DIELECTRIC BEHAVIOR OF SBS/POLYANILINE THERMALLY PROCESSABLE BLENDS

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Abstract. Styrene–butadiene–styrene triblock copolymer (SBS) blends were prepared using different weight ratios of conducting polyaniline (PANI). Several electrical properties such as volume resistivity, dielectric constant, dissipation factor and alternating current conductivity of the blends were studied pointing to understand the influence of different parameters like PANI concentration, frequency, and temperature on these properties. The prepared materials present a slight reduction in volume resistivity as a function of temperature, being useful in conditions where the temperature control is hard. More than this, dielectric constant tests allowed inferring that among tested samples, the one filled with 45 wt % of PANI is potentially able to be used in the electromagnetic dissipation field. Finally, the alternating current tests allowed to prove that the electrical conduction of the blend is mainly dominated by ionic transportation. Therefore, prepared materials can be considered as a strategic, low-cost, environmentally friendly material which can be used in various border fields.

Keywords: SBS, polyaniline, melt blending, dielectric behavior.

1. Introduction

Intrinsically conductive organic polymers constitute an exciting class of polymers, which have been extensively studied due to their physical and chemical properties. Among them, polyaniline (PANI) is one of the most frequently used in several applications [1-3] due to its low cost; high conductivity, achieved by protonation with mineral or organic acids; long-term stability of its conductivity and reversible redox properties [4-6]. On the other hand, processing of PANI by ordinary methods used in the polymer industry is hard due to a very rigid conjugated backbone, restraining the increase of the scale production [7, 8]. This disadvantage can be minimized if PANI is doped with functional protonic acids, such as the dodecylbenzenesulfonic acid (DBSA) [9]. DBSA presents surfactant properties and solubility in most of the organic solvents, allowing an easier mixing process of the PANI and polymer matrix [10].

In recent years, a considerable number of researchers is concerned on conducting blended materials prepared by several mixing processes with conventional polymers [11-16]. This kind of approach has managed to produce electrical conductive blends with a broad range of unusual mechanical and electrical properties [17]. Among them, thermomechanical processing is a well-known method for large-scale production of plastics in the conventional polymer industry, being also very interesting in thermally processable conducting polymer blends [18-22].

Among insulating polymer materials, the elastomers can produce a continuous phase which imparts their unique mechanical properties to the conducting composites [23-27]. Up to now, there are several methods of preparation of conductive polymer-rubber blends, such as: electrochemical [28, 29], latex [23, 30, 31], absorption [31], adsorption [32], inverse emulsion polymerization [33, 34], and blending [35-37]. Among elastomeric materials, styrene–butadiene–styrene triblock copolymer (SBS) has many commercial applications. Polystyrene (PS) segments are hard domains, and polybutadiene (PB) segments are soft domains in SBS. PS domains act as physical crosslinks inside SBS. Composites constituted by SBS and conducting fillers are useful in situations where flexibility is necessary. In fact, these composites can be molded into various complex shapes by thermomechanical processing as described previously [9, 38-41].

The primary goal of the present paper is related to the evaluation of the complex electrical behavior of conducting SBS/PANI blends. Electrical tests were performed on materials containing different PANI

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concentrations, under different temperatures. Results are significant for a better understanding of the materials properties, allowing for a future faster and improved design of electrical polymeric components. Besides that, to the best of our knowledge, this is the first time that SBS-PANI/DBSA blends prepared by melt mixing are deeply investigated regarding their electrical properties, such as volume resistivity (ρ_v), dielectric constant (ε_r), dissipation factor (tanδ) and alternating current (*ac*) conductivity aiming to understand the influence of different parameters like PANI concentration, frequency, and temperature on these properties.

2. Experimental

2.1. Materials

SBS (TR-1061; PBD content 70 wt%; M_w 120,000, density 0.98 g/cm^3) was kindly supplied by Petroflex S.A. (Rio de Janeiro, Brazil). Aniline, ammonium persulfate (APS) both of analytical grades obtained from Vetec, Brazil, and dodecylbenzene sulfonic acid (DBSA) (commercial grade from Solquim LTDA, Brazil) were used without further purification.

2.2. Synthesis of Polyaniline Doped with DBSA (PANI/DBSA)

Polyaniline doped with DBSA (PANI/DBSA) was synthesized by one-step route in toluene according to the procedure reported elsewhere [42]. In a typical procedure, 4.7 ml (0.051 mol) of aniline and 16.7 g (0.051 mol) of DBSA were dissolved in 250 ml of toluene under constant stirring. The reaction mixture was kept at 273 K, and an aqueous solution containing 11.36 g (0.051 mol) of APS in 40 ml of water was slowly added for a period of 20 min. After 6 h, the reaction medium was poured into a non-solvent such as ethanol, filtered, washed several times with ethanol and dried.

2.3. Preparation of the Blends

Blends were prepared by melt mixing method in a Haake Rheocord 9000 internal mixer. Temperature and speed operational conditions were fixed at 373 K and 60 rpm, respectively. CAM rotors were used. A known amount of SBS was first introduced into the mixer. After 2 min PANI/DBSA powder was quickly added. Total residence time was equal to 8 min. Finally, blends were removed from the mixer and quickly sheeted in a two-roll mill.

2.4. Characterization

The Fourier Transform Infrared spectra of materials were performed using a Varian model 3100 FTIR Excalibur Series spectrophotometer (Japan).

Samples were macerated with potassium bromide (1 mg/100 mg samples/KBr). The FT-IR spectra of the samples were recorded at room temperature and a resolution of 4 cm^{-1}. The ultraviolet-visible spectrometry measurements were performed on a Varian UV-Vis Spectrometer Model CARY 100. The samples were dissolved in toluene with the concentration of $5.5 \cdot 10^{-5}$ g/ml. Resolution was equal to 1 nm. Scanning electron microscopy (SEM) experiments were performed with a JEOL JSM-5610 LV microscope, using acceleration voltage of 15 kV. Samples were coated with gold to study the morphology of the prepared materials. Steel electrodes (with 15 mm in radius) were used aiming to measure the electrical properties following the ASTM D257-99. Capacitance and dissipation factor of SBS/PANI blends were measured using precision LCR HP meters models 4285A 4192A. Frequency was kept within 5 Hz–30 MHz range. Acquired capacitance results were utilized for the dielectric constant calculations following the procedure described by Grove, Masters, and Miers [43]. Tests at different temperatures (298–348 K) were performed in a temperature controlled oven.

3. Results and Discussion

Fig. 1a shows the FTIR spectrum of the PANI/DBSA. Among its characteristic bands the wide one placed at 3595 cm^{-1}, is related to the asymmetric stretching of NH$_2$ and NH. The band centered at 3244 cm^{-1} corresponds to stretching of NH. The bands at 1503 and 1630 cm^{-1} refer to stretching of N=Q=N and quinoneimine moiety, respectively. The band corresponding to the stretching of the benzenoid ring is centered at 1392 cm^{-1}. The peak at 1289 cm^{-1} is related to stretching of the C–N bond that exists between quinonoid and benzenoid rings. The band at 1221 cm^{-1} corresponds to stretching of C–O conjugated with the asymmetric stretching of S=O, both of DBSA. The peak at 1049 cm^{-1} is characteristic of the stretching of the sulfoxide group (C$_2$S=O). The band at 530 cm^{-1} is related to the bounding C–S of DBSA [38]. In turn, the UV-Vis spectrum, which is shown in Fig. 1b, presents the typical π–π* absorption peaks placed at 356 nm and two polaronic bands placed at 435 and 806 nm, confirming the successful production of PANI/DBSA [44].

Fig. 2 shows the SEM of SBS, SBS/PANI, and PANI/DBSA. PANI presents a typical granular morphology [42]. In turn, SBS presents a plane surface, while SBS/PANI presents a rough surface. This roughness is due to the presence of PANI, which keeps its granular aspect. Also, PANI (the whiter phase in Fig. 2c) is a continuous phase, which means that a conductive pathway was formed. This conductive pathway is mandatory to the obtaining of the improved electrical properties of the blended materials.
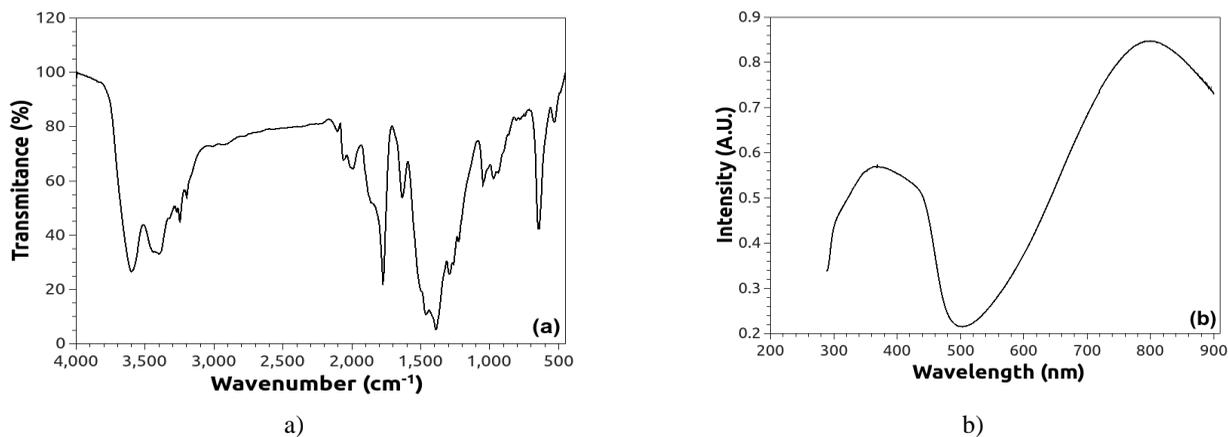
a)   b)

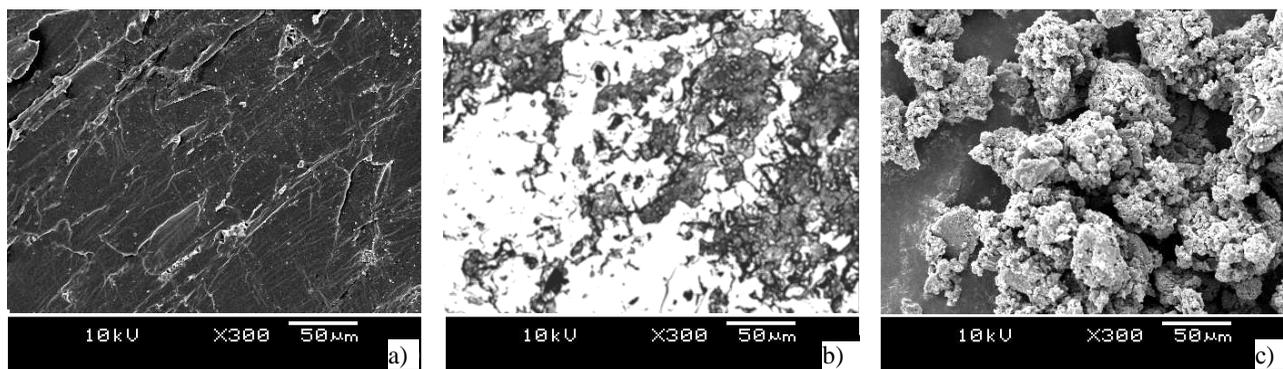**Fig. 1.** FTIR (a) and UV-Vis (b) of the PANI/DBSA sample

Fig. 2. SEM of SBS (a), SBS filled with 30 wt % of PANI (b) and PANI/DBSA (c)

The plot of volume resistivity, ρ_v, as a function of the amount of PANI inside blends is shown in Fig. 3. Results show that up to 15 wt % of PANI, tested materials remain in insulating values ($\rho_v > 10^{13}\,\Omega\cdot m$). Volume resistivity presents a slight decrease when PANI content reaches 15 wt %, which corresponds to the percolation threshold of the present polymeric system, as proved by SEM. Further amount of PANI produces a linear reduction of the ρ_v value. The same behavior is noticed regardless applied electrical voltage. Minimum values of ρ_v, 10^7–$10^8\,\Omega\cdot m$, are reached in blends containing 45 wt % of PANI. This result indicates that PANI acts as a conducting filler and the material changes from insulating to semiconducting one. Besides that, it is fascinating to notice that the amount of reduction in volume resistivity is slightly influenced by temperature changes (see Fig. 3). Therefore, prepared materials can be used in conditions where temperature control is hard, which is a crucial ability in a pressure sensor field [17].

The dielectric constant, ε_r, of the material is a measure of the extent to which it can concentrate electrostatic lines of flux [45-47]. It is a number relating the ability of the material to carry alternating current to the ability of vacuum to carry alternating current [48]. The dielectric constant is an essential information when designing capacitors and in other circumstances, where a material is expected to introduce capacitance into a circuit, allowing for the accurate circuit design of devices able to be used in a radar, RF admittance, or capacitance technologies [49-52]. The effect of V_f of PANI on ε_r of SBS/PANI blends, was studied at two different temperatures, equal to 298 and 348 K. Results are shown in Fig. 4. The increase of PANI content generates a linear increase in the ε_r values, proved by substantial correlations equal to 0.996±0.003 and 0.97±0.03, respectively.

All samples presented a decrease of the ε_r values with the increase in frequency, as can be better seen in Fig. 5. Also, observed frequency dependence of ε_r increases with the increase in PANI concentration. These observations are related to any factor able to affect the polarization of bound charges, so that the larger the polarizability of the molecule, the higher is ε_r of the material. Thus, if the frequency increases, the dipoles present in the system cannot reorient themselves fast enough to respond to applied electric field resulting in a dielectric constant decrease [53].
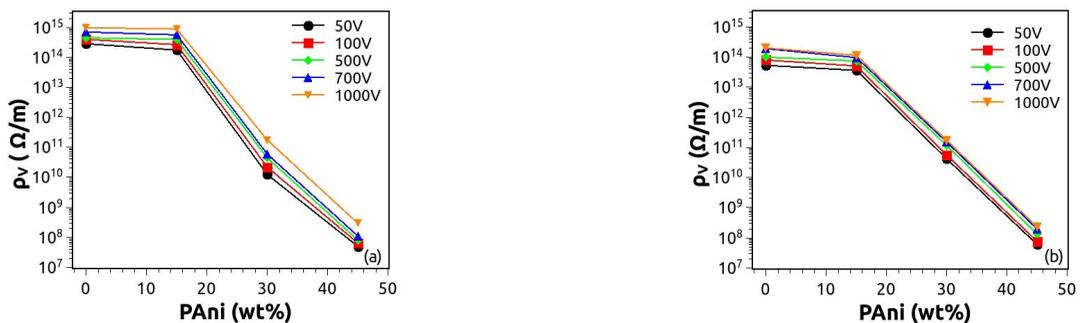
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Fig. 3. Effect of volume fraction of PANI on volume resistivity of SBS/PANI blends at 298 K (a) and 323 K (b)
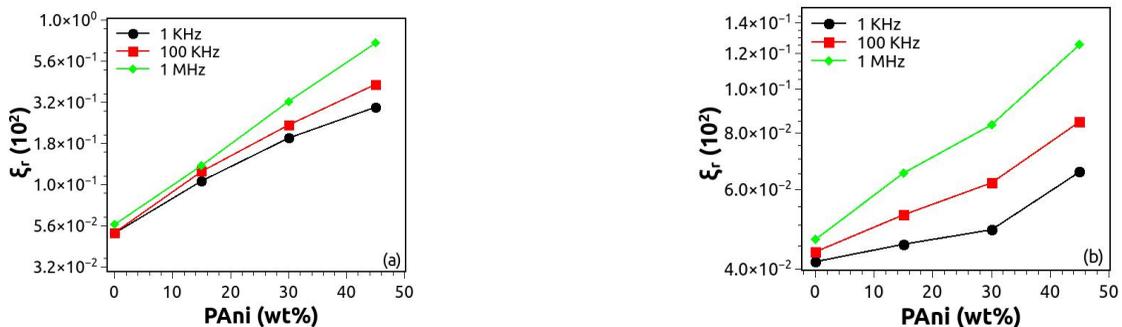

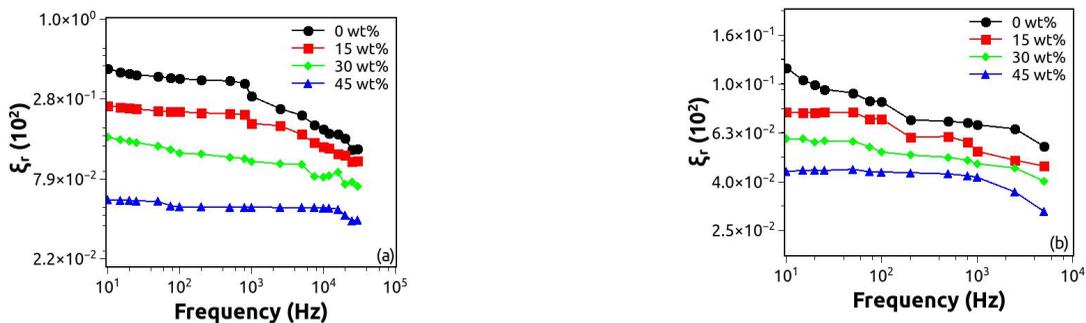**Fig. 4.** Effect of temperature on volume resistivity of SBS-PAn composites at 500 V (a) and 1000 V (b)

Fig. 5. Plots of dielectric constant as a function of frequency for SBS/PANI blends at 298 K (a) and 348 k (b)
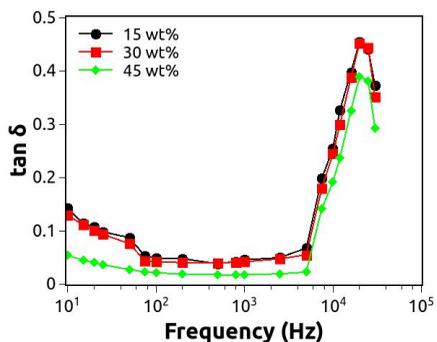

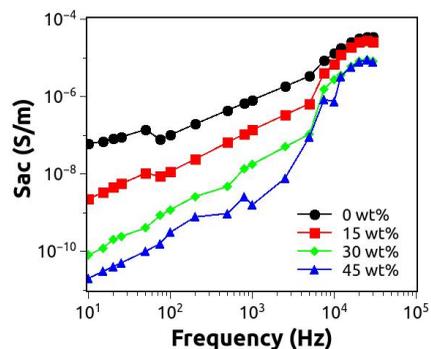**Fig. 6.** tan*δ vs.* frequency for SBS/PANI blends

Fig. 7. Variation of *ac* conductivity *vs.* frequency for SBS/PANI blends at 298 K

Due to the presence of insulating matrix (SBS), some charge carriers present in PANI simply are stuck, making hard the free discharge at the electrodes. Discharge difficulty leads to a charge accumulation at the interface of conducting filler and insulating matrix. This kind of event is known as Maxwell-Wagner-Sillers interfacial polarization (MWS) [53-56]. In the present case, MWS polarization leads to the generation of some space charge at the interface leading to a field distortion. As the frequency of the applied electric field is reduced, the contribution of ionic conduction towards the total loss becomes higher. With the increase in frequency, the tendency for the interfacial polarization is expected to be decreased resulting in a decrease in polarizability. At any particular frequency, because PANI presents a higher polar character and higher electrical conductivity than pure SBS, the increase of ε_r with PANI amount raise is possible due to the increased polarizability caused by the dipole concentration. Among tested samples, blends containing 45 wt % of PANI presented the lowest ε_r values in the function of the applied frequency, indicating that these materials are potentially able to be used in the electromagnetic dissipation field [53].

The dielectric dissipation factor, tanδ, is a fundamental electrical property of the polymer, being very important in several applications such as printed wiring board materials; interlay dielectrics and insulation of electric components [57]. Dissipation factor can be understood as the ratio between energy loss to the alternating current (*ac*) field and energy stored in the dielectric materials from ac electric field. In conventional terms, it is defined as the ratio of current which is in phase compared to the current which is 90° out of phase [58]. The dissipation factor tanδ of a polymer is the function of frequency, temperature, and material properties [59-61].

Fig. 6 shows the variation of tanδ against frequency. All blended materials present the same behavior, characterized by an insignificant influence of frequency on tanδ at lower frequencies (10–5000 kHz). On the other hand, tanδ peaks can be observed inside the range of $5 \cdot 10^3$–10^5 kHz, for all blended materials, indicating an increase of the energy loss to the ac field in this frequency range. Therefore, this result shows that these materials can be easily used in the 10^3–10^4 kHz frequency range without appreciable change in tanδ values of the blends.

Finally, Fig. 7 shows the *ac* conductivity results as a function of the applied frequency in SBS/PANI blends. Fig. 7 indicates a frequency dependence of conductivity, which leads to a conductivity increase with a frequency raise. The increase in *ac* conductivity can be considered linear (correlation equal to 0.98±0.02) inside the range of $(2 \cdot 10^2)$–$(4 \cdot 10^4)$ kHz, indicating that the conduction of the blends is mainly dominated by ions.

4. Conclusions

The present paper constitutes a significant step to better understanding of SBS/PANI electric characteristics. Obtained volume resistivity results prove that SBS/PANI blends prepared by the melt-mix process are materials able to present semiconductor properties. Besides that, the resistivity of the presented materials is slightly influenced by temperature changes, allowing for the preparation of sensing devices able to be used in conditions where a temperature control is hard without modifications in electrical resistivity. Beyond sensing field, dielectric constant tests indicate that materials containing 45 wt % of PANI are potentially able to be used in electromagnetic dissipation field allied with low costs of production. Therefore, present melt mix processing used to prepare SBS/PANI blends is very interesting, being able to produce strategic, low cost and environmentally friendly materials useful to several frontier fields.

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ДІЕЛЕКТРИЧНА ПОВЕДІНКА ТЕРМООБРОБЛЕНОЇ СУМІШІ СБС/ПОЛІАНІЛІН

Анотація. Одержано триблок-кополімерні суміші стирен-бутадієн-стирену (СБС) з різними концентраціями поліаніліну (ПАН). Визначено вплив таких параметрів, як концентрація ПАН, частота та температура на електричні властивості: густину, діелектричну проникність, тангенс кута втрат та провідність змінного струму. Показано, що незначне зменшення об'ємного питомого опору як функції температури, є корисним за умов утрудненого контролю температури. Встановлено, що зразок із 45 % мас. ПАН потенційно може бути використаний у полі електромагнітного випромінювання. Доведено, що електрична провідність суміші переважає йонне транспортування. Одержані матеріали можна використовувати як стратегічний, недорогий, екологічно чистий матеріал у різних суміжних сферах.

Ключові слова: СБС, поліанілін, суміш розплавів, діелектрична поведінка.