

Effect of Lithium Triflate (LiTf) Salt on the Structural and Electrochemical Properties of the Pectin-based Solid Polymer Electrolytes Films

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ABSTRACT

Currently, liquid electrolytes (LEs) that have been used in many electrochemical devices, face safety concerns due to leakage problems. Consequently, solid polymer electrolytes (SPE) consisting of a natural polymer host that is leak-proof, biodegradable, and flexible are widely studied. In the current study, the effect of various weight percentages (%) (i.e.: 10, 20, 30, 40, and 50 wt.%) of lithium triflate (LiTf) salt on the structural and electrochemical properties of pectin-based SPE films was investigated. The SPE films were prepared using the solvent casting technique. Solid, flexible, and self-supporting films of pectin-based SPE were successfully acquired by the addition of up to 50 wt.% of LiTf. As proven by FTIR analyses, there occur interactions between the lithium cation and the pectin coordinating sites (OH and C=O). These interactions minimized the formation of hydrogen bonding between pectin chains, hence explaining the formation of flexible films. Also, the polymer-salt interactions contributed to the improvement in the amorphous phase of the system. The minimized formation of hydrogen bonding, improvement in the amorphous phase, and the increased amount of charge carriers in the LiTf-doped pectin-based SPE had contributed to the increase in the ionic conductivity of the system with salt content. The highest conducting sample, P5 (50 wt.% LiTf) exhibited the ionic conductivity of $3.87 \times 10^{-5} \text{ S cm}^{-1}$ which was four orders of magnitude higher than pure pectin film of $2.24 \times 10^{-9} \text{ S cm}^{-1}$.



Keywords: Polysaccharides; Pectin; LiTf salt; Polymer Electrolytes; Flexible Electrolytes Films

INTRODUCTION

Liquid electrolytes (LEs) nonetheless remain prevalent in numerous applications owing to their high ionic conductivity. However, LEs have several drawbacks, such as safety concerns due to evaporation and leakage, environmental pollution, and limited battery design [1]. Solid polymer electrolyte (SPE) is a solvent-free system produced by dissolution of inorganic salt into a high molecular weight polymer matrix [2]. Although the ionic conductivity of SPE is low ($<10^{-6}$ S cm⁻¹) [3], it has high mechanical strength and stability which makes it a promising substitute for harmful LEs [4]. Aside from that, SPEs show a wider electrochemical and thermal stability range as well as light in weight [5]. Also, SPEs are reported to be the best alternative to LEs [6-8]. Thus, SPE is used in this research instead of gel polymer electrolyte (GPE) and composite polymer electrolyte (CPE).

The classification of polymer hosts can be divided into two categories: natural and synthetic polymers. Synthetic polymers are basic chemical compounds that are polymerized in a laboratory to produce fibers. For example, Nylon, polyethene (PE), polystyrene (PS), synthetic rubber, poly (vinyl chloride) (PVC), Teflon, etc. [9-10]. Nevertheless, employing a synthetic polymer host in SPE results in environmental pollution caused by the significant accumulation of non-degradable waste [11-12].

Natural polymers are made from natural raw materials as their base materials. Thus, they are non-toxic, biodegradable, and sustainable. The natural polymers can be divided into three primary groups based on their origin: (i) plant's origin, (ii) animal's origin and (iii) microbe's origin [13]. The first category includes polymers like pectin, starch, guar gum, cellulose, hemicellulose, and agar [14-15]. The second group focuses on polymers made from animal's origins such as chitin, chitosan, gelatin, albumin, carrageenans and chondroitin [14-15]. Meanwhile, the third category involves polymers produced by microbes or bacteria such as dextran, poly(hydroxyalkanoate), hyaluronate, and pullulan [14-15].

Pectin is a complex polysaccharide that functions as a binding substance in the cellular structures of all plant tissues [16]. Among various natural polymers, pectin is an excellent choice as polymer host in SPE due to its abundance in nature [17]. This is due to its natural occurrence in citrus fruits such as berries, apples, plums, oranges, and guavas [18]. In addition, pectin contains oxygen atoms that can function as possible coordinating sites with ions of salt, hence providing conduction mechanism to the system [18].

The salts such as lithium salt, sodium salt and magnesium salt are usually added into the polymer host to provide the conducting species. Sodium batteries are more affordable than lithium-ion batteries. However, sodium has a lower energy density compared to lithium. The size of sodium cation has a negative influence on cathode materials, which raises more demanding requirements for embedding and removing it from cathode materials. So, over time, lithium has become more economical because of their extended lifespan [19].

Therefore, in this research, pectin of polysaccharide and lithium triflate (LiTf) salt were used as polymer host and ionic dopant respectively to produce flexible electrolyte films. According to Kumar *et al.* [20], the rise in ionic salt concentration contributes to the amorphous character of the polymer electrolyte, hence increasing its ionic conductivity. Nevertheless, at high salt concentration, the formation of ion pairs or aggregate ions leads to a decrease in free volume, thereby causes the decrease in ionic conductivity [21]. Therefore, in this study, the effect of the amounts of LiTf (i.e.: 10, 20, 30, 40 and 50 wt.%) on the structural and electrochemical properties of pectin-based SPE films was investigated.

EXPERIMENTAL

Chemicals and Materials

Pectin ($C_6H_{10}O_7$) (Galacturonic acid $\geq 74.0\%$) and LiTf ($LiCF_3SO_3$) were purchased from Merck (Darmstadt, Germany). All the listed chemicals were used without further purification.

Preparation of pectin-based PE films

By using solvent casting technique adapted from the study of Kumar *et al.* [20], 6 films were prepared as listed in Table 1. A total of 2.0000 g of pectin was dissolved in hot double-distilled water at roughly 80 °C using hotplate magnetic stirrer. Then, the various wt.% of LiTf salt (10, 20, 30, 40 and 50 wt.%) was dissolved in distilled water for a few hours at room temperature using magnetic stirrer. After that, the host polymer and ionic salt were mixed and totally dissolved by continuous stirring at room temperature. The resulting solutions were then cast onto a petri dish, and films were obtained by gradual drying at 60 °C inside the hot air oven. The calculation for mass of LiTf in relation to its weight percentage is shown as in Eq. (1).

Table 1: Composition of LiTf-doped pectin-based SPE films

Designation	Mass of pectin (g)	Mass of LiTf (g)	Percentage of LiTf (g)
Pure pectin	2.0000	0.0000	0
P1		0.2222	10
P2		0.5000	20
P3		0.8571	30
P4		1.3333	40
P5		2.0000	50

$$x\% = \frac{x}{100} = \frac{y}{y + 2.0000 \text{ g}} \quad (1)$$

where x equals to weight percentage of LiTf (i.e.: 10, 20, 30, 40 & 50 %) while y equals to mass of the LiTf.

Characterization techniques

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR (Thermo Fischer Scientific Nicolet iS 10) with Attenuated Total Reflectance (ATR) was used to determine the interactions occurred between pectin and LiTf in the SPE films. Firstly, the sample was positioned onto the crystal. Following that, the FTIR spectra of the sample was obtained

by conducting measurement at the wavenumber range of 4000 to 600 cm^{-1} at 16 scans and resolution of 2 cm^{-1} .

Electrochemical Impedance Spectroscopy (EIS)

The ionic conductivities of the pectin-based SPE films were determined using the EIS (HIOKI 35232-01 LCR). Prior to measurement, the micrometer screw gauge was used to measure the thickness of the films. Then, the film was put between the two stainless steel blocking electrodes. After that, at room temperature (25 °C), the impedance of the pectin-based SPE films were measured at three separate areas using a frequency range of 100 Hz to 1 MHz. The bulk resistance (R_b) of the samples were determined from the impedance plots obtained and the ionic conductivity (σ) of the samples were calculated using Eq. (2).

$$\sigma = \frac{l}{R_b \cdot A} \quad (2)$$

where R_b is the bulk resistance (Ω), l is the thickness of the film (cm) and A is the effective contact area of the electrode and the electrolyte (cm^2).

RESULT AND DISCUSSIONS

Formation of LiTf-doped pectin-based PE films

The pectin-based SPE films were successfully prepared with addition of lithium salt, LiTf into the polymer matrix. Figure 1 (a) shows the brittle structure of pure pectin film. This occurred due to the presence of OH groups in pectin that tends to form intermolecular interaction via hydrogen bonding, inducing ordered arrangement and high crystallinity which leads to the brittleness of the film. According to Ramesh *et al.* [22], the strong hydrogen bonding in the -OH functional group of the natural polymers causes high crystallinity, leading to brittle films. This statement was supported by the study done by Lin *et al.* [23], who reported that the hydrogen bonding in sodium carboxymethyl cellulose (SCMC) leads to its poor thermoplasticity. Solid, flexible, and freestanding films were successfully produced (Figure

1 (b)-(f)) after the addition of 10 – 50 wt.% of LiTf salt (P1 – P5). This might be due to the occurrence of the polymer-salt interaction as confirmed from FTIR analysis that will prevent the formation of hydrogen bonding between polysaccharide chains. The similar finding was reported by Kumar et al. [20] on the improvement in the amorphous nature and flexibility of the tamarind seed polysaccharide (TSP) film after the incorporation of LiTf.

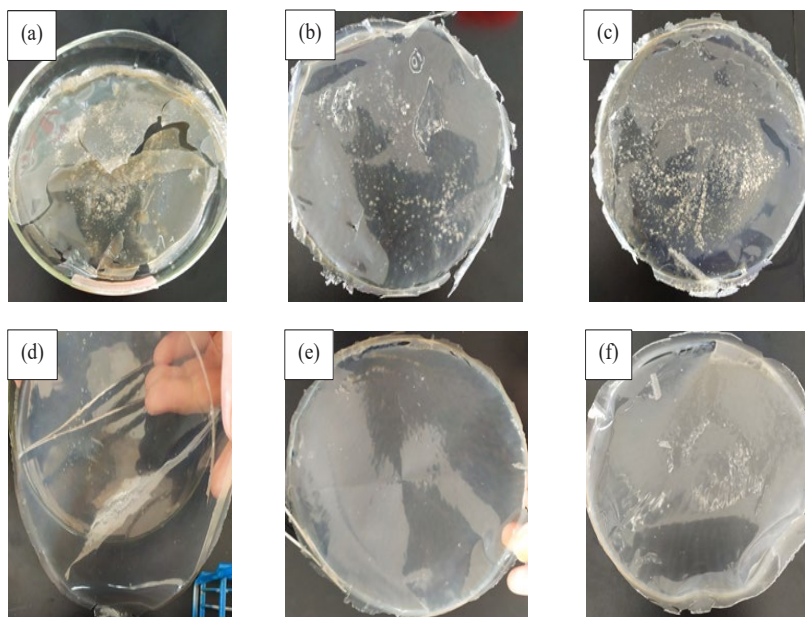


Figure 1: LiTf-doped pectin-based SPE films, (a) Pure pectin, (b) P1, (c) P2, (d) P3, (e) P4, (f) P5

FTIR studies of LiTf-doped pectin-based SPE films

Figure 2 displays the FTIR spectra for pure pectin, pure LiTf, and LiTf-doped pectin-based SPE films (i.e.: P1, P2, P3, P4, and P5).

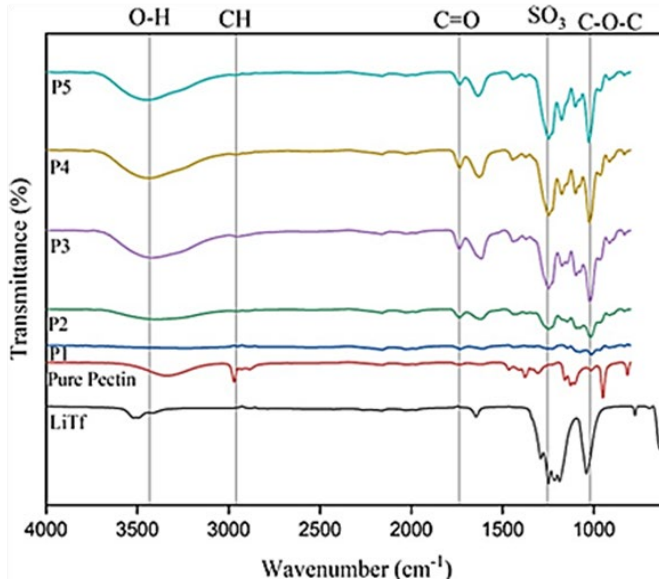


Figure 2: FTIR spectra of pure pectin, LiTf and LiTf-doped pectin-based SPE system

In the FTIR spectrum of pure pectin, the peaks were observed at 3547, 2971, 1736 and 1013 cm^{-1} representing O-H stretching, CH stretching, C=O stretching and C-O-C stretching, respectively. The same observation for the FTIR peaks of pectin was also reported by Vijaya *et al.* [24] and Santos *et al.* [25]. Meanwhile, for pure LiTf, the peak at 1249 cm^{-1} representing SO_3 stretching matched with the study reported by Chitra *et al.* [26].

After the addition of LiTf into pectin, the new peak representing SO_3 stretching of LiTf appeared at 1252 cm^{-1} , 1247 cm^{-1} , 1249 cm^{-1} , 1249 cm^{-1} , 1249 cm^{-1} for P1, P2, P3, P4 and P5, respectively. The appearance of this peak indicates that the LiTf was successfully incorporated into the pectin matrix. Due to that, after adding increased wt.% of LiTf, the shifting in the wavenumbers of O-H stretching (from 3547 cm^{-1} to 3435 cm^{-1}) (Figure 3), C-O-C stretching (from 1013 cm^{-1} to 1026 cm^{-1}) (Figure 4) and C=O

stretching (from 1730 to 1735 cm^{-1}) (Figure 5) were observed, along with change in their peak intensities. The change in the peak intensity of SO_3 stretching of LiTf as shown in Figure 6 was also observed. The wavenumber shifting and change in these peak intensities confirmed the occurrence of interaction between the oxygen atoms from hydroxyl (OH) and carbonyl (C=O) groups of pectin with the Li^+ ion of the salt. The same interaction was also reported by the other studies done by Kiruthika *et al.* [27] on pectin/magnesium nitrate ($\text{Mg}(\text{NO}_3)_2$) system and by Kumar *et al.* [20] on TSP/LiTf system.

It was also noted that the highest intensity of SO_3 stretching peak was observed for P5 that confirmed the presence of the highest number of charges in the sample (Figure 6). Thus, it is expected that P5 film will exhibit the highest ionic conductivity. Apart from that, the interaction between oxygen in OH group of pectin and Li^+ from the salt has prevented the formation of hydrogen bonding between polysaccharide chains. This explains the flexible polysaccharide-based SPE films obtained after the incorporation of LiTf (Figure 1 (b) – (f))

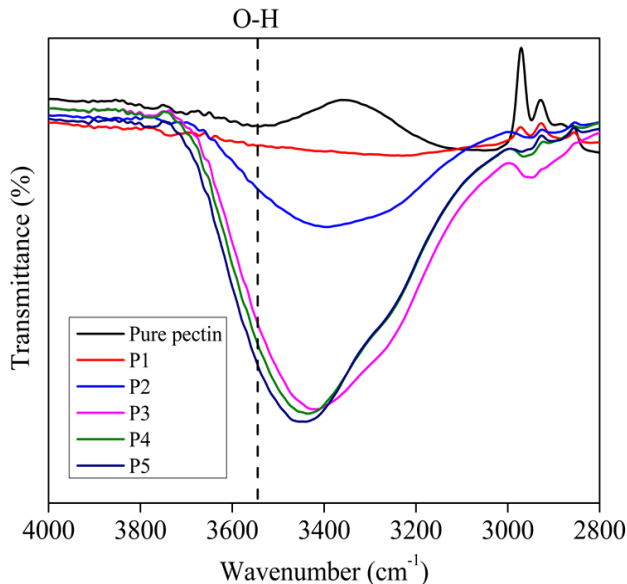


Figure 3: OH stretching peaks for LiTf-doped pectin-based SPE system

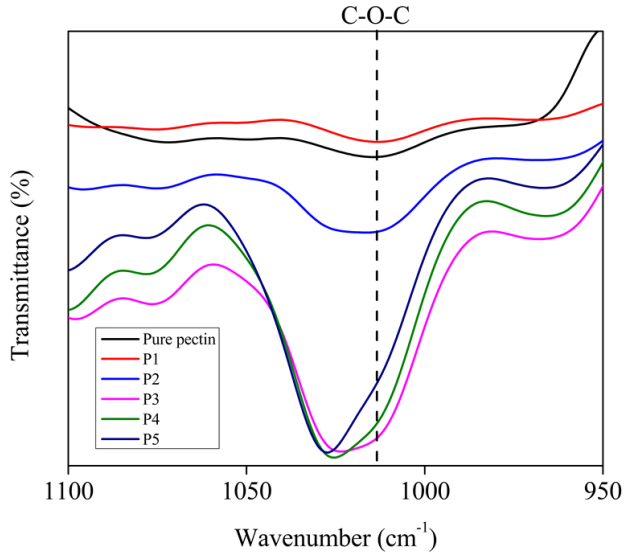


Figure 4: C-O-C stretching peaks for LiTf-doped pectin-based SPE system

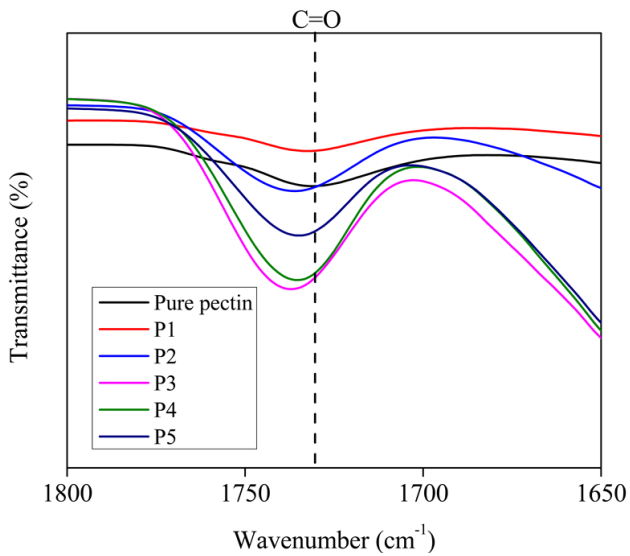


Figure 5: C=O stretching peaks for LiTf-doped pectin-based SPE system

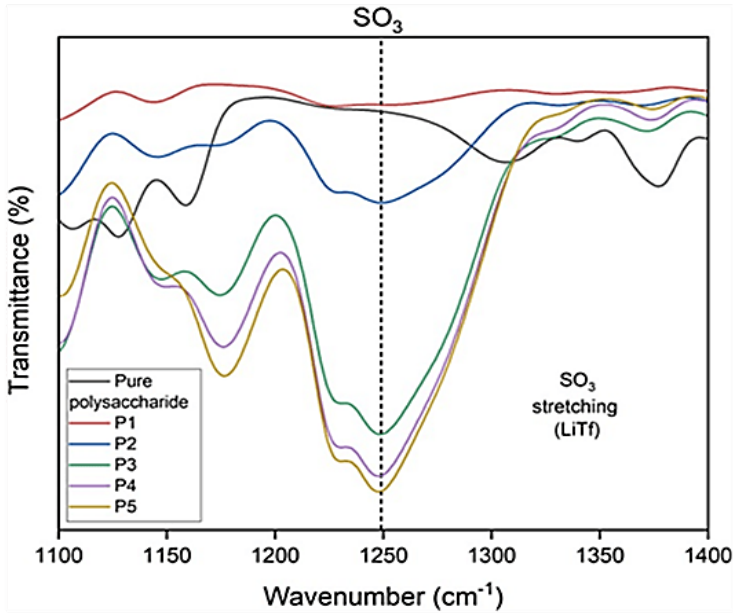


Figure 6: SO₃ stretching peaks for LiTf-doped pectin-based SPE system

Ionic Conductivity studies of LiTf-doped pectin-based PE films

Ionic conductivity is a significant characteristic of a polymer electrolyte [28]. A high ionic conductivity of SPE is primarily influenced by two key variables, namely (i) the concentration of ionic conducting species and (ii) the charge carrier mobility, which is impacted by the type of charge carriers (cation/anion) and temperature [29]. The Cole-cole plots produced following EIS measurement are shown in Figure 4.

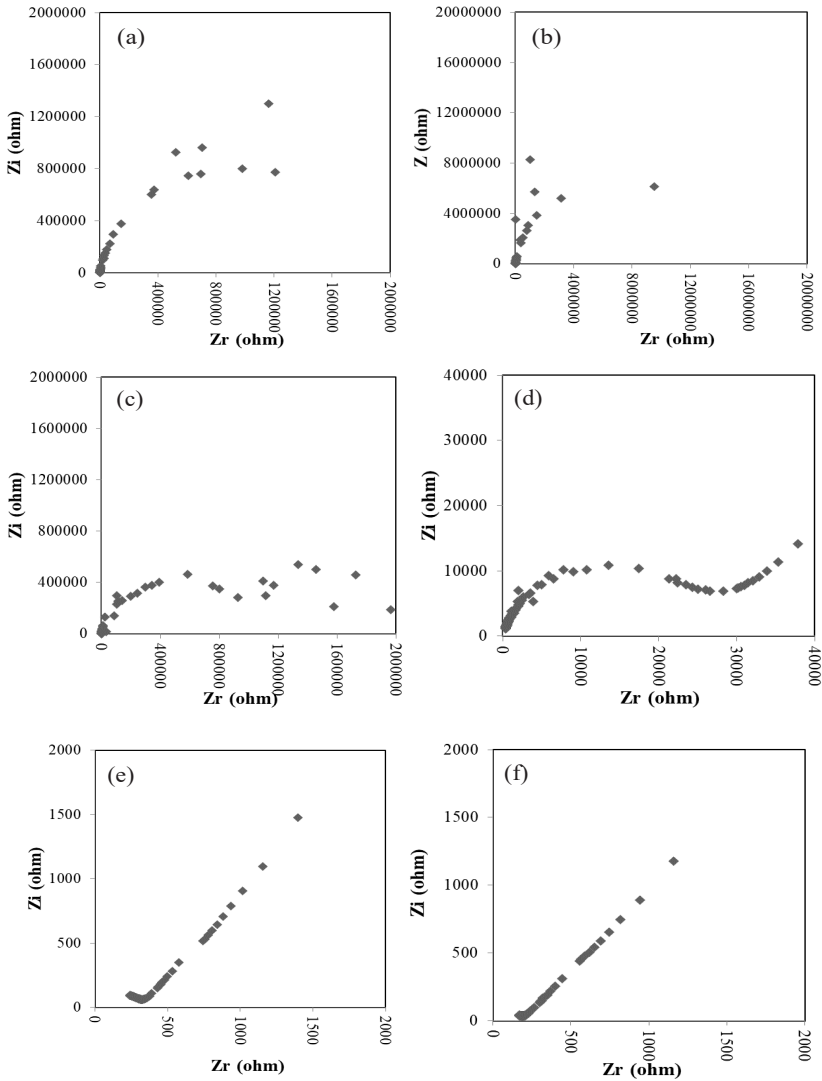


Figure 7: The Cole-cole plots for (a) Pure pectin, (b) P1, (c) P2, (d) P3, (e) P4, (f) P5

The Cole-cole plots for pure pectin and P1 plots show half semicircle (Figure 4 (a-b)) at high frequency. Meanwhile the plots for P2-P5 show depressed semicircles (Figure 4 (c-f)) indicating the occurrence of ion migration [30]. The exact semicircular patterns were not observed for pure

pectin and P1 indicating their low ionic conductivity. The same observation was also reported for the Cole-cole plots for polyvinyl alcohol (PVA)/polyvinyl pyrrolidone (PVP)/cadmium chloride (CdCl_2) system at room temperature [31]. The appearance of spike was observed for the Cole-cole plots of P2-P5 showing ion diffusion in the samples. When compared to other samples, P5 exhibited a spike with the highest slope, indicating a quicker rate of ion diffusion. This was also observed in the study done by Tang *et al.* [32].

The R_b was obtained by extrapolating the intercept of the spike to the semicircular region on the Zr axis. The ionic conductivity was then calculated using Eq. (2). The ionic conductivity values for LiTf-doped pectin-based SPE films are listed in Table 2. The ionic conductivity for pure pectin film was $2.24 \times 10^{-9} \text{ S cm}^{-1}$. The ionic conductivity then gradually increased for addition 10%-50% of LiTf (P1-P5). The highest ionic conductivity obtained was $3.87 \times 10^{-5} \text{ S cm}^{-1}$ which was four orders of magnitude higher than pure polysaccharide when 50 wt.% of LiTf was added. The rise in the ionic conductivity with salt content is due to the increase in number of charge carrier. This observation has been proven by the FTIR findings presented in Figure 6, which show that the P5 sample has the highest amount of Li^+ charge. The same observation was also reported by the study done by Zailani *et al.* [33].

Apart from that, the increase in the ionic conductivity can be related with the improved amorphous phase of the system due to the polymer-salt interaction as proven in the earlier FTIR analysis. In addition, the hydrogen bonds that cause the brittle structure of polysaccharide also has been minimized as the OH stretching of polysaccharide tends to interact with the Li^+ of the salt. These factors can contribute to the improvement of the ionic transport inside the polymer complex which then boost the ionic conductivity.

Table 2: Ionic conductivity for LiTf-doped pectin-based SPE system

Designation	Average Ionic Conductivity (S cm ⁻¹)	Standard Deviation
Pure pectin	2.24×10^{-9}	5.54×10^{-10}
P1	2.94×10^{-9}	4.03×10^{-9}
P2	2.94×10^{-9}	4.03×10^{-9}
P3	1.90×10^{-8}	1.12×10^{-8}
P4	1.92×10^{-7}	9.54×10^{-8}
P5	1.25×10^{-5}	9.34×10^{-6}

CONCLUSION

Solid, flexible, and self-supporting films of pectin-based PE were successfully obtained by adding up to 50 wt.% of LiTf salt into the pectin matrix via solvent casting technique. The flexible films were successfully produced due to the minimize formation of hydrogen bonding between pectin chains as the interaction between oxygen in OH group of pectin and Li⁺ from the salt occurred as confirmed by FTIR analysis. The ionic conductivity increased with increase amount of LiTf salt added. When 50 wt.% of LiTf was added, highest ionic conductivity was 3.87×10^{-5} S cm⁻¹, which was four orders of magnitude higher than pure polysaccharide. The minimized formation of hydrogen bonding, improvement in the amorphous phase and the increased amount of charge carriers are among the factors contributing to the improvement of the ionic conductivity of the LiTf-doped pectin-based SPE with salt content. Overall, the P5 sample has potential to be applied in lithium-ion batteries as its ionic conductivity ($\sim 10^{-5}$ S cm⁻¹) is sufficient for the requirement of the devices [34].

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