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Conductive Thin Films based on Sodium Alginate/ PEDOT:PSS Blends

Filmes finos condutores baseados em misturas de alginato de sódio/ PEDOT:PSS

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The association between the polymers (3,4-ethylenedioxythiophene) and poly (4-styrenesulfonic acid) PEDOT:PSS results in properties found in plastics with electrical characteristics similar to metals. The combination of the PEDOT:PSS with sodium alginate, a renewable polymer derived from alginic acid, allows the creation of homogeneous thin films. As it has a renewable polymer matrix, this blend appears as an alternative to the replacement of polymers from non-renewable sources in applications. The investigation of the resulting electrical properties, as well as the material morphological characteristics, are the main objectives of this work. The films were assembled using the solvent evaporation method, casting, from aqueous solutions of PEDOT:PSS and sodium alginate in various proportions. Analysis of the voltage versus current curve showed a PEDOT:PSS concentration dependence for increasing the conductivity of the formed film and its conductivity results proved to be superior to works found in the literature. Profile analysis showed that casting technique produced films of circa 1000 nm and the spin casting technique produced films circa 250 nm of thickness; X ray diffraction (XRD) revealed that the material is predominantly amorphous; UV-Vis absorbance measurements indicated great absorption of light energy in the RGB pattern at length of 650 nm; images from scanning electron microscopy indicated the components' homogeneity in the formation of the films and the optical microscopy showed the continuity of the films formed. The DSC curves were in agreement with data from other researchers. FTIR analysis demonstrated that the absorption bands are equivalent to those found in the literature. In this way, the synthesized material presents suitable characteristics to be applied in the construction of an electrochromic device.

Keywords: PEDOT:PSS; sodium alginate; renewable material

1. Introduction

In view of the growing need for increasingly specific materials, the idea of associating properties found in metals with those of polymers was successful when the first semiconductor polymer was synthesized and studied in the 1970s.[1] From this point, science was able to reach a new level in the development of instruments and devices to meet the new human needs.

Semiconductor polymers are a class of polymers that have the property of electrical conduction[2] and an alternating structure of pi and sigma bonds between carbons.[3] Unlike metals that naturally have a high density of charge carriers, semiconductors need to be doped with a charge carrier, either by inserting or removing an electron. This type of doping is called p or n, and depending on the content of the doping, the material's semi conductivity can be modified, allowing a fine adjustment of this property.[4]

Research on semiconductor polymers paved the way for the most diverse applications such as LED, solar cells, sensors, electrodes, antistatic protectors, organic transistors, anti-corrosion, electromagnetic shielding, and in medicine. This justifies research on these materials.[5-10]

The association of the semiconductor polymer PEDOT (poly(3,4-ethylenedioxythiophene)) when associated with the dopant PSS (polystyrene sulfonate) has a conductivity of 10^2 S.cm^{-1}, being comparable with the conductivity of metals (10^3 S.cm^{-1}). Furthermore, PSS improves PEDOT's low solubility in water, increasing the ease of production of thin films and solutions. Figure 1 (a) and 1 (b) represent the structure of PEDOT and PSS.

PEDOT:PSS has good thermal stability, transparency and besides being a semiconductor, it has the effect of electrochromism in thin films (adjustment of absorbance through the

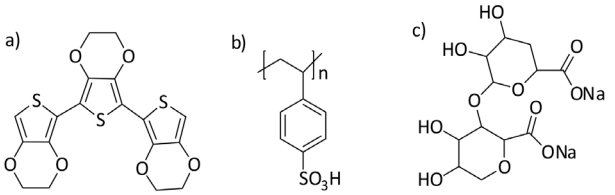

Figure 1. Structures of (a) PEDOT, (b) PSS e (c) Sodium Alginate

passage of electric current)[11,12]. This last characteristic, has attracted several researches for the production of electronic devices such as, smart windows, solar cells, mirrors, slates, commercial inks, among others.[4,13,14]

The advent of new architectures combining materials[15] from natural sources was the motivator for the association of a polymer from a renewable natural source,[16] sodium alginate (Figure 1 (c)). This polymer is a mixture of two acids, β-D-mannuronic (M) and α-L-guluronic acid in various proportions and it is used to construct thin and homogeneous films in medical area, as well as thickener and homogenizer. It was used as a stabilizer for PEDOT:PSS and as a support polymer, since it is transparent in the wavelength of visible light, and offers rigidity to the formed film.[17]

This work presents the development of thin conductive films obtained from the PEDOT:PSS and alginate blend. The quality of the films was determined based on the morphological characterization by measurements such as Scanning Electron Microscopy, Transmission/reflection optical microscopy and profilometry. In addition, UV-Vis measurements and current–voltage curves were performed to determine the optical absorption and conductivity of the films, respectively.

2. Materials and Methods

2.1 Materials

Sodium alginate from IHARA, PEDOT:PSS from Clevios PH-1000, glycerol, ethylene glycol, propanone and isopropyl alcohol from Labsynth.

The aqueous solution was prepared by dissolving the sodium alginate in deionized water (2% w/w). The mixture was made at room temperature with constant mechanical stirring for two hours. After complete dissolution, glycerol was inserted in the proportion of 60% of the alginate mass in order to increase the plasticity of the sodium alginate solution. The solution was stirred for another two hours.

PEDOT:PSS was mixed with ethylene glycol (5% w/w) and stirred for two hours. According to results in the literature[18] the conductivity of the PEDOT:PSS polymer was increased due to the secondary doping effect.[4]

After preparing the sodium alginate solution with glycerol and PEDOT:PSS with ethylene glycol, different proportions were obtained for preparing the solution and making the thin films (Table 1).

Table 1. Preparation of solutions

Sample	Sodium alginate with glycerol	PEDOT:PSS with ethylene glycol
Sample 1	100	0
Sample 2	99	1
Sample 3	95	5
Sample 4	90	10
Sample 5	80	20
Sample 6	70	30
Sample 7	60	40
Sample 8	50	50
Sample 9	60	40
Sample 10	70	30
Sample 11	80	20
Sample 12	90	10
Sample 13	100	0

Thin films were made according to the procedure shown in Figure 2.

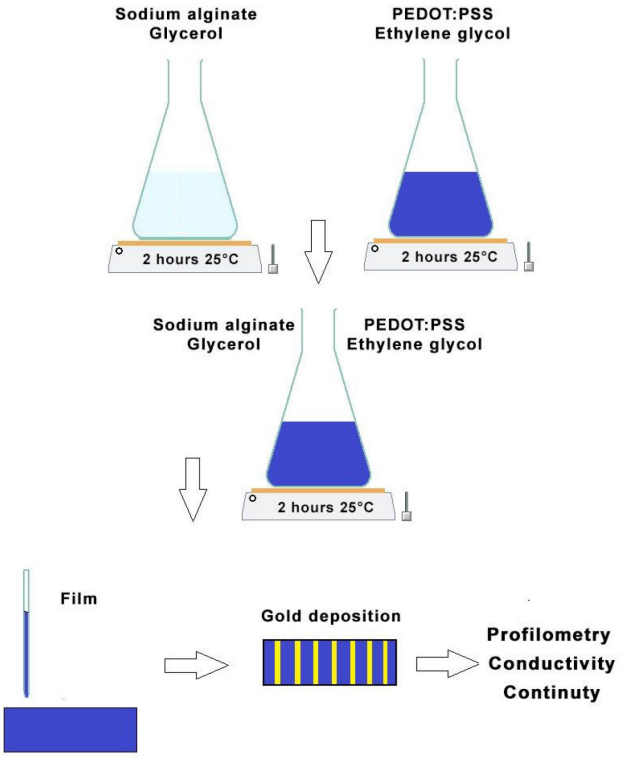

Figure 2. Production of the solutions and the films

2.2. Thin films preparation

Thin films of PEDOT:PSS/alginate were prepared by two methods: i) casting, where the solution is deposited on the pre-cleaned substrate, and; ii) spin casting, where the solution is deposited on the substrate, rotating it with a speed of 1500 rpm during 15 seconds. In both procedures, the films were placed under a heating plate at 50 °C for the evaporation of the solvent.

The thickness of the films was determined by means of profilometry measurements using Veeco Dektak 150.

2.3. Optical and Morphological Characterization

Fourier Transform Infrared Spectroscopy (FTIR) was performed from the solutions used to make thin films. Measurements were performed on the Agilent FTIR Carry 630 spectroscope and analyzed in the ATR mode in the energy range 4000 cm^{-1} to 650 cm^{-1}.

The thin spin casting films were analyzed on the Hitachi U-2900 spectrophotometer in the scanning mode in the range of 400 to 1100nm. As presented in Results, films deposited via casting had a thickness of 119 µm, making it difficult to acquire the absorption spectrum since the detected intensity exceeded the equipment limit.

Surface images of Alginate/PEDOT:PSS films were obtained through scanning electron microscopy using a Zeiss SEM-FEG model Sigma. The films were deposited on carbon tape and dried in an oven at 50ºC for two hours. Then, carbon fibers were deposited via evaporation on the films.

2.4. Electrical conductivity

The electrical conductivity of the films is the most important parameter to be observed in materials that can be used for the manufacture of electronic devices. The electrical conductivity measurements were performed using IxV measurements at steady state. For this purpose, the films were deposited on a glass substrate by the casting technique, and after the solvent evaporation had ended, the gold electrodes were deposited using a metallic mask on the thin films. For evaporation of the electrodes, the Edwards evaporator model AUTO 360 was used with a vacuum chamber of 10^{-6} mBar. The deposited electrodes had an average thickness of 100 nm, and the metallic mask allowed the formation of 9 electrodes, according to Figure 3 (a). After metallization, the films were connected to a voltage and current source as shown in Figure 3 (b).

Electrical resistance of the device was determined by voltage-current measurements. According to the first Ohm's Law, current flowing through the device is proportional to the applied voltage. Mathematically,

$$V = RI \tag{1}$$

The resistivity (ρ) of a material is a characteristic that comes from the way the constituents of the material are organized and the temperature, while the resistance (R) is related to the geometry of each material. Ohm's second law states that the electrical resistance is directly proportional to the length (L), but inversely proportional to its transverse area (a). Thus, it is possible to determine the resistivity from the resistance, according to the equation (2)

$$R = \frac{L}{a}\rho \tag{2}$$

Each film was metallized aiming 9 different aspect ratios to observe the dependence between film's geometry and conductivity calculation. Each of the 9 aspect ratios (n) is 1 cm wide (h) and the films have thickness (w), equation 2 relates these two factors, becoming equation (3)

$$R = \rho\frac{nh}{wh} = n\frac{\rho}{w} \tag{3}$$

For thin films, is the sheet resistance (Rs), the ratio between the film resistance and the aspect ratio n, is given by equation (4)

$$Rs = \frac{R}{n} \tag{4}$$

where n varies from 1 to 9, as shown in Figure 3 (a). Taking the conductivity ($S.cm^{-1}$) of the material as the inverse of the resistivity, we can rewrite Eq. 03 in the following form:

$$\sigma = \frac{1}{R_s w} \tag{5}$$

2.5. Scanning electron microscopy

The morphological surfaces of the thin films of Alginate, PEDOT:PSS and the Alginate/PEDOT:PSS composite were analyzed using Scanning Electron Microscopy (SEM). Sigma microscope from Zeiss with GEMINI column and equipped with field-emission electron gun. The images were obtained with 4.00 KV.

2.6. X-ray (XRD)

The samples were deposited in a sample holder and dried in an oven at 50 °C for two hours. The Shimadzu

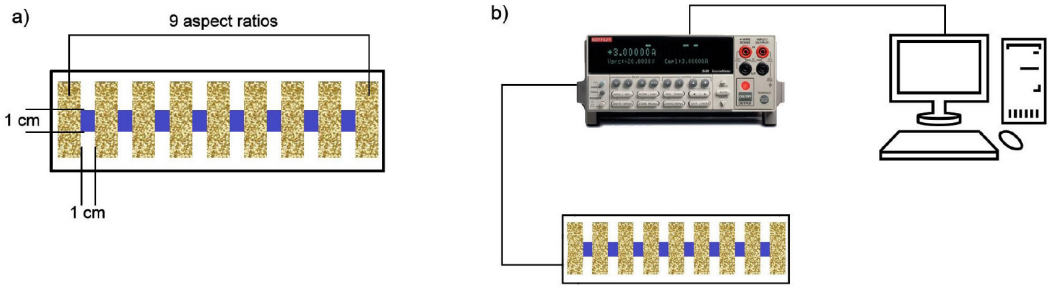

Figure 3. Conductivity determination scheme

diffractometer operated under a voltage of 40 kV and a current of 15 mA, at a step of 10° every 5 min, in the range of 10 to 70°.

2.7. Fourier transform infrared spectroscopy

With spectroscopy we expected to observe the appearance of new bands due to the connections between the components of the material. The liquid sample was analyzed on the Agilent FTIR Carry 630 spectrophotometer and analyzed on the 4000 cm^{-1} to 650 cm^{-1} range.

2.8. Differential scanning calorimetry

The differential scanning calorimetry aimed to observe the transitions associated with the heat flow as a function of temperature and time variation. The DSC131evo calorimeter was used in the temperature range of 40 to 550 °C, heating rate of 3 °C.min^{-1}, under nitrogen atmosphere**.**

3. Results and Discussion

3.1. Optical and morphological characterization

As the present work seeks to develop a blend with semiconductor characteristics from a biopolymer, alginate, it is essential to determine the thickness of the thin films obtained from the PEDOT:PSS/Alginate solution, since the sheet resistance depends on the thickness of the films. Figure 4 represents the profile measurements of thin films. As expected, thin films by spin casting showed smaller thickness due to the centrifugal force present in the deposition process, decreasing the amount of solution on the substrate. Although films via casting are thicker, the dependence on the concentration of the solution appears to be the same for both deposition methods. Considering the average value (straight red line) between the maximum and minimum values of the thickness (dashed straight black line), the percentage variation between the maximum/minimum values and the average value corresponds to 14.4% for casting method and 29.7% for spin casting.

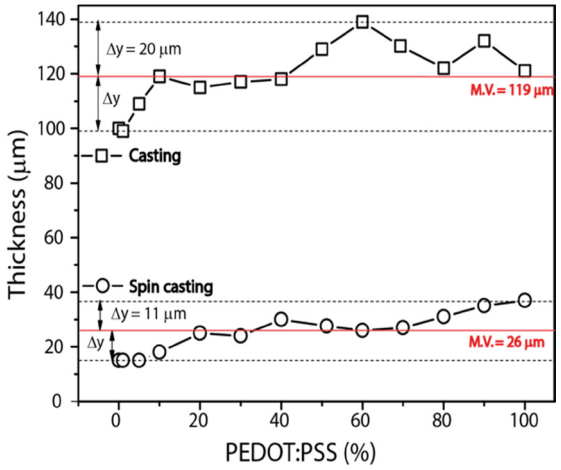

Figure 4. Film thickness in both casting and spin casting construction methods

3.2. X-ray diffraction

The crystallinity index of the material and its relationship with the variation of the semiconductor material content were determined by XRD.

Figure 5 shows the XRD patterns of the samples analyzed and the increase in the content of the semiconductor polymer.

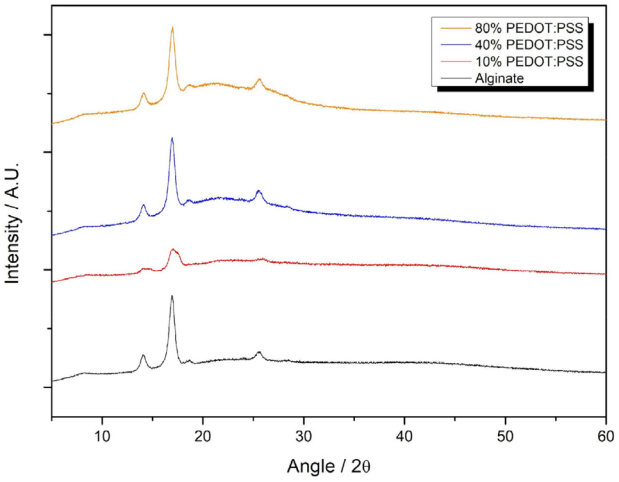

Figure 5. XRD patterns of samples with different compositions

The 2θ peaks at 13.7, associated with the crystallographic plane (110) correspond to peaks found by other researchers about materials containing sodium alginate[19] Peak in 24.1 is the same found in material containing PEDOT:PSS.[20] The degree of crystallinity has a small variation in relation to the variation in the concentration of the semiconductor polymer. The low crystallinity content, around 7%, imposes restrictions on the passage of electric current.

3.3. FTIR

The samples were submitted to spectroscopy and the main groups present were observed, as shown in Figure 6.

Among the various bands observed, a special attention must be given to the 3300 cm^{-1} one, which is compatible with the OH vibration mode present in both sodium alginate and glycerol. A lower transmittance was observed for this band in the spectra of samples with PEDOT:PSS, explained by the presence of ethylene glycol due to the O–H bonds in its structure. The 2926 cm^{-1} and 2874 cm^{-1} bands correspond to the C–H stretch present in glycerol and alginate. The 1594 cm^{-1} and 1410 cm^{-1} correspond to the presence of the O=C–O– carboxylic bond present in the sodium alginate, since the compound is a carboxylic salt. The peak at 1040 cm^{-1} refers to the PSS sulfonated group. Peaks at 1368 cm^{-1} correspond to the asymmetric elongations of the carbon-carbon bonds.

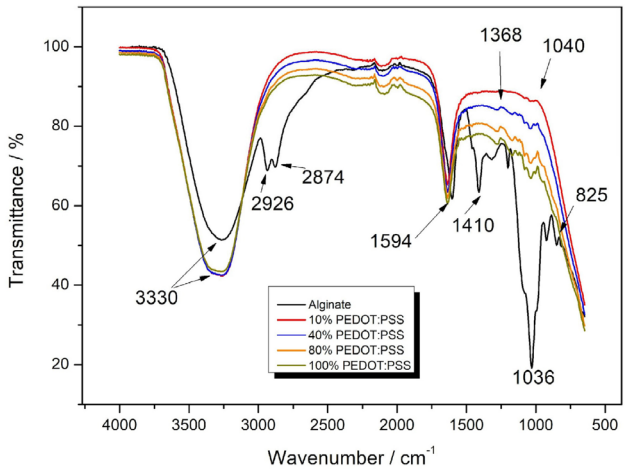

Figure 6. FT-IR spectra of samples with different compositions

From the FTIR spectra obtained, we can see that the mixture between Alginate, Glycerol and PEDOT:PSS was characterized as a physical mixture and as corroborated by the spectra of Figure 6, it did not produce new chemical bonds between the components of the mixture.

3.4. UV-Vis

UV-Vis absorption measurements were made from thin films deposited via spin casting, as presented in Figure 7 (a). As expected, the increase in the concentration of PEDOT:PSS caused an increase in absorption, as a consequence of the increase in the number of chromophore groups in the films. It can also be observed that there was no change in the profile of the spectra, meaning that the mixture between the two polymers does not cause the appearance of new optically accessible energy states. Once the thickness of the films is known (Figure 4) as well as their absorbance, the optical absorption coefficient was determined using the Lambert-Beer equation.

The absorption coefficient was calculated for the wavelengths in blue (λ = 440 nm), green (λ = 550 nm) and red (λ =740 nm) regions, since these represent the primary colors of the RGB standard. The increase in the optical absorption coefficient showed a linear dependence with the increase in the PEDOT:PSS concentration in the blend films, as shown in Figure 7 (b).

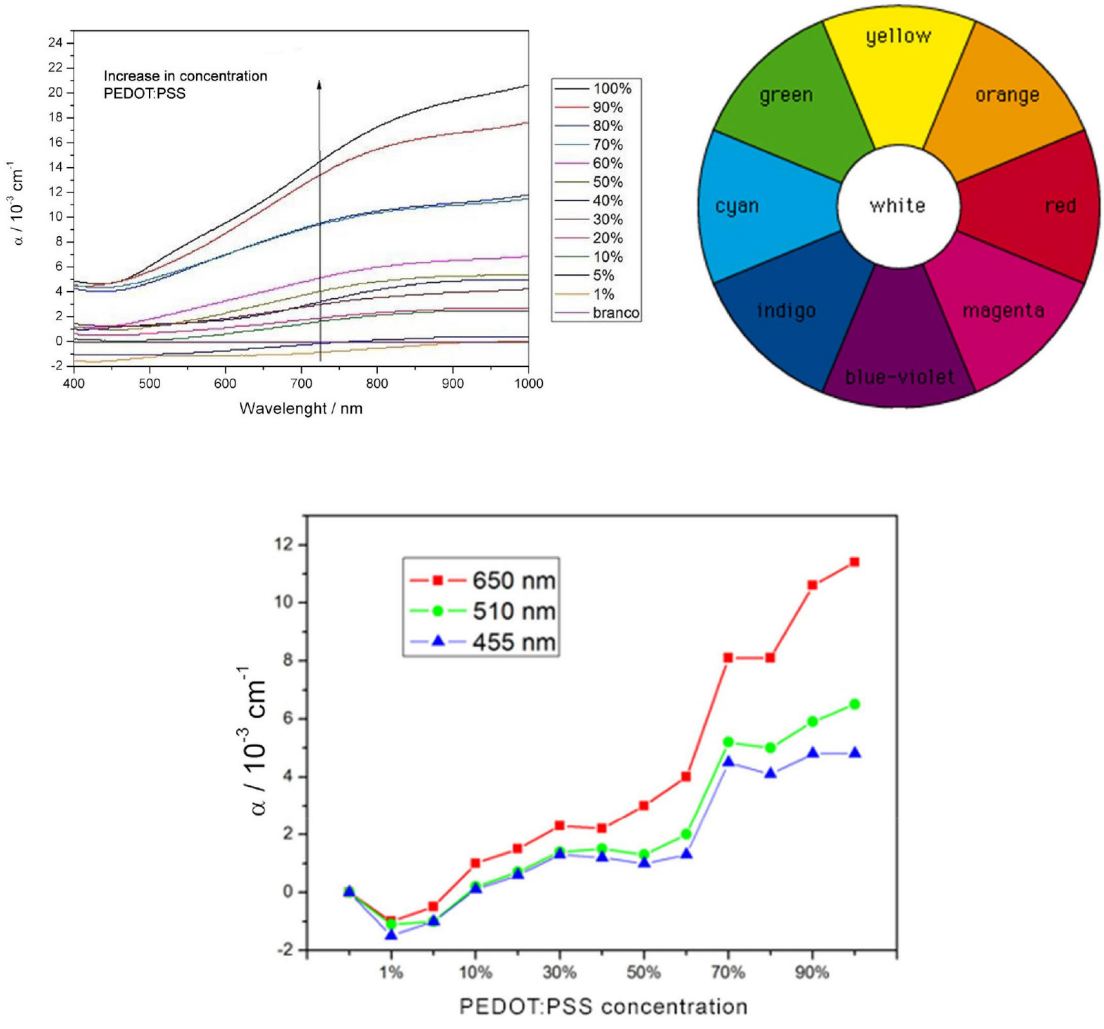

Figure 7. UV-Vis spectra of samples with different compositions

3.5. SEM

Scanning electron microscopy images were performed to analyze the morphological surface of the thin films of the Alginate/PEDOT:PSS blend and to investigate a possible change on that surface with the increase in the PEDOT:PSS amount of the analyzed samples. Thin alginate films were uniform and visibly rough on the 200 nm scale, as shown in Figure 8 in the upper left corner. For PEDOT:PSS films, a fibrous surface was observed, characterized by the PEDOT:PSS chains, as shown in Figure 8 in the lower right corner.

With the addition of PEDOT:PSS to the alginate solution, in Figure 8 (b), it is possible to observe that the film presents clusters regions, this is explained by the thready characteristic of PEDOT:PSS. Sodium alginate is permeated between the chains of the semiconductor polymer serving as a support, so it does not have a structure that can be seen in the central image. From this observation, it is noted that the homogenization time of the material in about 2 hours was sufficient for the mixing to be carried out.

3.6. Electrical conductivity

IxV measurements in stationary mode were performed as described in section 2.2.3: Electrical Conductivity. From the voltage-current curves (Figure 9 (a)), a linear dependence of the current with the applied voltage is clearly observed, thus characterizing the device as an ohmic device, regardless of the aspect ratio. From the voltage-current curves, the sheet resistance (Rs) was determined for each blend composition (Table 02). Figure 9 (b) represents the variation in the conductivity of thin films obtained for different concentrations of PEDOT:PSS.

Table 2. Sheet Resistance (RS) values and conductivity of thin films

Samples	Sheet Resistance (Ω/Sq)	Conductivity (S/cm)
Alginate	$1.9x10^{5}$	$5.0x10^{-2}$
1%	$3.7x10^{6}$	$3.0x10^{-3}$
5%	$1.1x10^{5}$	$8.0x10^{-2}$
10%	$9.4x10^{4}$	$9.0x10^{-2}$
20%	$2.4x10^{5}$	$4.0x10^{-2}$
30%	$6.0x10^{4}$	10^{-1}
40%	$5.9x10^{2}$	$1.3x10^{1}$
50%	$3.4x10^{1}$	$2.5x10^{2}$
60%	$2.1x10^{1}$	$3.4x10^{2}$

3.7. DSC

The DSC curves of the samples are shown in Figure 10 and some thermal events can be observed.

The first thermal event observed refers to the sample's dehydration process. As the process remains until close to 120 °C, it can be associated with a coordinated water molecule. The exothermic event above 200 °C can be related to the beginning degradation of the alginate material.[21] Samples containing PSS undergo a decomposition process close to 290 °C. Above 450 °C the degradation of PEDOT begins.[22]

4. Conclusion

The research presented refers to the study of the association between sodium alginate, a polymer from a renewable source, together with PEDOT:PSS, semiconductor polymer, aiming to elucidate the morphological, optical and electrical

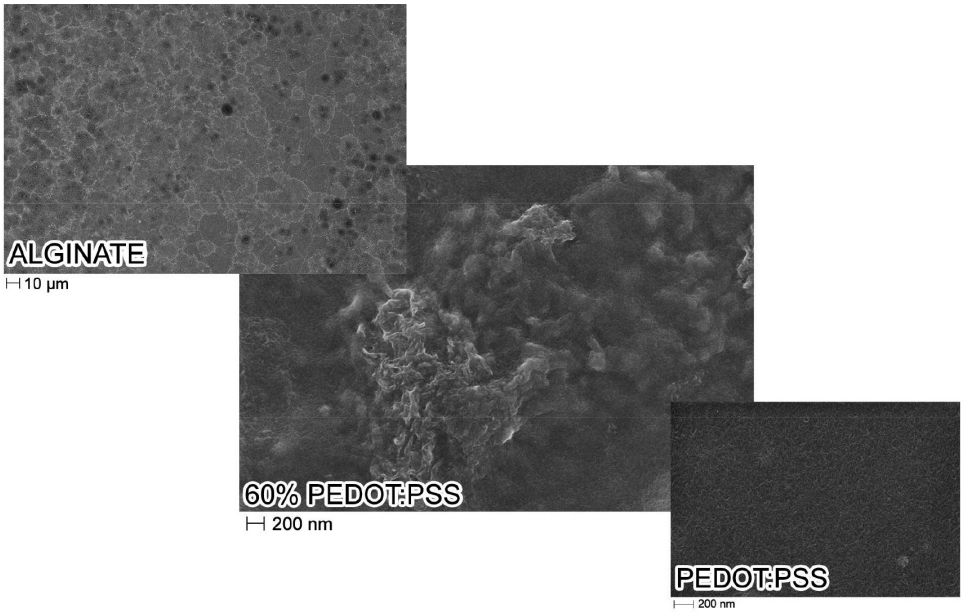

Figure 8. SEM sample 60%

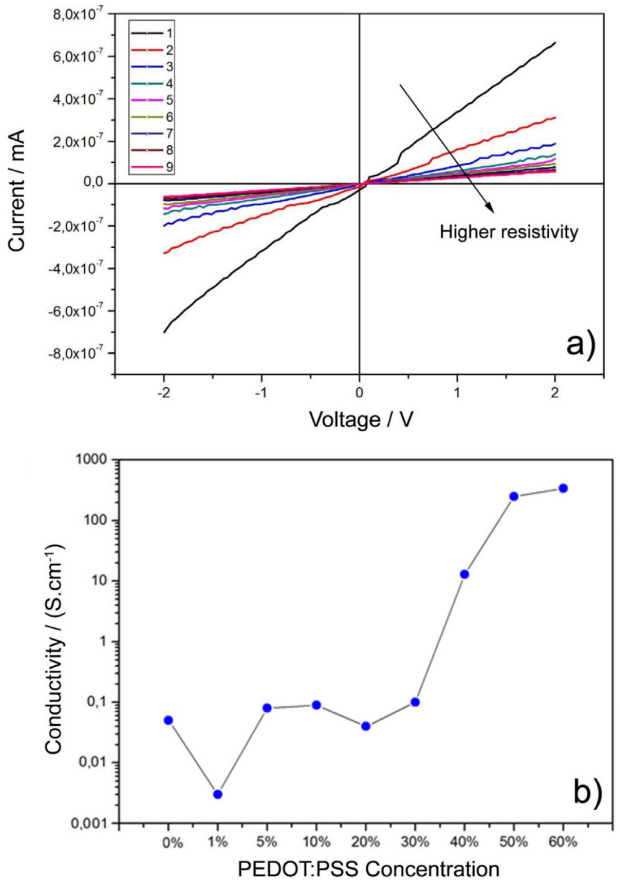

Figure 9. (a) Voltage-current curves, (b)variation in the conductivity of thin films

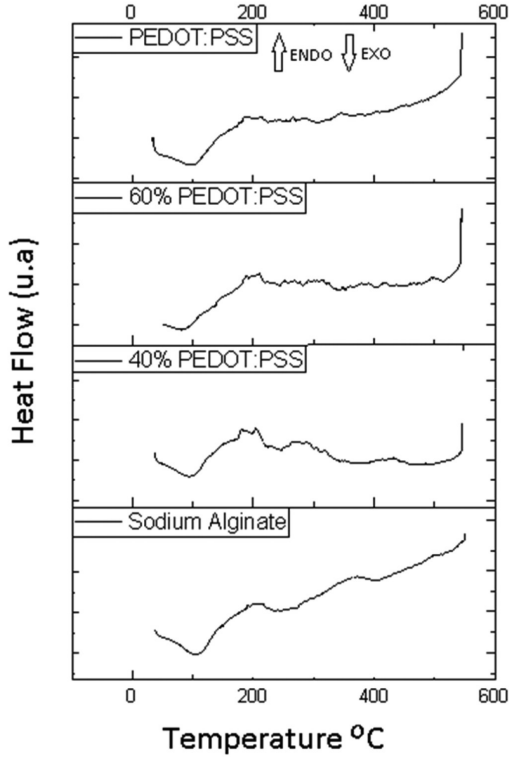

Figure 10. DSC curves of samples with different compositions

characteristics of thin films of Alginate/PEDOT:PSS blends. Such parameters should be fundamental for the construction of an electronic device that combines the electrical and optical characteristics presented, such as the electrochromic devices.

The FTIR characterization showed infrared absorption bands that are characteristic of samples containing alginate and PEDOT:PSS, reinforcing the concept of mixing between the two polymers. Spectroscopy measurements have shown UV-Vis absorption dependence of the PEDOT:PSS percentage used in the production of films. Concomitant with UV-Vis measurements, voltage-current measurements demonstrated a gain in the blend's conductivity, turning the Alginate/PEDOT:PSS blend as a good candidate in the manufacture of electrochromic devices, since the material under optimized conditions has low absorption, good conductivity ($\sigma \sim 10^{-2}$ to 10^{2} S.cm^{-1}) and electrochromism. The images obtained by scanning electron microscopy demonstrate the homogeneity of the films when the percolation threshold is reached and show how is the association between the semiconductor polymer and sodium alginate. The DSC results obtained corroborate the results of the literature. Subsequent studies will be carried out in order to decrease the content of glycerol (plasticizer) in the production of the films to increase the crystallinity and consequently increase the obtained conductivity.

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Author's contribution

Jorge Fernandes Filho -Conceptualization
Samir Leite Mathias – Formal Analysis
Bruno Millan Bassi Torres - Resources
Caio Henrique Gomes - Investigation
Aparecido Junior de Menezes – Funding acquisition
Rafael Henriques Longaresi - Supervision

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