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Thermal and electrochemical impedance spectroscopy analysis of PAADDA film incorporated with Li_2SO_4

Análise Térmica e de espectroscopia de impedância eletroquímica de filmes de PAADDA incorporado com Li_2SO_4

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Resumo
Neste trabalho, filmes de poli(acrilamida-co-dialidimetilamônio)-PAADDA contendo sulfato de lítio (Li_2SO_4) foram preparados pelo método da evaporação do solvente a partir de soluções aquosas. Os filmes obtidos (PAADDA, PAADDA/Li_2SO_4-0,25% m/m e PAADDA/Li_2SO_4-0,5% m/m) foram caracterizados por espectroscopia de infravermelho por transformada de Fourier com refletância total atenuada (ATR-FTIR), análise termogravimétrica (TGA) e espectroscopia de impedância eletroquímica (EIE). Os espectros ATR-FTIR dos filmes de PAADDA/Li_2SO_4 mostraram as principais bandas do PAADDA e sugeriram uma interação intermolecular significativa entre o polímero e o Li_2SO_4. As curvas TGA mostraram que a estabilidade térmica do filme de PAADDA é dependente da concentração do Li_2SO_4. A condutividade iônica dos filmes seguiu a ordem: PAADDA/Li_2SO_4-0.25% w/w < PAADDA < PAADDA/Li_2SO_4-0.5% w/w.
Palavras-chave: PAADDA, sulfato de lítio, espectroscopia de impedância eletroquímica.

Abstract
In this paper, poly(acrylamide-co-dialidimethylamonium)-PAADDA films containing lithium sulfate (Li_2SO_4) were prepared by casting method from aqueous solutions. The obtained films (pristine PAADDA, PAADDA/Li_2SO_4-0.25% w/w, and PAADDA/Li_2SO_4-0.5% w/w) were characterized by means of attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR), thermogravimetric analysis (TGA), and electrochemical impedance spectroscopy

(EIS). ATR-FTIR spectra of PAADDA/Li_2SO_4 films showed the main bands of PAADDA and suggested a significative intermolecular interaction between the polymer and Li_2SO_4. TGA curves displayed that the thermal stability of PAADDA film is dependent on the Li_2SO_4 concentration. The ionic conductivity of the films followed the order: PAADDA/Li_2SO_4-0.25% w/w < pristine PAADDA <PAADDA/Li_2SO_4-0.5% w/w.
Keywords: PAADDA, lithium sulfate, electrochemistry impedance spectroscopy.

1. Introduction

Solid composite polymer electrolytes (SCPEs) are a class of materials formed by mixture of polymer and inorganic salts that can be applicated in electrochemical systems such as batteries, super capacitors, fuel cells and sensors (Fu *et al*., 2022; Vanitha *et al*., 2017; Devangamath *et al*., 2020). These materials possess excellent properties like flexibility, film forming property, elasticity, light weight, and fairly easy processability (Fu *et al*., 2022; Vanitha *et al*., 2017; Devangamath *et al*., 2020). In addition to these properties, SCPEs present ionic conductivity that is influenced by dissociation of the salt and polymer morphology (Deshmukh *et al*., 2016). Among the salts used in the preparation of SCPES, the lithium salts have been widely used in batteries and supercapacitors due to their high conducting medium and recyclability (Uma *et al*., 2005, 2004; Muthuvinayagam *et al*., 2019; Ullah *et al*., 2022; Virya and Lian, 2017; Zhang *et al*., 2015). Specially, lithium sulfate (Li_2SO_4) has been applied in the development of SCPES because it has low dissociaton energy and can increase the ionic conductivy of polymer film (Zhang *et al*., 2015).

The poly(acrylamide-*co*-diallyldimethyl-ammonium chloride), PAADDA, is a cationic copolymer, water soluble widely used in oil exploitation, papermaking, wastewater treatment, mining, printing and dyeing of textiles, daily chemical industry, and slurry dehydration (Guan *et al*., 2015; Vajihinejad *et al*., 2017). Besides, PAADDA has been used as conducting membrane and hydrogels. For example, AO et al. obtained a new alkaline anion-exchange membrane composed of chitosan (CS) and poly (acrylamid-co-diallyldimethylammonium chloride) (PAADDA) that presented great OH^- conductivity (Ao *et al*., 2018). In addition, Zhou et al. synthesized a novel membrane of chitosan (CS), PAADDA and linear structured poly-bis (2-chloroethyl) ether-1,3-bis [3-(dimethylamino)propyl] urea copolymer (PUB) with great physical-chemistry properties such as high oxidative stability and excelente alkaline resistance stability (Zhou *et al*., 2020). Zhang et al. prepared hydrogels composed by cellouronic acid sodium (CAS) and PAADDA which displayed a thermo-sensibility with an upper critical solution temperature (UCST) and a high water-absorbency (Zhang *et al*., 2018).

The aim of the current work was to obtain PAADDA/Li_2SO_4 composite films with different concentrations of Li_2SO_4 using casting technique for eletrochemical applications. The obtained films were characterized by means of attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR), thermogravimetric analysis (TGA), and electrochemical impedance spectroscopy (EIS).

2. Materials and Methods

2.1 *Materials*

The lithium sulfate monohydrate ($Li_2SO_4.H_2O$) was acquired from VETEC (Brasil). PAADDA solution (10 wt% in H_2O) was purchased from Sigma-Aldrich. All reagents were used as received.

2.2 *Preparation of the films*

PAADDA films containing different concentrations of lithium sulfate, 0.25 and 0.5% (w/w), were obtained by casting method from aqueous solutions at 40ºC. Pristine PAADDA film was also prepared as control. The aqueous solutions were poured in plastic dishes and left to evaporate the solvent. The obtained films were codded as PAADDA, PAADDA/Li_2SO_4 (0.25% w/w), and PAADDA/Li_2SO_4 (0.5% w/w).

2.3 *Attenuated Total Reflectance-Fourier Transform InfraRed Spectroscopy (ATR-FTIR)*

ATR-FTIR spectra of the samples were obtained using a Shimadzu IRAffinity-1spectrometer equipped with a single-reflection attenuated total reflectance (ATR) accessory. A ZnSe crystal mounted in tungsten carbide was used. The analysis was carried out in the frequency range 4000–700 cm^{-1}.

2.4 *Thermogravimetric Analysis (TGA)*

Thermal stability of the samples was investigated using a Perkin thermogravimetric analyzer (TGA4000) under dynamic N_2 atmosphere (gas flow of 20 mL/min). The samples were heated in a alumina crucible at a rate of 10°C/min over a temperature range of 30–550°C.

2.5 *Eletrochemical Impedance Spectroscopy (EIS)*

The impedance measurements were carried out using AUTOLAB PGSTAT 128N in the frequency range of 1 Hz to 1MHz at 25°C. The amplitude of the applied potential signal was 5 mV. For this experiment, films with diameter of 2 cm were disposed between two electrodes of stainless steel. The thickness of the obtained films was determined using a micrometer (Digimess).

3. Results and Discussion

3.1 *Attenuated Total Reflectance-Fourier Transform InfraRed Spectroscopy (ATR-FTIR)*

ATR-FTIR spectroscopy was used to investigate possible interactions between Li_2SO_4 and the PAADDA chains. Figure 1 displays the ATR-FTIR spectra of the obtained films.

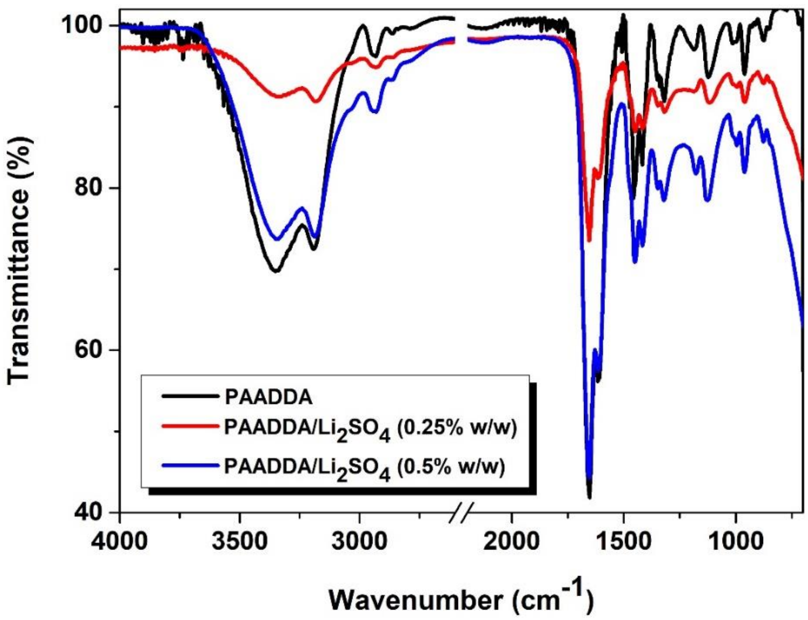
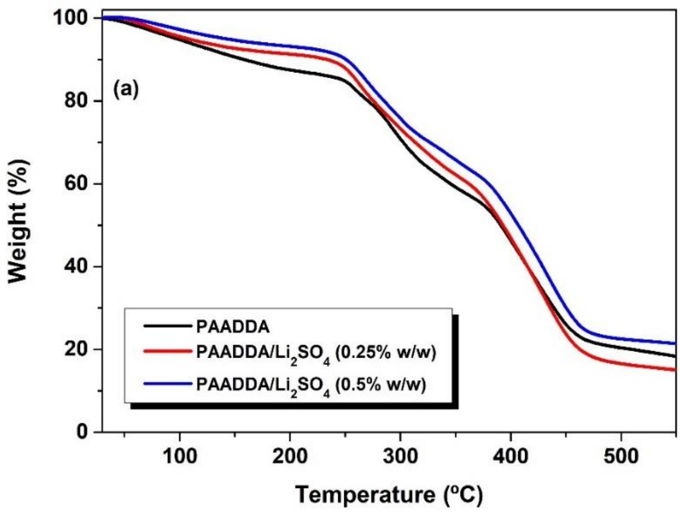


Figure 1. ATR-FTIR spectra of pristine PADDA and PAADDA/Li_2SO_4 films.

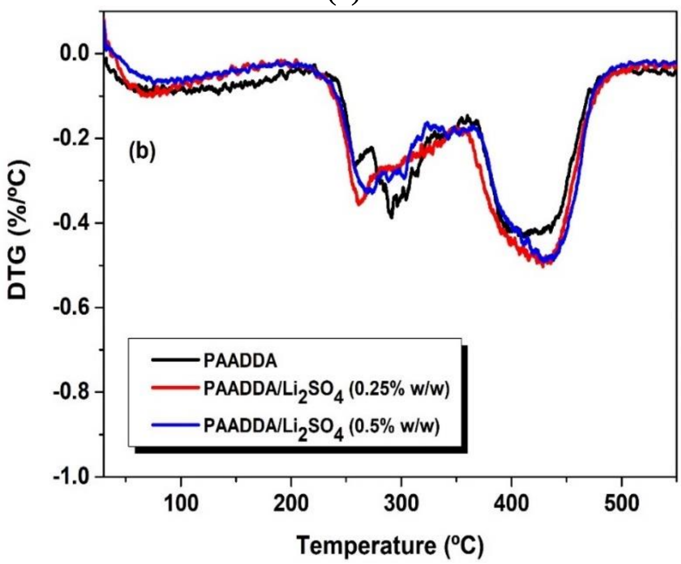
ATR-FTIR spectrum of PAADDA showed the mains bands of PADDA at 3346, 3184, 2936, 2866, 1654, 1611, 1448 e 1117 cm^{-1}. The vibrational bands at 3346 and 3184 cm^{-1} correspond to C-NH_2 e -NH_2 groups, respectively (Zhang *et al.*, 2018; Yang *et al.*, 2020; Afacam *et al.*, 2021). The vibrational bands at 2936, 2866, 1654, 1611, 1448, and 1117 cm^{-1} are due to the CH_2, CH, C=O, NH, $N(CH_3)_2^+$ e C-N groups, respectively (Zhang *et al.*, 2018; Yang *et al.*, 2020; Afacam *et al.*, 2021). As described in the literature, the main bands of Li_2SO_4 are exhibited at 3464 and 1609 cm^{-1} that can be attributed to vibrational modes of water molecules and between 1012–1016 cm^{-1},1110–1116 cm^{-1}, and 1170–1176 cm^{-1} that are associated to SO_4^{-2} anion (Najar *et al.*, 2017; Naik *et al.*, 2020). In the case of ATR-FTIR spectra of PAADDA/Li_2SO_4 (0.25% w/w) and PAADDA/Li_2SO_4 (0.5% w/w), it is possible to observe a significative decreasing of the intensity of the NH_2 groups compared to spectrum of pristine PAADDA. This behavior suggests a favorable interaction between the PAADDA and Li_2SO_4. Similar behavior was found for hydroxypropyl methylcellulose (HPMC) film incorporated with different sodium bromide (NaBr) concentrations (Kumar *et al.*, 2022).

3.2 *Thermogravimetric Analysis (TGA)*

TGA is an analytical technique widely used to study the thermal stability of polymer films. Therefore, TGA curves for pristine PAADDA and PAADDA/Li_2SO_4 films were obtained to evaluate the effect of the incorporation of Li_2SO_4 on the thermal behavior of the PAADDA film. The TGA and DTG curves of all the prepared films are reported in Figure 2.



(a)



(b)

Figure 2. (a) TGA and (b) DTG curves for pristine PAADDA and PAADDA/Li_2SO_4 films.

Thermal degradation of pristine PAADDA film occurred at four stages as exhibited by TGA and DTG curves (Figure 2). The first stage indicates the presence of water molecules in the PAADDA film and possesses a temperature of maximum of mass loss (T_{max}) = 113°C. The following three stages of thermal degradation were observed between 220°C and 500°C and are associated to the degradation of PAADDA chains (Zhang *et al*., 2018). The T_{max} for these three stages of thermal degradation were found at 257°C, 296°C, and 414°C. In order to investigate the effect of the incorporation of Li_2SO_4 into PAADDA film, the onset thermal decomposition temperature (T_{onset}) for the second stage of thermal degradation of PAADDA was determined from TGA curve. Then, T_{onset} was observed at 249°C.

Thermal behavior of the PAADDA film was changed in the presence of Li_2SO_4 salt as shown in Figure 2. PAADDA/Li_2SO_4 (0.25% w/w) film exhibited three stages of thermal degradation with T_{max} at 70°C, 261°C, and 427°C. In addition, T_{onset} for the second stage of thermal degradation of PAADDA was found at 247°C. For the PAADDA/Li_2SO_4 (0.5% w/w) film, TGA curve showed three stages with T_{max} at 110°C, 279°C, 431°C and T_{onset} for the second stage of thermal degradation of PAADDA at 250°C. As observed for the PAADDA film, the first stage of mass loss of the PAADDA/Li_2SO_4 films is associated to release of water molecules and the following stages are related to PAADDA degradation. Furthermore, it can be observed in Figure 2a that the TGA curve of the PAADDA/Li_2SO_4 (0.5% w/w) film displays a lower mass loss than pristine PAADDA film. These results suggest that the thermal stability of PAADDA increases with the rise of the Li_2SO_4 concentration. Probably, new ion-dipole interactions between PAADDA and the Li_2SO_4 salt favors the increasing of the thermal stability of the PAADDA film. ATR-FTIR results corroborate with this tendency. Ullah *et al*., (2022) observed the same behavior for polyvinylalcohol/multi walled carbon nanotubes films filled with Li_2SO_4.

3.3 *Eletrochemical Impedance Spectroscopy (EIS)*

Electrochemical impedance spectroscopy (EIS) is a technique widely used to investigate eletrochemical properties of different materials in liquid or solid phase (Abdul Halim *et al*., 2021). Therefore, EIS of pristine PAADDA and PAADDA/Li_2SO_4 films were evaluated to determine DC conductivity (σ), dielectric constant (ε_r), and the dielectric loss (ε_i) of all the obtained films. Nyquist plots for the pristine PAADDA and PAADDA/Li_2SO_4 films at room temperature are shown in Figure 3.

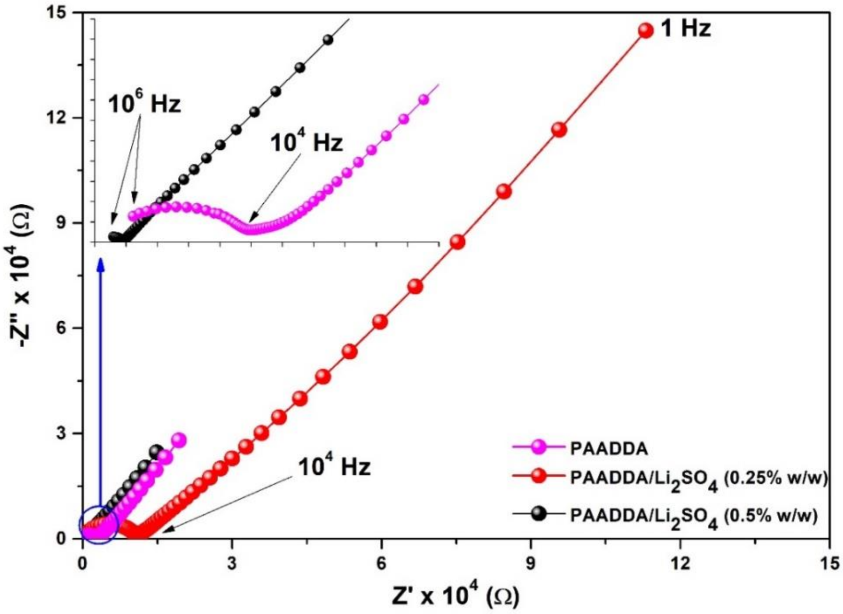


Figure 3. Nyquist plot for pristine PAADDA and PAADDA/Li_2SO_4 films.

Nyquist plots for the pristine PAADDA film and PAADDA/Li_2SO_4 films are formed by two different regions: (i) a semicircle that is related to conduction of charge carries at the bulk of the samples in the high frequency range and (ii) an inclined straight line in the low-frequency range that is associated to blocking effect of the stainless steel electrodes (Rauf *et al.*, 2022). From Nyquist plots, bulk resistance (R_B) was determined and thus R_B values were used to evaluate the DC conductivity (σ) values for all the obtained films. The following equation was used to calculate σ (Brza *et al.*, 2020):

$$\sigma = \frac{t}{R_B A} \qquad (1)$$

Where: t is the thickness of all obtained films, R_B is the bulk resistance (R_B) determined from Nyquist plot, and A is the area of the stainless steel electrodes.

The σ values obtained for pristine PAADDA and PAADDA/Li_2SO_4 films are shown in Table 1.

Table 1. DC conductivity (σ) values of the pristine PAADDA and PAADDA/Li_2SO_4 films at room temperature.

Films	**σ ($S.cm^{-1}$)**
PAADDA	$3{,}44.\ 10^{-6}$
PAADDA/Li_2SO_4 (0.25% w/w)	$6{,}02.10^{-7}$
PAADDA/Li_2SO_4 (0.5% w/w)	$1{,}33.10^{-5}$

As seem from Table 1, the σ values for the films followed the order: PAADDA/Li_2SO_4 (0.25% w/w) < PAADDA < PAADDA/Li_2SO_4 (0.5% w/w). The σ is dependence of the density of carriers (η) and carrier's mobility (μ) and then their values increase with the salt concentration (Brza *et al.*, 2020). Therefore, these results suggest that for 0.5% (w/w) of Li_2SO_4, the higher values of η and μ contribute to this σ value. Furthermore, the σ value for PAADDA/Li_2SO_4 (0.25% w/w) is smaller than pristine PAADDA. Probably, the μ for this film is smaller due to interaction between the salt and the polymer chains as shown by ATR-FTIR results. Table 2 presents the comparison of the σ values obtained in this work with other papers.

Table 2. Comparison of the σ values of the pristine PAADDA and PAADDA/Li_2SO_4 films with different systems.

Films	**σ ($S.cm^{-1}$)**	**Reference**
PAADDA	$3{,}44.\ 10^{-6}$	This work
PAADDA/Li_2SO_4 (0.25% w/w)	$6{,}02.10^{-7}$	This work
PAADDA/Li_2SO_4 (0.5% w/w)	$1{,}33.10^{-5}$	This work
PVA/PEG/$LiClO_4$ (7 wt%)	$1{,}04.10^{-5}$	Amiri *et al.*, 2017
Poly(vinyl alcohol)	$2.87.10^{-11}$	Brza *et al.*, 2020
Poly(vinyl alcohol)-Cu complex	$7.71.10^{-8}$	Brza *et al.*, 2020
Poly(vinyl alcohol)-Ce complex	$4.80.10^{-8}$	Brza *et al.*, 2020
Pectin	$2.68.10^{-9}$	Muthuvinayagam *et al.*, 2019
Pectin/ Li_2SO_4	$6.06.10^{-8}$	Muthuvinayagam *et al.*, 2019
Poly(vinyl alcohol)/BMIMCl/ Li_2SO_4	37.10^{-3}	Zhang *et al.*, 2015

.

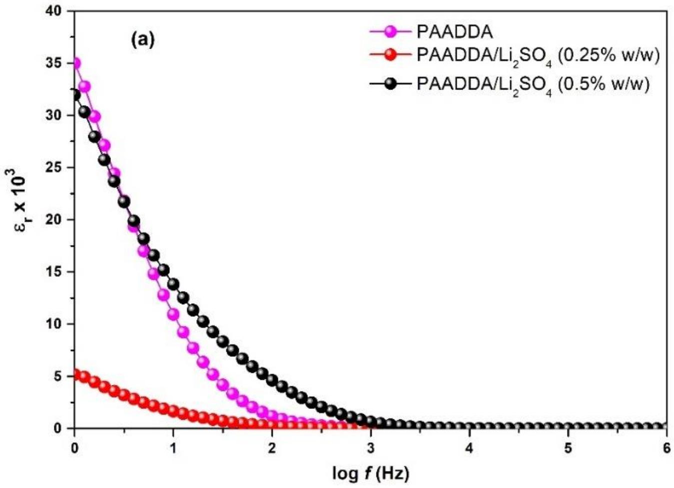
Furthermore, dielectric constant (ε_r), and the dielectric loss (ε_i) of all the obtained films were also determined. The ε_r is associated with the storage of energy while ε_i indicates the loss of energy due to flowing of charged entities, ionic conduction, or conversion into thermal energy (Abdul Halim *et al.*, 2021) in the films. The ε_r and ε_i values were calculated using the equations:

$$\varepsilon_r = \frac{Z_i}{\omega\, C_0(Z_r^2 + Z_i^2)} \qquad (2)$$

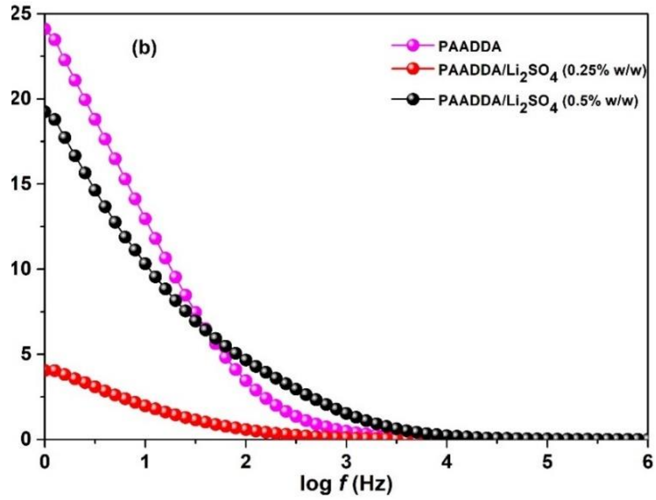
$$\varepsilon_i = \frac{Z_r}{\omega\, C_0(Z_r^2 + Z_i^2)} \qquad (3)$$

Where: Z_r is the real part of impedance, Z_i is the imaginary part of impedance, C_0 is the geometric capacitance, and ω is the angular frequency.

The dependence of the ε_r and ε_i values of the pristine PAADDA and PAADDA/Li_2SO_4 films with the frequency range are exhibited in Figures 4 (a) and (b), respectively. As can be observed in Figure 3 (a), there is a decreasing of the ε_r and ε_i values with the frequency that is attributed to difficulty of the dipoles to rotate quickly which provides a lag between the frequency of oscillating of the dipoles and the applied field.



(a)



(b)

4. (a) Dieletric constant (ε_r) and (b) dieletric loss (ε_i) as function of log*f* for pristine PAADDA film and PAADDA/Li_2SO_4 films.

For the low frequency region, the high ε_r and ε_i values are related to the interfacial polarization effect (Hema *et al*., 2009). In addition, the highest ε_r and ε_i values were found for the pristine PAADDA and PAADDA/Li_2SO_4 (0.5% w/w) films. Some researchers have demonstrated that high ε_r values are very interesting to the development of supercapacitors (Brza *et al*., 2021; Nath *et al*., 2022).

Conclusions

In this paper, PAADDA/Li_2SO_4 films were successfully obtained by casting method from aqueous solutions and characterized by ATR-FTIR, TGA and EIS. The incorporation of Li_2SO_4 into PAADDA film affected its thermal stability and electrochemical properties. For the PAADDA/Li_2SO_4 (0.25% w/w) film, thermal stability and ionic conductivity decreased. In the case of the PAADDA/Li_2SO_4 (0.5% w/w) film, the thermal and electrochemical properties were improved. Therefore, the results obtained showed that PAADDA/Li_2SO_4 (0.5% w/w) film is a promising material for electrochemistry applications.

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