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Effect of LiTFSI Electrolyte Salt Composition on Characteristics of PVDF-PEO-LiTFSI-Based Solid Polymer Electrolyte (SPE) for Lithium-Ion Battery**Hasim Muzadi¹, Nayla Zahra Kamalia¹, Titik Lestariningsih², Yayuk Astuti^{1*}**¹Chemistry Department, Faculty of Sciences and Mathematics, Diponegoro University
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ABSTRACT. A lithium-ion battery with PVDF-PEO synthetic polymer sheet added by LiTFSI electrolyte salt has been made by assembling method. This study aims to determine the effect of LiTFSI salt concentration on the performance of lithium-ion batteries. The composition of LiTFSI electrolyte salts was varied into 5%; 10%; 15%; and 20%. Several characterizations were carried out to determine battery performance, including Electrochemical Impedance Spectrometry (EIS), Cyclic Voltammetry (CV), Charge/Discharge (CD), and Lithium Transference Number (LTN). The results showed that the synthesized separator sheet with a LiTFSI salt composition of 20% producing voltage, ionic conductivity, and lithium-ion transfer number of 0.72 V; $3.94 \times 10^{-8} \text{ SCm}^{-1}$; and 0.895, respectively is potential for lithium-ion batteries application. These results indicate the use of LiTFSI electrolyte salts with a concentration of 20% shows the best performance for PVDF-PEO-LiTFSI-based lithium-ion batteries.

Keywords: assembling method; lithium bis-trifluoromethane sulfonimide (LiTFSI), polyethylene oxide (PEO), polyvinylidene fluoride (PVDF)

INTRODUCTION

A battery is an energy storage device that converts chemical energy into electrical energy where electrons will move from the anode to the cathode. The battery must function effectively, and the raw materials must be simple, affordable, environmentally acceptable, and high capacity. Batteries are divided into two types, namely primary and secondary batteries (Putra et al., 2021). In a secondary battery, a reversible chemical reaction occurs. The battery will have a charging process when the electron moves from the cathode to the anode or there is a change in electrical energy into chemical energy. There is a discharging process where ions are intercalated from the anode to the cathode (Sabrina & Majid, 2017). The most advantageous type of secondary battery is a lithium-ion battery because it has a long lifecycle and has a higher potential, energy, and specific capacity differences than other secondary batteries (Karuppasamy et al., 2020). Battery arrangements generally consist of electrodes (anode and cathode), electrolyte, separator, container, and terminal (current collector).

Solid polymer electrolyte (SPE) is a substitute for liquid electrolytes due to its safety and dual function as an electrolyte and a separator. The basic materials for making SPE are polyvinylidene fluoride (PVDF) and polyethylene oxide (PEO). While the electrolyte salt used is lithium bis trifluoromethane sulfonamide (LiTFSI). LiTFSI is widely used in SPE development because it has excellent electrochemical properties, as well as chemical stability (Armand et al., 1990). In SPE, LiTFSI acts as a conducting salt and a provider of Li^+ ions since LiTFSI has a large anion that is TFSI^- which is highly delocalized to facilitate the dissociation and solubility of the salt in the SPE. Since TFSI^- is a highly delocalized anion and has a weak bond with Li^+ , Li becomes a free-moving and flexible ion in the polymer chain. This free Li^+ ion will then pair with the EO of the PEO which will then be transported in the SPE (Borodin & Smith, 2006). In addition, LiTFSI has a highly delocalized and flexible sulfonimide anion center ($-\text{SO}_2\text{N}(-)\text{SO}_2-$), which effectively reduces the interaction between TFSI^- anions and Li^+ ions (Mauger & Julien, 2022). Consequently, the distribution of Li^+ ions in SPE is getting better.

In addition, the melting point of PEO can be reduced by adding LiTFSI showing a high degree of salt dissociation due to the strong electron-withdrawing group, high flexibility, and excellent thermal, chemical, and electrochemical stabilities. Furthermore, the PEO-LiTFSI complex shows high ionic conductivity values (Xue et al., 2015). Previous studies have reported the difference of LiPF₆ and LiTFSI usage as additive salts in lithium-ion batteries (Dahbi, Ghamouss, An-Van, Lemordant, & Anouti, 2011). This includes the utilization of LiTFSI as an electrolyte salt capable of increasing the specific capacitance on carbon with increased ionicity in the EC/DMC binary system in which LiTFSI 1 M in EC/DMC is more ionic than LiPF₆ 1 M in the same binary solvent (Dahbi et al., 2011).

Other studies state that LiPF₆ as an electrolyte salt in lithium-ion batteries is less effective since its combination with some solvents makes LiPF₆ unstable at high temperatures and even decomposed into LiF and PF₅. PF₅ and LiPF₆ compounds can react with water to form HF, potentially damaging positive and negative electrode cells and significantly affecting battery performance (Lestariningsih, Prihandoko, & Wigayati, 2016). Related to this problem, using other lithium salts to replace LiPF₆ has become very important, and LiTFSI is the most promising candidate as a substitute for LiPF₆. LiTFSI can be used as an electrolyte in lithium-ion batteries due to its high conductivity, insensitivity to moisture, high thermal stability, and electrochemical stability compared to LiPF₆ (Septiana, Kar, Honggowiranto, Sudaryanto, & Hidayat, 2019). The ionic conductivity and lithium transfer number ideal of LiTFSI are above 10⁻¹ mS/cm (Goodenough & Kim, 2010); the ideal LTN is equal to 1 (Zhang et al., 2017); the cyclic voltammetry of electrodes with imide additives display higher current densities between 4.5 and 5.3 V vs Li/Li⁺ than the bare electrolyte (Sharova et al., 2018); CD analysis of LiTFSI showed that charge-discharge occurs. A study conducted by Liu et al. (2022) made a flexible composite solid electrolyte membrane (CSE) consisting of a poly(vinylidene fluoride) (PVDF) matrix, high concentration lithium salt (LiTFSI), solvent (DMF), and ceramic filler Li_{1.3}Al_{0.3}Ti_{1.7}(PO₄)₃ (LATP). In this research, an electrolyte membrane based on PVDF-PEO-LiTFSI was made without using a filler. However,

in this study, the concentration of LiTFSI salt was varied, namely 5%; 10%; 15% and 20% to be applied in lithium-ion batteries to know how the relationship between the concentration of LiTFSI salt which acts as a provider of Li⁺ ions and the ionic conductivity of lithium-ion batteries is related. This study also carried out several characterizations of EIS, CV, and CD. Also, a LTN test was carried out to find out how the transfer of lithium ions in Li-ion batteries increases with the increasing content of LiTFSI which acts as a provider of Li⁺ ions. Therefore, based on previous studies findings, this study aims to determine the effect of variation of LiTFSI salt concentrations on SPE to the performance of lithium-ion batteries identified by current values and battery conductivity. Lithium-ion batteries were characterized using EIS, CV, CD, and LTN.

EXPERIMENTAL SECTION

Materials and Equipment

The tools used in this study were tweezers, scissors, petri dish from IWAKI, fume hood, IKA Oven 125 Basic, magnetic stirrer, beaker cup from IWAKI, glove box from Fisher scientific, EIS instrument (HIOKI 3532-50), CV instrument (Automatic Battery Cycler (WBCS 3000)), digital multimeter type MASDA DT830B, Hydraulic Crimping Machine MSK-110, digital balance Sartorius ENTRIS224i-1S, and digital screw micrometer from EISCO.

The materials used in this study were separator membranes, caps, cans, wave springs, spacers, steel plates, Li metal (Li-metal), Poly (vinylidene fluoride) (PVDF) Aldrich brand, Lithium bis (trifluoromethane) sulfonamide (LiTFSI) Aldrich brand, Poly (ethylene oxide) (PEO) brand Sigma Aldrich, and N, N-Dimethylformamide (DMF) brand Sigma Aldrich.

Fabrication of Membrane Separator

The manufacture of separator membranes was carried out by mixing PVDF, PEO, and LiTFSI in a solution of 7.5 mL DMF with a PVDF content of 25%; PEO 75%; and LiTFSI salts by 5%; 10%; 15%; and 20% as shown in Table 1. Then the sample was stirred with a magnetic stirrer ± 3-5 hours until a slurry was formed. The formed slurry was poured on the printer glass and allowed to stand for ± 1-2 days at room temperature.

Table 1. PVDF PEO-LiTFSI-based Li-ion battery sample

Sample	Membrane Composition		LiTFSI
	PVDF	PEO	
A1	25%	75%	5%
A2	25%	75%	10%
A3	25%	75%	15%
A4	25%	75%	20%

Assembling Batteries

Cathode sheet, aluminum foil, and membrane separators were cut into a circle with a diameter of 19 mm. LiFePO_4 cathode and aluminum foil were weighed to determine the weight of the pure cathode, namely by reducing the weight of the cathode along with the aluminum foil by the weight of aluminum foil only. The lithium-ion battery casing consisting of a cap (top cap), can (bottom cap), wave spring/spacer, and steel plate was sterilized with an acetone solution. The battery casing was oven-dried for ± 15 -30 minutes, then continued with assembling the battery in a glove box pressurized with -0.01 mbar pressure with Argon inert gas. The preparation of battery components was carried out in the order of the bottom cap (can), LiFePO_4 cathode, then the separator membrane, then the Li-metal measuring $1 \times 1 \text{ cm}^2$, then covered with a spacer/wave spring, and the top cap. Furthermore, the battery crimping process was carried out at a pressure of 500 psi (kg/cm^2).

Characterization

The characterization of lithium-ion batteries includes EIS, CV, CD, and LTN testing. EIS testing used the EIS Instrument (HIOKI 3532-50) to study the electrical (conductive) properties. CV testing applied the CV (Automatic Battery Cycler (WBCS 3000)) Instrument to determine the oxidation and reduction reaction patterns of lithium-ion batteries and determine the current and voltage values when oxidation occurs. Moreover, reduction processes occur in lithium-ion batteries, and LTN testing applied WBCS 3000 instruments with a voltage limit of 1V. A testing time of 1000 second was to determine the number of Li ions migrating in the electrolyte and their contribution to battery performance.

RESULTS AND DISCUSSION

Membrane Separator and Battery Assembly

Manufacturing of solid electrolyte separator membranes based on PVDF-PEO-LiTFSI applied the solution casting method. The solution casting method is based on Stokes' law, where polymers and prepolymers both combine and make them dissolve in the appropriate solution (Kufian & Osman, 2021). This study used variations of LiTFSI salts to determine functions as a lithium salt or lithium-ion conductor from the anode on the electrochemical properties of SPE. This salt was used because it has a lower sensitivity to hydrolysis, high solubility, high ionic conductivity, and high electrochemical stability.

The lithium salt forms a complex when combined with PEO. Its ethylene oxide (EO) unit has a high electron donor number for Li^+ and high chain flexibility, which is important for driving ion transport. In addition, PEO has

a high dielectric constant and strong Li^+ dissolving ability. PVDF was chosen specifically to prepare PEO-based electrolytes due to its compatibility with PEO, excellent electrochemical stability, and high dielectric constant (Xue et al., 2015). The cathode used in this study was LiFePO_4 with a phosphor-olivine structure, a capacity of 170 mAh/g, specific energy of 659 Wh/g, a density of 3.60 g/cm^3 , an average voltage of 3.5 V, low price, non-toxic, environmentally friendly and good thermal stability (Gunawan, 2012).

The results of the SPE thickness in A1 were 0.0416 mm, A2 by 0.0536 mm, A3 by 0.0938 mm, A4 by 0.0716 mm, and A5 by 0.0774 mm. Meanwhile, the measurement of pure cathode weight of LiFePO_4 was carried out by reducing the weight of the cathode and coating with coatings only and was obtained for A1 by 0.02057 g, A2 by 0.02038 g, A3 by 0.02066 g, and 0.02038 g. The assembling process was undertaken inside the glove box with an arrangement from under the can (positive cap), LiFePO_4 cathode, SPE, Lithium metal anode, spacer, spring, and cap (negative cap). The glove box contains air gas so that no outside air was induced to enter and did not affect the electrochemical performance of the Li-ion battery to be tested (E. A. P. Putra, 2015). The battery arranged in the glove box was then removed and measured the voltage to be characterized with CV and EIS tests.

Electrochemical Behavior of Batteries

Electrochemical Impedance Spectroscopy (EIS) test was performed using EIS AUTOLAB to determine the ionic conductivity of the impedance or measure resistance to study the electrochemical properties of the battery without damaging the cells. The impedance value determines the electrochemical properties of the battery in the form of ion movement and ion conductivity. The test was carried out on assembling certificates, which were half-cell batteries using LiFePO_4 as cathode and Li-metal as anode. The results of the EIS test are presented in Figure 1.

Figure 1 shows a good chart, as there is no scatter, and it forms a semicircle pattern, and a straight line. The semicircle pattern indicates the presence of electrolyte resistance due to electrochemical reactions in the electrolyte (Ningsih, 2017). The determination of the conductivity value is obtained from the width of the charge-transfer resistance (R_{ct}). R_{ct} is the distance from R_e (the starting point of the semicircle pattern) to the endpoint of the semicircle pattern. The conductivity value is inversely proportional to the width of the pattern, where the wider the pattern, the smaller the conductivity, and vice versa. This is because the smaller the semicircle pattern the smaller the total resistance/impedance (Putri et al., 2017)

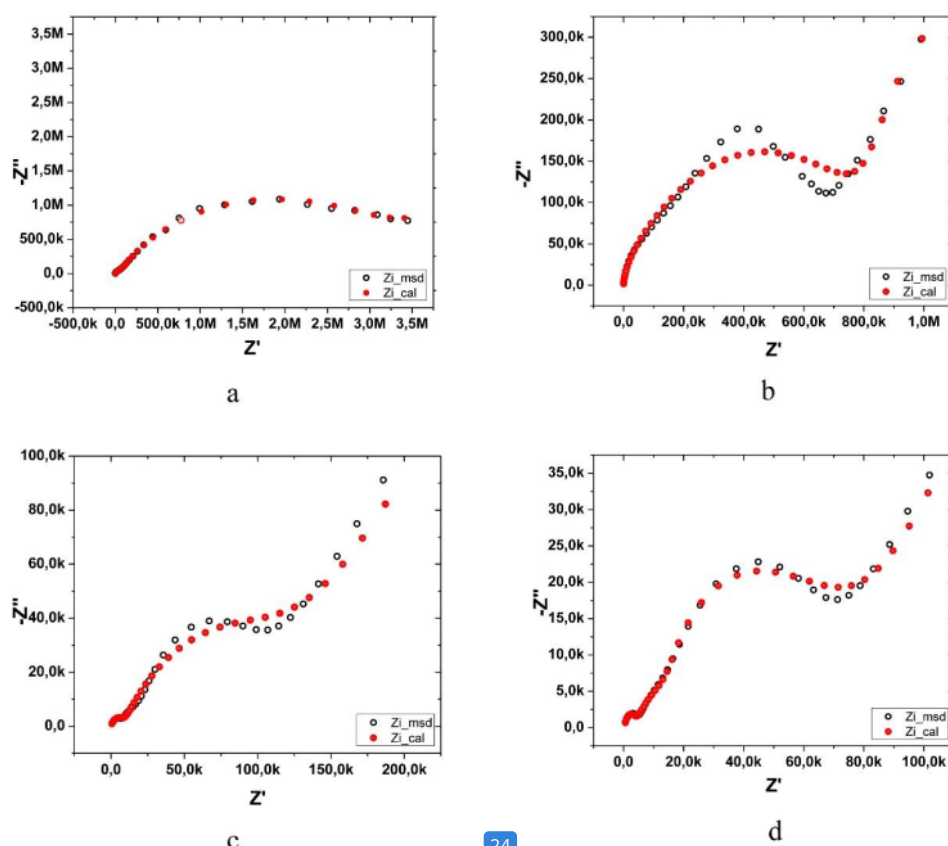


Figure 1. Graph of EIS test results of samples (a) A1, (b) A2, (c) A3, (d) A4

Table 2. Calculation of the value of ion conductivity

Sample	T (cm)	A (cm ²)	R _b (Ω)	σ (S cm ⁻¹)
A1	0.00536	283.385	709.617	2.665 × 10 ⁻⁸
A2	0.00938	283.385	870.418	3.803 × 10 ⁻⁸
A3	0.00716	283.385	653.008	3.869 × 10 ⁻⁸
A4	0.00774	283.385	693.022	3.941 × 10 ⁻⁸

Figure 1 indicates that the largest pattern width is sample A1 with a LiTFSI salt content of 5%, showing that the impedance or resistance of the sample is the largest and its electrical conductivity is the smallest compared to other samples. The ion conductivity of all samples is presented in Table 2. The calculation of the conductivity ionic value uses the following equation:

$$\sigma = \frac{T}{R_b \times A}$$

where T is the thickness of the electrolyte separator membrane (cm), A is the area of the effective area of the interconnected electrodes (cm²), and R_b is the bulk

resistance (Ω). The R_b value is derived from the Nyquist plot, which is the Z' (real) impedance plot versus the Z'' (imaginary) impedance (Osman et al., 2012). Using this formula, the results of the conductivity values of each sample can be seen in Table 2. Based on data in Table 2, the greatest ionic conductivity value is indicated in sample A4 with a LiTFSI salt content of 20%. A LiTFSI functions as a lithium salt or lithium-ion conductor from the anode to the cathode or vice versa. The more electrolyte salt content in the sample, the more lithium ions migrate (being delivered) or in other words, the mobility of Li ions is greater, and the ionic conductivity

increases. The higher the concentration of lithium salt, the greater the number of free lithium ions, and the TFSI anions also aggrag to form ionic groups, so that the battery ion conductivity is high and the electrochemical stability is good (Liu et al., 2022). By criteria, an electrolyte that can be used for Li-ion battery applications, its ionic conductivity value is above 10^{-1} mS/cm (Goodenough & Kim, 2010). The SPE ion conductivity value of the research results showed a low value due to the solid-state and high crystallinity (Stephan & Nahm, 2006).

Battery Reduction and Oxidation Cycle

The CV test was conducted using the cyclic voltammetry (Automatic Battery Cycler) WBCS 3000. The principle of the 102oltametric test is the measurement of current as a function of the potential applied when electrode polarization occurs so that the

battery undergoes reduction and oxidation. CV data shows not only cathodic and anodic peaks but also the cycles in which when the battery reaches a specific voltage; it will return to the initial voltage (Kim et al., 2020).

The CV curve describes the electrochemical process of the battery at the time of charging and discharging. The test results of A1, A2, A3, and A4 samples as presented in Figure 2 show the charging and discharging process of PVDF-PEO-LiTFSI SPE. The A1, A2, A3, and A4 charts show oxidation peaks and reduction. The oxidation peak is indicated by the upper peak, while the reduction peak is indicated by the lower peak. On A1 and A2 samples, the graphs show uneven images. This is because the data generated in the CV test is too small. The A3 and A4 images show smooth and clear pictures of their reduction and oxidation peaks.

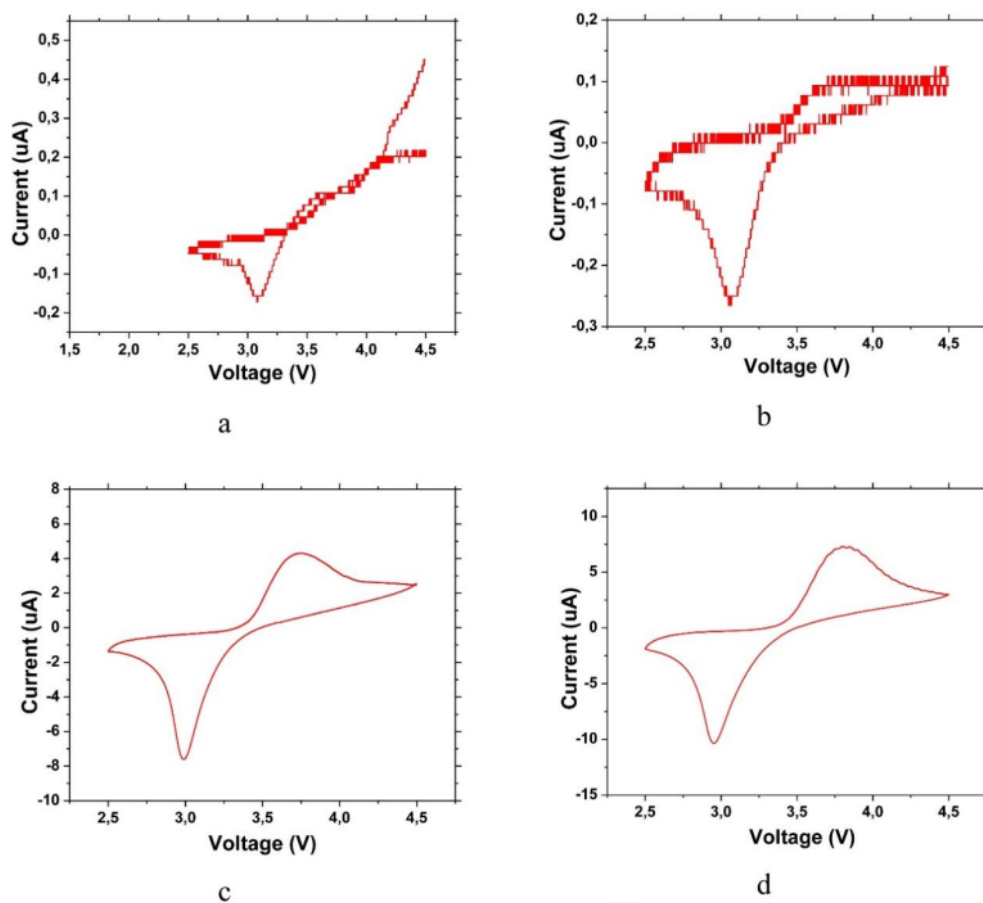
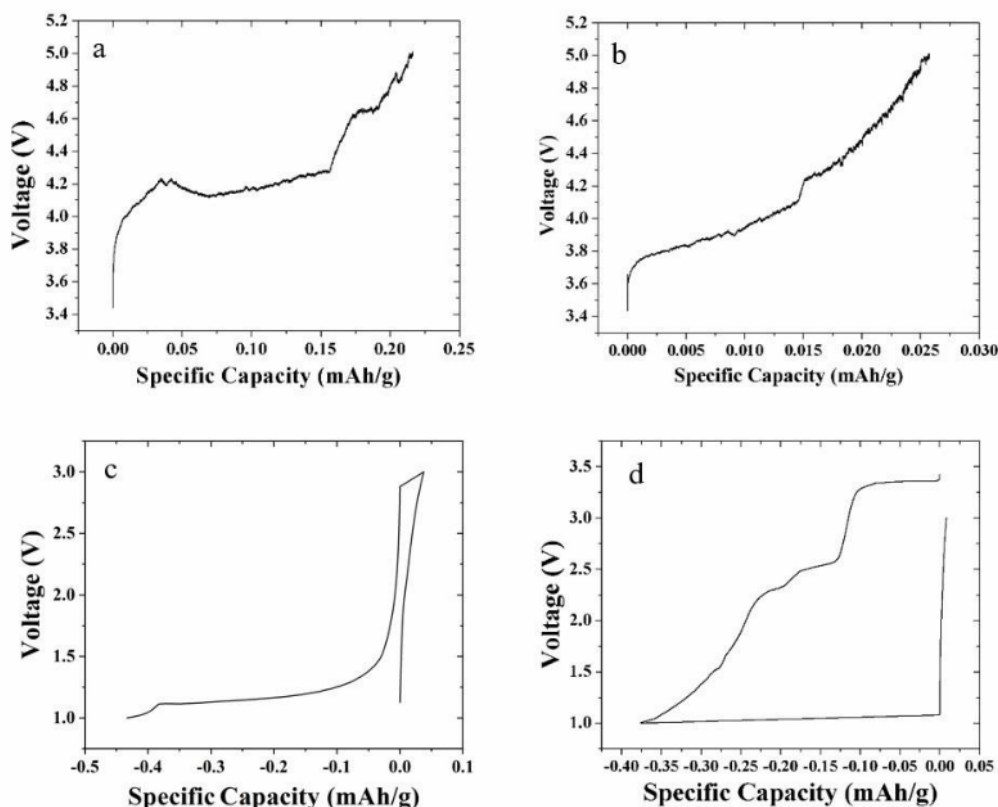


Figure 2 Curve of CV test results of lithium-ion battery of samples (a)A1, (b)A2, (c)A3, (d)A4.

Table 3. Current and voltage values from samples A1, A2, A3, and A4 CV test results

Sample	Current		Voltage		ΔV
	Oxidation	Reduction	Oxidation	Reduction	
A1	0.46642	0.046161	4.2268	3.131	1.096
A2	0.13963	0.23441	3.7868	3.0972	0.690
A3	3.7554	-5.3464	3.7594	3.0498	0.710
A4	5.7119	-6.7869	3.76	3.0354	0.725

**Figure 3.** Charge/discharge Li-ion batteries in samples (a) A1, (b) A2, (c) A3, (d) A4

According to the theory, ΔV represents the distance between oxidation and reduction peaks. From the calculation of the current and voltage values, the smaller the value of ΔV , the better the reversibility of the ions. It can be seen in Table 3 that the smallest ΔV value is that the A2 sample (10% LiTFSI) is better than A3 (15% LiTFSI), A4 (20% LiTFSI), and A1 (5% LiTFSI). However, the current generated in the A2 sample is of a minimal value. The proximity of the peaks will make it easier for Li-ions to intercalate or deintercalation.

Charge-Discharge

Charge-discharge testing was carried out to determine the ability of the battery to store energy. The charge-discharge graph is a relationship between specific capacity and resistance. One charge-discharge cycle consists of one charge (release of Li ions and electrons to the anode) and 1-time discharge (Li-ions and electrons enter the cathode).

On curves presented in Figure 3(a) and (b) there is no visible charge-discharge process, and only the

charge process occurs. This situation can be caused by a less-than-perfect charge-discharge process and the condition of the battery that begins to decrease in capacity, while a non-linear curve is caused because the battery has reached the maximum charge capacity limit. In samples (c) and (d) there is a Charge-Discharge process.

Lithium Transference Numbers

Lithium transference number testing ⁸ was carried out to determine the number of Li ions migrating in the electrolyte and their contribution to battery performance. LTN testing was performed assembly for assembling batteries using Li-metal as electrodes. During battery charge/discharge cycles, only Li⁺ migration contributes to battery performance. If the obtained LTN value is low, there will be local polarization and make Li⁺ deposition uneven. As a result, the life cycle and power density of the battery decrease. Therefore, the higher the LTN

value of an SPE, the better the battery performance. Overall, the ideal LTN is equal to 1 (Zhang et al., 2017).

Figure 4 shows that the initial total current (I_i) decreases with increasing time. In a stable state, the electron mobility has reached a saturation state, this is likely because the ionic current passing through the blocking ⁸ electrode drops drastically over time. Once a stable state is reached, only ions can be depolarized ⁵. In this case, lithium ions can move and provide a current flow from one electrode to another, in other words, the number of active charge carriers decreases and as a result, ⁵ the current value decreases as well. From the graph of the normalized polarization current against time, the transference number can be calculated using the following equation: (Mazuki., 2020).

$$t_{\text{ion}} = (I_i - I_f) / I_i$$

Where t_{ion} is the value of lithium transference number, I_i is the initial current (uA) and I_f is the final current (uA).

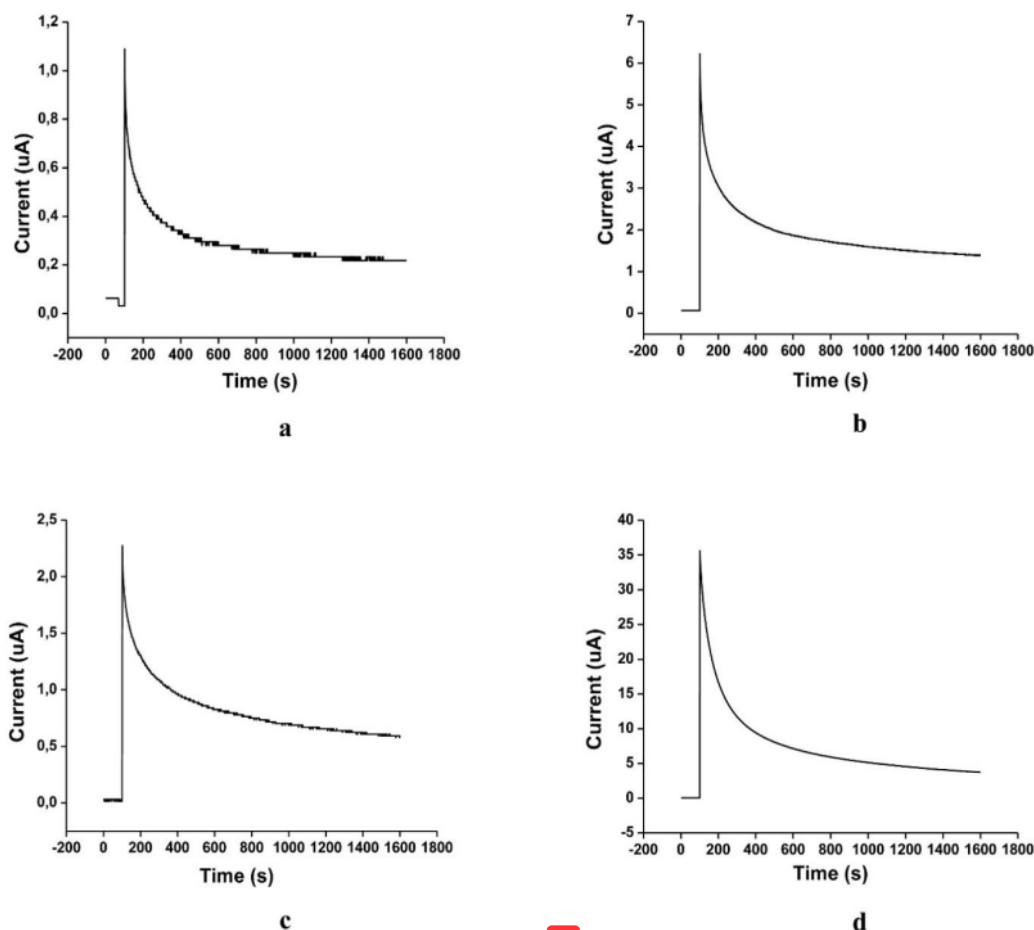


Figure 4. Lithium transference number curve of samples (a) A1; (b) A2; (c) A3; (d) A4

Table 4. Lithium-ion transfer value measurement results based on lithium transference number test

Sample	Initial Current (I_i) (μ A)	Final current (I_f) (μ A)	$t_{ion} = \frac{I_i - I_f}{I_i}$
A1	1.089	0.217	0.800
A2	6.225	1.385	0.778
A3	2.275	0.576	0.747
A4	35.627	3.724	0.895

The results of the calculation of the transfer number of lithium ions from each coin cell battery are presented in **Table 4**. The highest lithium-ion transfer number obtained for A4 batteries is 0.895 which contains 20% LiTFSI salt. This result is in accordance with Gao (2021) reporting that the more electrolyte salts, the greater the transfer number of lithium ions. Large/high Li^+ transfer number values can be attributed to the formation of ionic aggregate clusters in polymer salt electrolytes, which significantly limit the movement of large anions [TFSI]² and only promote the transfer of Li^+ ions due to their smaller size (Gao et al., 2021). The high number of lithium-ion transfers is expected to reduce polarization and improve the performance of lithium-ion batteries.

CONCLUSIONS

The high content of LiTFSI salts in the separator in the battery can improve the performance of PVDF-PEO-LiTFSI based SPE for lithium-ion battery. The synthesized separator sheet with a LiTFSI salt composition of 5% produces voltage, ionic conductivity, and lithium-ion transfer number is 1.096 V; $2.665 \times 10^{-8} \text{ S cm}^{-1}$; and 0.800, respectively. SPE with a LiTFSI salt composition of 10% produces voltage, ionic conductivity, and lithium-ion transfer number is 0.690 V; $3.803 \times 10^{-8} \text{ S cm}^{-1}$; and 0.778, respectively. Meanwhile, SPE with a LiTFSI salt composition of 15% produces voltage, ionic conductivity, and lithium-ion transfer number is respectively 0.710 V; $3.869 \times 10^{-8} \text{ S cm}^{-1}$; and 0.747. Furthermore, SPE with a LiTFSI salt composition of 20% produces voltage, ionic conductivity, and lithium-ion transfer numbers of 0.725 V; $3.941 \times 10^{-8} \text{ S cm}^{-1}$; and 0.895 respectively.

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