properties.

EXPERIMENTAL PART

Materials

– Styrene monomer (Fisher Co.) was purified by passing the monomer through an alumina-charged column.

– Potassium peroxydisulfate ($K_2S_2O_8$, Merck) was used as an initiator without further purification.

– Sodium dodecyl sulfate (SDS, ACROS) and nonylphenol ethoxylate with an average of 30 oxyethylene units per molecule (NP-30, Dow Co.) were used as emulsifiers.

– As the raw elastomer, the acrylonitrile butadiene copolymer (NBR, Lanxess AG, Germany) with an acrylonitrile content of 32 wt % was used.

– Technical-grade divinylbenzene (DVB) was used as the crosslinker (laboratory grade, 70–85 %) stabilized with 0.2 wt % 4-*tert*-butylcatechol (Sigma Aldrich, Germany). The DVB was purified with 10 wt % sodium hydroxide three times and distilled water before use.

– Sodium bicarbonate ($NaHCO_3$) was used as a buffer solution, distilled water was used as a polymerization medium.

– Dicumyl peroxide (DCP) was obtained from Aldrich-Sigma (Germany).

– All the rubber ingredients: zinc oxide, stearic acid, dioctyl phthalate (DOP) and tetramethylthiuram disulfide (TMTD) were of commercial grades, *N*-cyclohexyl-2-benzothiazole sulphenamide (CBS), pale gray powder, with specific gravity of 1.27–1.31 at room temperature (25 ± 1 °C), melting point 95–100 °C. Polymerized 2,2,4-trimethyl 1,2-dihydro-quinoline (TMQ), tetramethylthiuram disulfide in addition to sulfur were obtained from the Aldrich Company, Germany.

– Carbon black (HAF) and Silica which used as black and white fillers supplied by Transport and Engineering Company, Alexandria, Egypt.

Synthesis of polystyrene

The polymerization was carried out in a 250 cm³ three neck round-bottom flask with stirring of 200 rpm at 80 °C, the flask was first charged with appropriate emulsifiers SDS/NP-30 at different ratios and distilled water. Stirring at 200 rpm was performed for 30 min's until an evenly blended mixture was obtained. The batch was then charged with styrene monomer. Following another 30 mins emulsification, the batch was heated to 80 °C. When the temperature stabilized, polymerization was initiated by adding an aqueous $K_2S_2O_8$ solution. Aqueous $NaHCO_3$ solution was added in order to adjust the pH value of the latex equal to 7. Three hours later, the emulsion polymerization reaction was completed and the polystyrene latex was obtained.

The crosslinked polystyrene was prepared by the same method using DVB at the given ratios as the crosslinking agent.

The formula of the emulsion polymerization employed 5 cm³ styrene, 0.05 g $K_2S_2O_8$, 0.025 g $NaHCO_3$ and 62.5 cm³ distilled water with different ratios of anionic and nonionic emulsifiers.

Preparation of the NBR/PS composites

The crosslinked polystyrene containing 20 wt % DVB and non crosslinked polystyrene at emulsifier ratio (20 wt % SDS: 80 wt % NP-30) were selected due to their high monomer conversion and suitable particle size and mixed with NBR and vulcanized by 3 phr (3 parts per hundred parts of rubber) dicumyl peroxide.

From these results, crosslinked polystyrene was selected to fill NBR in two different ratios, namely: 5 phr using different ratios of carbon black (HAF) as a conducting filler, and 15 phr using different ratios of silica as an insulating filler. The rubber formulations are shown in Table 1.

The mixes were vulcanized at their optimum curing time.

Methods of testing

– The FT-IR spectra of samples were obtained using a Jasco (Japan) FTIR 430 series infrared spectrophotometer equipped with KBr discs.

Table 1. Formulation of NBR/PS composition using carbon black and silica (phr)

Ingredients				
NBR	100	100	100	100
Crosslinked PS	0	5	0	15
HAF	0, 5, 10, 15, 20, 30, 40, 50			
Silica	-----		0, 10, 20, 30, 40	
ZnO	3	3	3	3
Stearic acid	1.5	1.5	1.5	1.5
DOP	3	3	3	3
CBS	0.8	0.8	0.8	0.8
TMTD	1	1	1	1
Sulfur	1.5	1.5	1.5	1.5
TMQ	1	1	1	1

— Polystyrene nanoparticles were examined by Transmission Electron Microscopy (TEM) with a JEOL JX 1230 technique and microanalyzer electron probe. This technique was used to determine the particle size of the investigated fillers.

— Monomer conversions were measured by a gravimetric method. Small amounts of latex samples were taken by syringing and injecting into a weighing dish containing aqueous hydroquinone solution. After air-drying to remove the unreacted monomer, the samples were finally dried in an oven to a constant weight at 60 °C.

— The tensile stress σ_R was determined at room temperature on an Electronic Universal Tensile Testing Machine Zwick 1425, Germany, according to ASTM D 412-98a:1998.

— Dielectric measurements were carried out in the frequency range 100 Hz up to 100 kHz using an LCR meter type AG-411 B (Ando electric Ltd. Japan). The capacitance C , the loss tangent $\tan \delta$ and the resistance R were obtained from which the permittivity ϵ' , the dielectric

loss ϵ'' and the electrical conductivity σ were calculated. The samples were molded in the form of discs 5 cm diameter and 3 mm thickness. A guard ring capacitor (type NFM/5T Wiss Tech. Werkstätten GMBH, Germany) was used as a measuring cell. The cell was calibrated with standard materials, and the experimental errors in ϵ' and ϵ'' were found to be ± 3 and ± 5 %, respectively.

— Scanning Electron Microscope (SEM) model JSM T20 (JEOL, Japan) was used to characterize the blend morphology. SEM was performed by mounting the polymer blend samples on a standard specimen tube, and then depositing a very thin layer of gold on the samples.

RESULTS AND DISCUSSION

Synthesis of PS

The obtained conversion-time curves are shown in Fig. 1. For all systems, conversion increases almost linearly with time over a conversion range from 10 % to 30–40 %. Figure 1a shows the effect of a mixture of anionic and nonionic emulsifiers on the overall conversions of emulsion polymerization of styrene. As can be observed, an increasing proportion of anionic emulsifier (SDS) led to a value of maximum conversion much higher than that obtained using nonionic emulsifier alone (NP-30).

The anionic emulsifier (SDS) is extensively preferred in many emulsion polymerization systems. SDS serves as a strong particle generator by stabilizing the latex particles *via* an electrostatic repulsion mechanism. However, latexes stabilized with this type of emulsifier are often unstable upon addition of electrolytes and in freeze-thaw cycles. Furthermore, this emulsifier has a limited stabilizing effectiveness at high solids (*e.g.* > 40 wt %) and high water sensitivity. To overcome these problems, mixtures of anionic and nonionic emulsifiers have been widely used together as shown in Fig. 1a. The mixed emulsifier system can greatly improve the latex stability *via* syner-

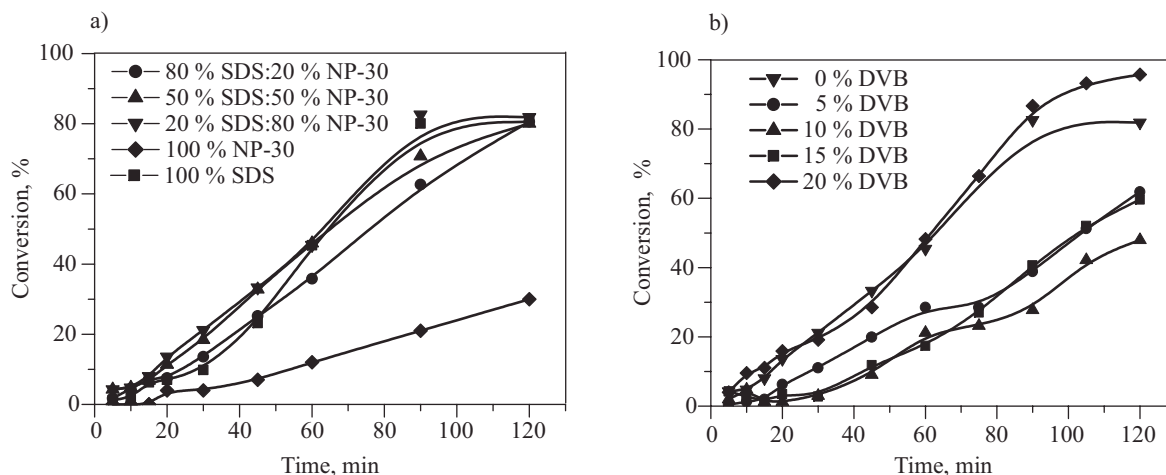


Fig. 1. Conversion-time curves for emulsion polymerization of styrene at 80 °C: a) using different ratios of emulsifiers, b) at emulsifier ratio (20 % SDS: 80 % NP-30) with DVB as a crosslinking agent

gistic effects provided by both the electrostatic and steric stabilization mechanisms, and thereby reduce the degree of the limited flocculation process [16]. From Fig. 1a we observed that the mixed emulsifier system (SDS:NP-30 = 20:80) gives the highest monomer conversion (81.85 %) and the highest degree of emulsion stability in comparison with other series.

Figure 1b shows the effect of DVB as a crosslinking agent on the overall conversions of emulsion polymerization of styrene. It is clear that the crosslinked polystyrene at an emulsifier ratio (SDS:NP-30 = 20:80) containing 20 wt % DVB gave a high monomer conversion (95.75 %) – more than other ratios of DVB.

Fourier transform infrared analysis of the polystyrene

Figure 2 illustrates the infrared absorption spectra of the non crosslinked polystyrene spheres. There are several absorption peaks within the involved wave number range. There are absorption bands at wave numbers 3059.51 and 3025.76 cm^{-1} due to aromatic C-H stretching vibrations and there are three absorption bands at wave numbers 1600.63, 1491.67, and 1449.24 cm^{-1} due to aromatic C=C stretching vibrations. These absorption bands indicate the existence of benzene rings. The absorption bands at wave numbers 755.959 and 698.105 cm^{-1} correspond to C-H out-of-plane bending vibrations and indicate that there is only one substituent in the benzene ring. Figure 2 also shows the absorption bands at wave numbers 2921.63 and 2851.24 cm^{-1} , corresponding to the existence of methylene groups.

These IR results have confirmed that the styrene reacts to produce polystyrene through a polymerization reaction. In addition, the absorption bands at wave number 3413.39 cm^{-1} is for the stretching vibration absorption of O-H, which indicates the existence of hydroxyl. The hydroxyl may come from water or hydrolysis of a strong acid, weak alkali salt such as sodium bicarbonate.

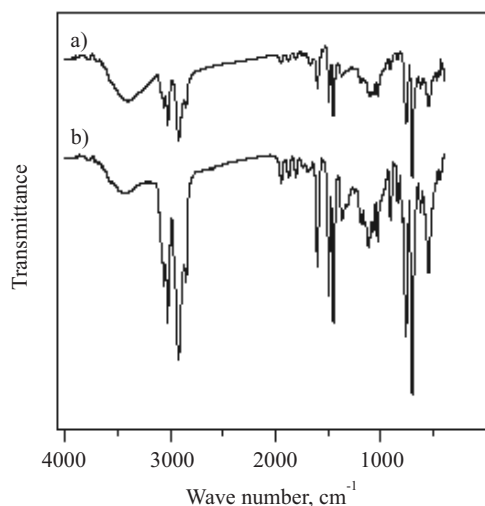


Fig. 2. FT-IR spectrum for: a) non crosslinked PS, b) crosslinked PS

In the case of the IR spectra of crosslinked PS there was a slight difference. This is because the crosslinked PS contains the same functional group of non crosslinked PS plus the functional group of DVB. From this figure we observed that the intensity of the bands increased and this confirmed the occurrence of crosslinking.

Morphology and particle size of the polystyrene

The TEM images of non crosslinked polystyrene synthesized by emulsion polymerization using different ratios of anionic and nonionic emulsifiers are shown in Fig. 3. From this figure it is clear that the particles are spherical and monodisperse up to around 170 nm and the particle size of PS using mixed emulsifiers was smaller and more monodisperse than the particle size of PS using a single emulsifier.

Figure 4 illustrates the micrographs of crosslinked polystyrene using different ratios of DVB. From this figure it is clear that the crosslinked polystyrene takes a spherical shape with a monodisperse distribution and particle size between 75 nm and 160 nm. We observed a larger diameter with increasing DVB concentrations until 15 wt % DVB. Above this ratio, about 20 wt %, the particle size will decrease [20].

Mechanical and dielectrical properties of the NBR/PS composites

Figure 5 shows the relation between tensile strength of NBR/PS composites and PS content. From this figure it is clear that when PS content increases, the tensile strength increases but in the case of crosslinked polystyrene there was a more significant increase in tensile strength than in the case of non crosslinked polystyrene.

The obtained data of the permittivity ϵ' and dielectric loss ϵ'' for NBR with different content of non crosslinked PS and crosslinked PS are illustrated in Fig. 6.

The calculated values of ϵ' decrease over the whole investigated series of samples as a function of higher frequency, which shows anomalous dispersion. The rotational motion of the polar molecules in the dielectric medium is not sufficiently rapid to attain equilibrium with the field [21].

This behavior is expected in most polymer dielectrics and is due to the dielectric relaxation phenomena of the polymer materials. Within the measured frequency range, the dielectric relaxation involves the dipolar (rotational) polarization, which depends on the molecular structure of the material. At higher frequencies, the rotational motion of the molecules lag behind the electric field, leading to reduced permittivity at higher frequency [22]. Also, it is seen from the same figure that ϵ' decreases with higher PS contents.

It is also apparent from Fig. 6, which presents the variation in ϵ'' with the applied frequency, that ϵ'' decreases with higher PS contents. The dielectric loss values at

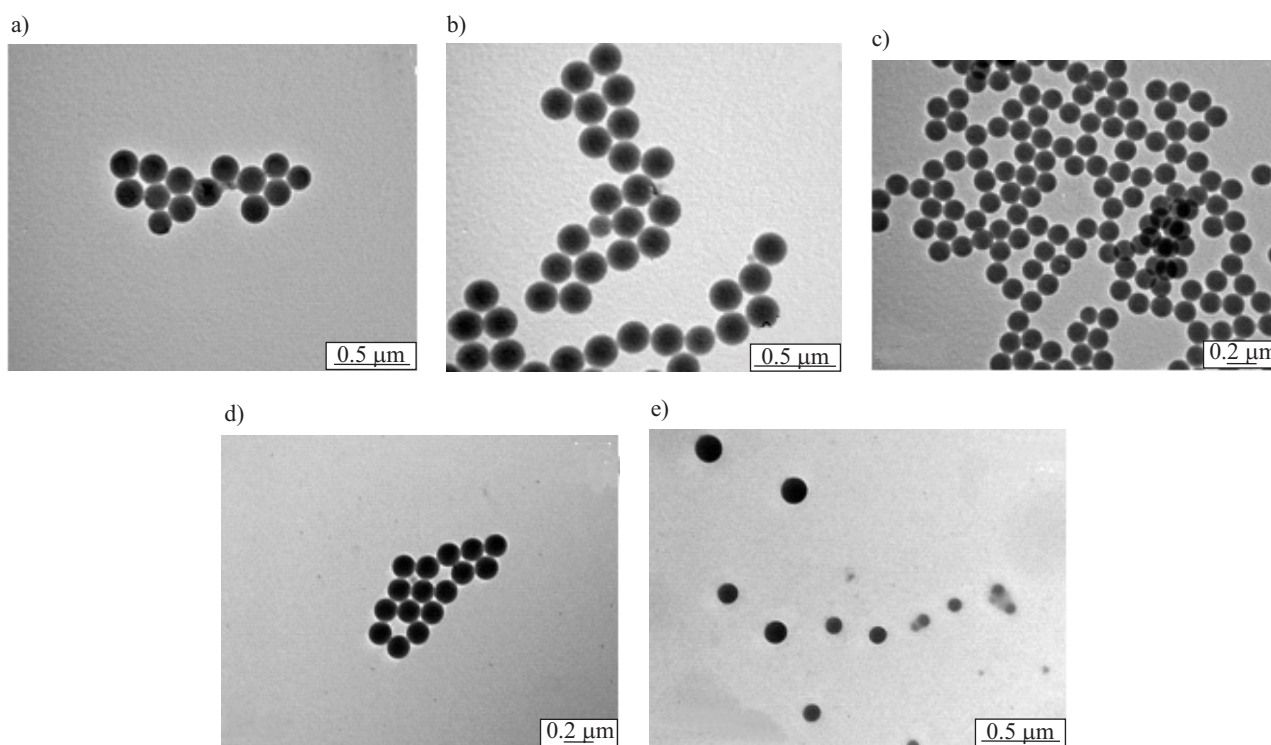


Fig. 3. TEM images of polystyrene emulsion using different ratio of emulsifiers: a) 100 % SDS, b) 80 % SDS:20 % NP-30, c) 50 % SDS:50 % NP-30, d) 20 % SDS:80 % NP-30, e) 100 % NP-30

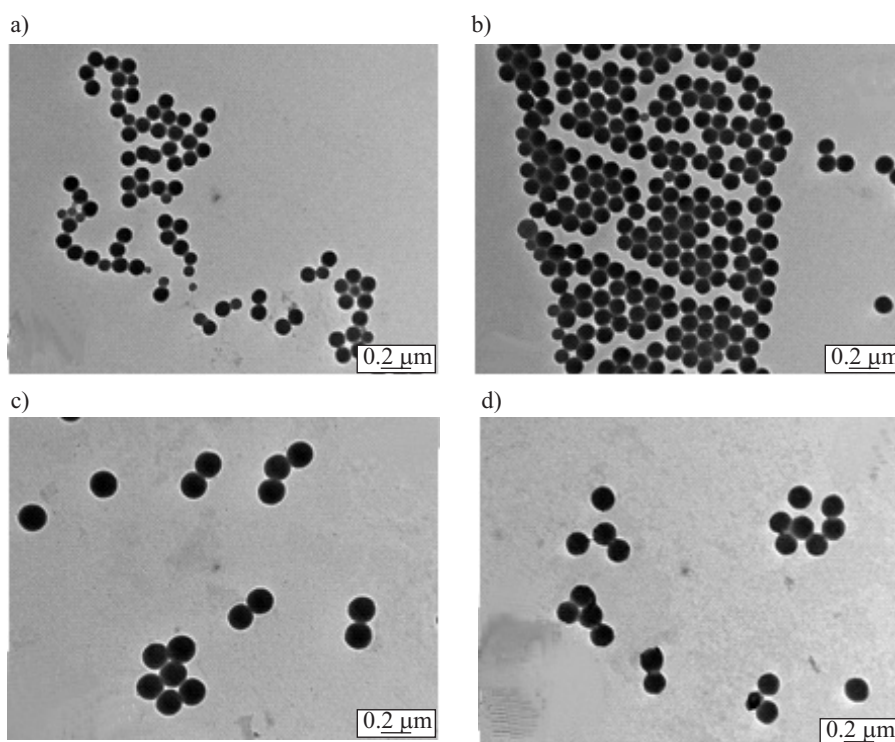


Fig. 4 TEM images of crosslinked polystyrene using (20 % SDS:80 % NP-30): a) 5 % DVB, b) 10 % DVB, c) 15 % DVB, d) 20 % DVB

low-frequency are found to be high due to the fact that not all losses are due to direct current (dc) losses [23]. These losses are also result from increased ion mobility and include Maxwell–Wagner losses [24] resulting from an alternating current (ac) in phase with the applied potential, because the differences between the permittivity

and conductivity of the different ingredients in the investigated samples are relatively large [25].

The permittivity ϵ' and dielectric loss ϵ'' of the NBR/PS composites as a function of crosslinked and non crosslinked PS content, at fixed frequency $f = 100$ Hz, are illustrated graphically in Fig. 7. From this figure it is seen that

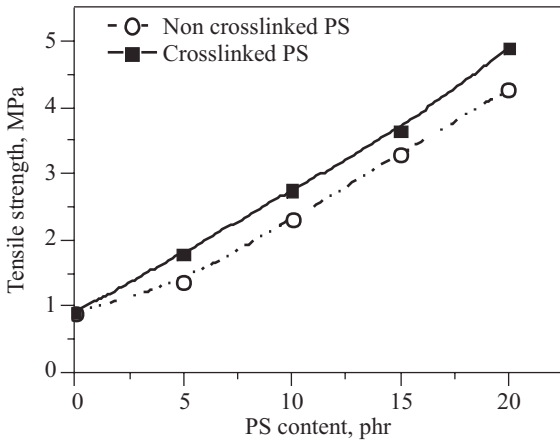


Fig. 5. Tensile strength of the NBR/PS composites versus PS content

there is a slight decrease in both values for non crosslinked PS compared to crosslinked PS. Also, it is clear that 5 phr PS slightly increases the permittivity while 15 phr PS decreases the permittivity.

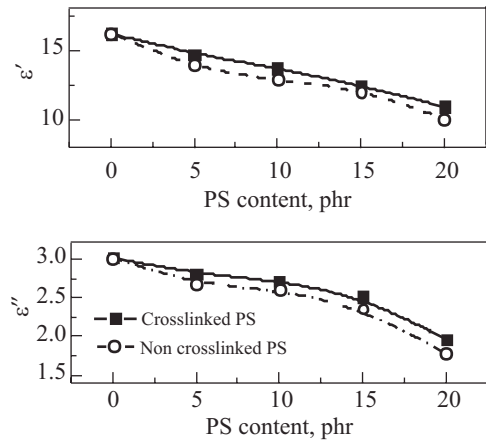


Fig. 7. The permittivity ϵ' and dielectric loss ϵ'' of the NBR/PS composites at fixed frequency $f = 100$ Hz versus PS content

SEM analysis

The SEM micrographs of the NBR and NBR containing 5 and 15 phr of crosslinked PS are shown in Fig. 8.

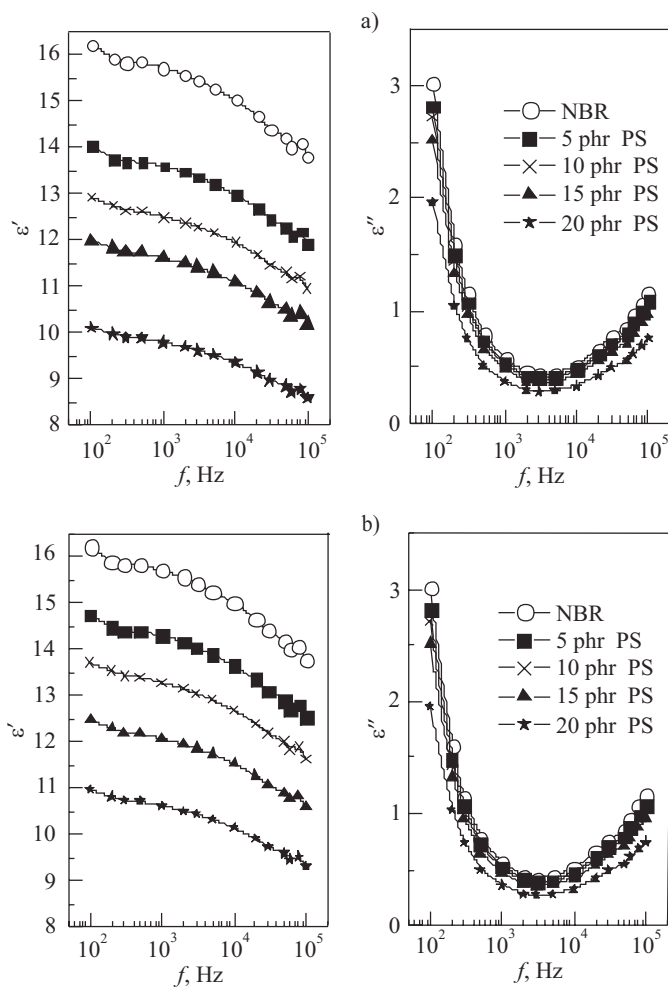


Fig. 6. The permittivity ϵ' and dielectric loss ϵ'' versus the applied frequency (f) for NBR with different contents of: a) non crosslinked PS, b) crosslinked PS

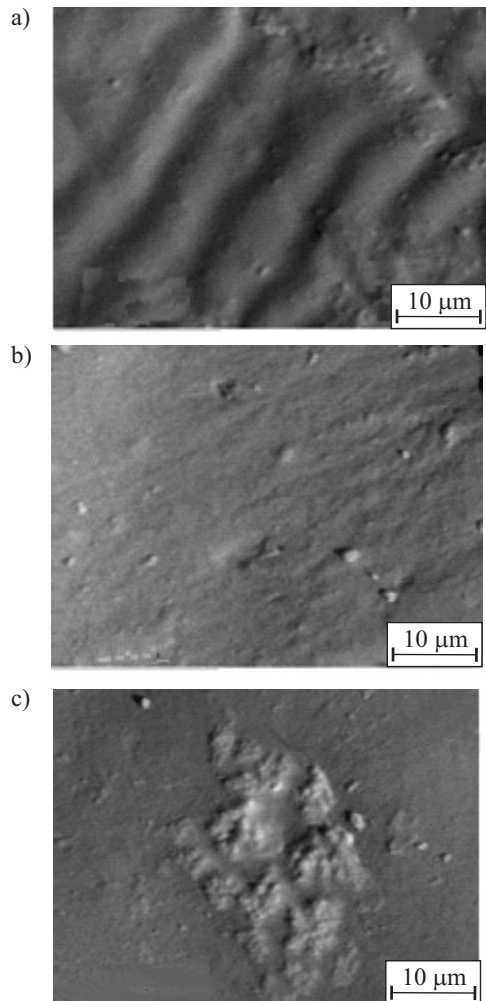


Fig. 8. SEM images of: a) NBR, b) NBR + 5 phr crosslinked PS, c) NBR + 15 phr crosslinked PS (cured by DCP) with magnification 2000 \times

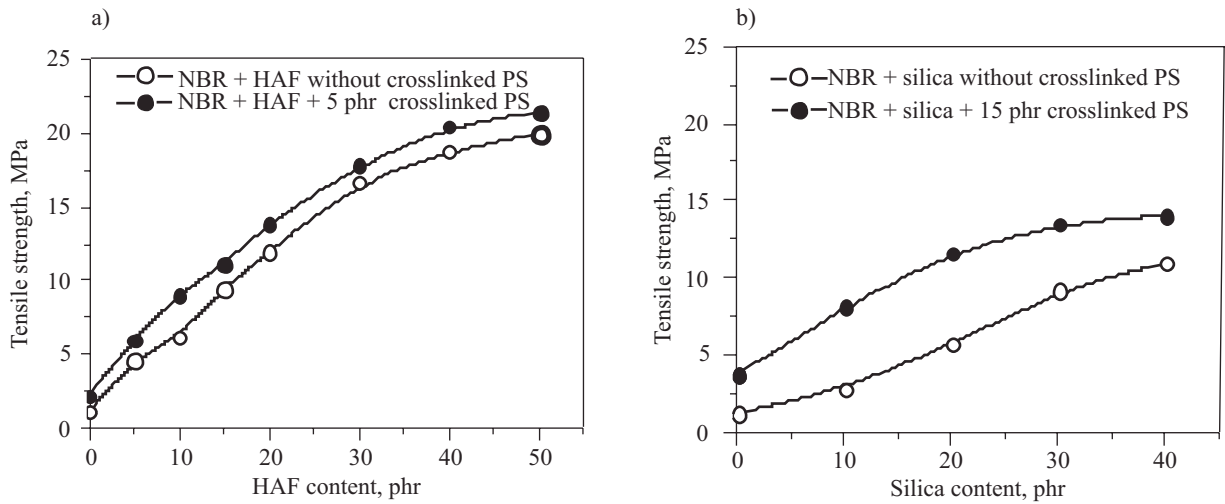


Fig. 9. Tensile strength of the NBR composites versus filler content: a) NBR + HAF composites, b) NBR + silica composites

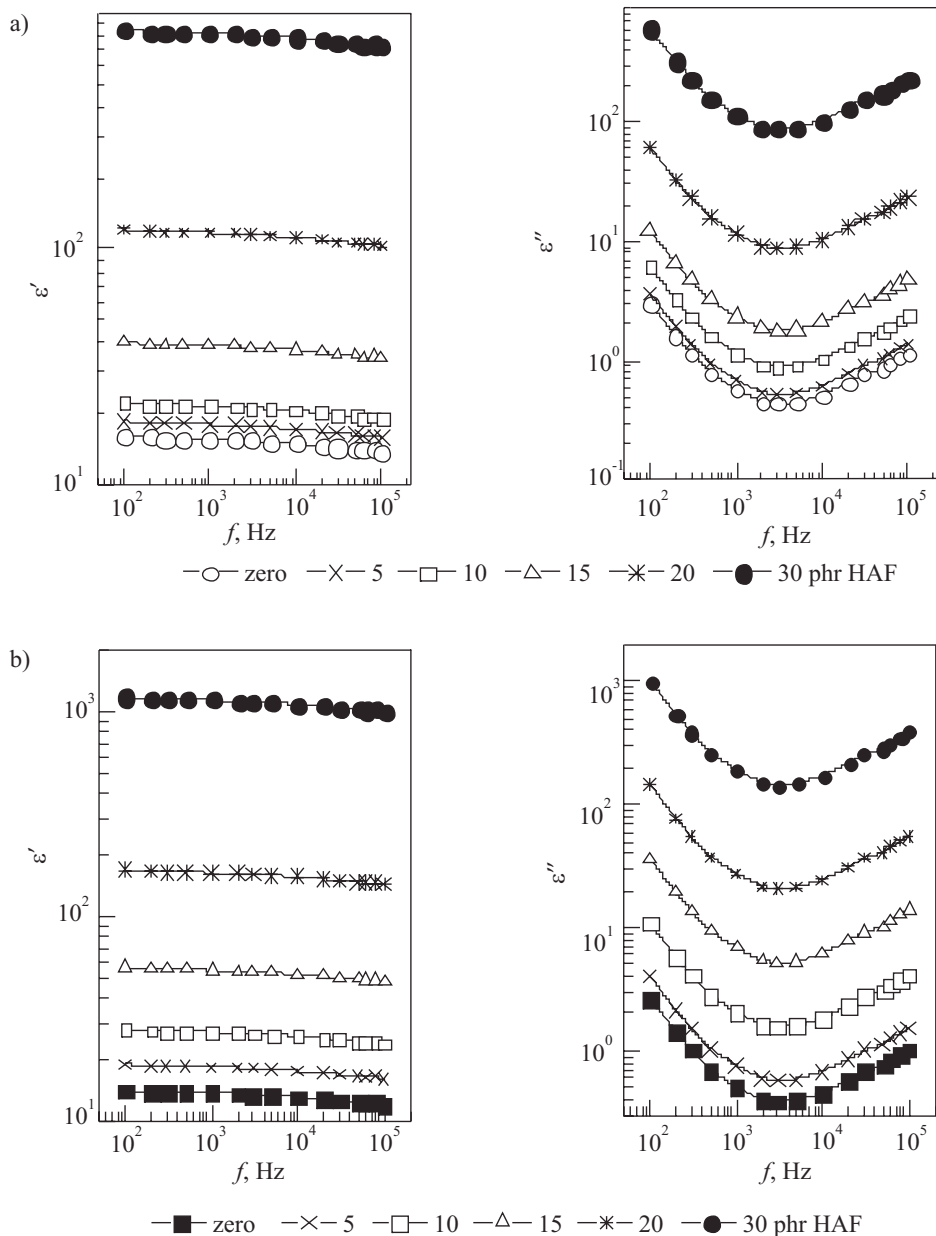


Fig. 10. The permittivity ϵ' and dielectric loss ϵ'' versus the applied frequency f for: a) NBR composites without crosslinked PS, b) NBR composites with 5 phr crosslinked PS loaded with different contents of HAF

From this figure it is clear that a fine morphology is observed for the sample blend where the surface appeared to be smooth and uniform. On the other hand, the SEM micrographs of the NBR/crosslinked PS (5 and 15 phr) show two phases as the PS granules appear very easy to aggregate as seen in Figs. 8b, 8c.

The NBR/crosslinked PS filled with HAF or silica

Filler type plays an important role in the properties of the end polymeric product. For example, when the filler is able to conduct by itself so the end product will behave like a semiconductor or conductor. If the filler is inert, *i.e.* its electrical conductivity is low, the end product will be an insulator [26].

From Fig. 9a it is clear that the tensile strength of the NBR/crosslinked PS/HAF composites increase with higher HAF contents. Also, the presence of crosslinked PS increases the tensile strength for all samples.

Figure 9b represents the tensile strength of the NBR/crosslinked PS/silica composites versus silica concentrations. It is shown that crosslinked PS enhances the tensile strengths, which increase with higher silica concentration.

In order to enhance electrical conductivity of the polymeric matrix, HAF was chosen as a conducting filler to be filled with NBR and NBR/crosslinked PS (100/5) with various contents. The permittivity ϵ' and dielectric loss ϵ'' for the prepared composites are given in Fig. 10. From this figure it is noted that ϵ' and ϵ'' increase with higher HAF contents. The sharp increase in ϵ'' is attributed to the increase in contributions from both interfacial polarization and conductivity, while for higher content of HAF this is mainly due to conductivity [27]. The data of ϵ' and ϵ'' given in Fig. 10 at the different frequencies indicate that an abrupt increase is noted when the concentration of HAF reaches 20 phr in the case of NBR, 15 phr for NBR/crosslinked PS blend. At these points, there is a tendency for increased conductivity chain formation

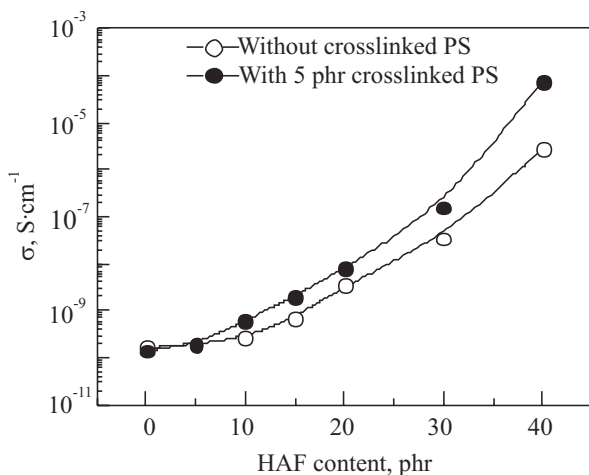


Fig. 11. Electrical conductivity of the NBR/HAF composites versus HAF content

through the aggregation of the carbon black particles network, while with low concentration, the particles of HAF are widely dispersed through the polymeric matrix.

This result finds further justification through the electrical conductivity of the composites under investigation, as seen in Fig. 11. It is clear that the electrical conductivity σ was enhanced by the addition of crosslinked PS. This is due to the uneven distribution of the filler in the incompatible blend matrix [27] that enhances the electrical conductivity of the composite.

NBR is characterized by good mechanical properties and good oil resistance [28] but it has moderate insulating properties even it has higher values of permittivity ϵ' (17–14) [29]. The considerably high dielectric loss ϵ'' values (1.33–3.50) mean that NBR loses its insulating properties to some extent. For such a reason, silica was chosen as the composite with NBR to reduce the dielectric loss of NBR in order to obtain an end product suitable for insulation purposes.

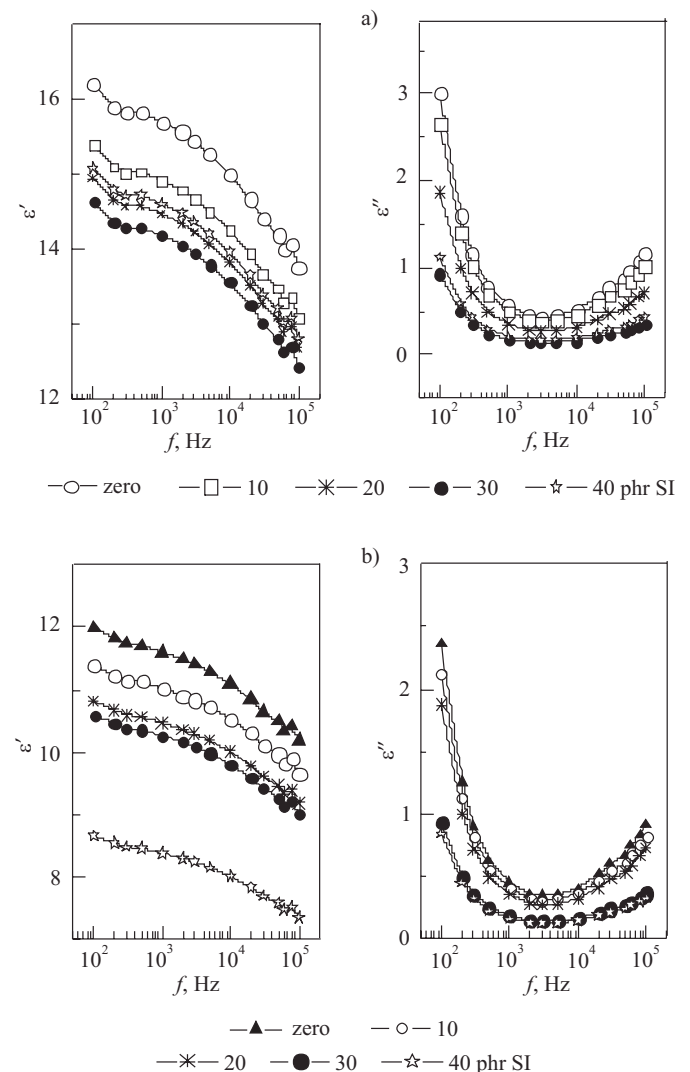


Fig. 12. The permittivity ϵ' and dielectric loss ϵ'' versus the applied frequency f for: a) NBR, b) NBR/15 phr crosslinked PS, loaded with different contents of Si

The permittivity ϵ' and dielectric loss ϵ'' for the prepared composites are given in Fig. 12.

It is clear that ϵ' decreases with higher applied frequency (f) showing an anomalous dispersion. Also, the variation of ϵ'' with the applied frequency indicate more than one relaxation processes.

From this figure it is seen that both ϵ values decrease with higher silica contents, which is due to the restriction of the polarity of nitrile group ($C\equiv N$).

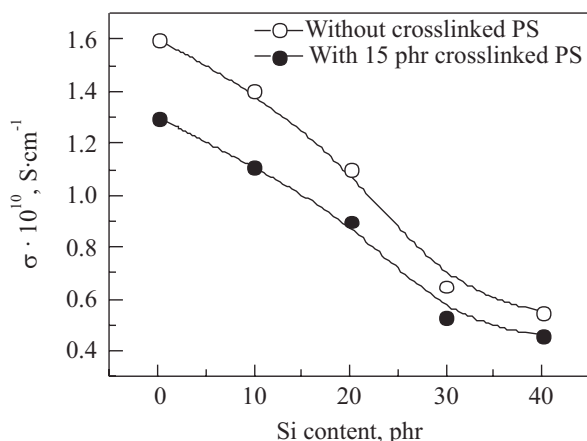


Fig. 13. The permittivity ϵ' and dielectric loss ϵ'' of NBR/Si composites, at fixed frequency $f = 100$ Hz, versus silica content

It is concluded that the optimum silica (Si) loading is 30 phr, after this concentration a pronounced decrease in ϵ' is accompanied by a slight decrease in ϵ'' values.

The electrical conductivity σ was calculated for all composites under investigation and illustrated graphically in Fig. 13. As can be seen, the electrical conductivity σ decreases with higher Si contents. Also, it is clear that σ for NBR/Si composites is slightly higher than that for NBR/15 phr crosslinked PS/Si composites.

This finding recommends NBR/15 phr crosslinked PS/Si composites to be used for insulating purposes. Also, it is clear that 30 phr Si is recommended for insulating purposes because a slight decrease in σ values was noticed above this value.

CONCLUSIONS

Crosslinked polystyrene with DVB was successfully synthesized by radical emulsion polymerization. The crosslinked PS took a spherical shape with an average diameter between 75 nm and 160 nm. When filled into elastomeric matrices, the crosslinked PS was found to be both effective and efficient in elastomer reinforcement. The crosslinked PS filled with NBR composites exhibited a dramatic enhancement in mechanical performances and in electrical properties compared with the neat elastomer matrices.

HAF was chosen as conducting filler filled with NBR and NBR/5 phr crosslinked PS with various content to

enhance the electrical conductivity of the polymeric matrix. The result shows that the electrical conductivity σ was enhanced by the addition of crosslinked PS.

Silica was chosen to be the composite with NBR and NBR/15 phr crosslinked PS at various contents to reduce the dielectric loss of NBR in order to obtain an end product suitable for insulation purposes. The result shows that the electrical conductivity σ decreases at higher Si contents. Also, it is clear that σ for NBR/Si composites is slightly higher than that for NBR/15 phr crosslinked PS/Si composites.

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