

# The Effects of LiBOB and LiTFSI Compositions on the Conductivity of PVDF-HFP based Electrolyte Polymer Membrane for Lithium Ion Battery Applications

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**Abstract**— This study is an effort to increase ionic conductivity of electrolyte polymer membranes in Lithium ion battery applications by combining electrolyte salts. An electrolyte polymer membrane functions as electrolytes and a separator. Such a membrane is made using a solution casting method at room temperature and consists of Polyvinylidene fluoride co-hexafluoropropylene (PVDF-HFP) as the polymer, titanium oxide ( $\text{TiO}_2$ ) as a filler, and combinations of Lithium bis(oxalate) borate (LiBOB) and Lithium bis-trifluoromethanesulfonimide (LiTFSI) as electrolyte salts. The electrolyte polymer membrane was characterized using Electrochemical Impedance Spectroscopy (EIS) for conductivity, X-Ray Diffractometer (XRD) for material crystallinity, and Scanning Electron Microscope (SEM) for surface morphology. The results of EIS tests show that the sample containing 23% LiTFSI and 5% LiBOB has the highest conductivity ( $2,35 \times 10^{-5}$  S/cm) compared to the other samples. This finding is supported by the results from XRD, as well as SEM, analyses, in which the sample has the lowest crystallinity, which results in higher ionic mobility, and the formation of pores on the membrane surface with the diameter of  $\pm 2-5 \mu\text{m}$ . The results of the 4th order polynomial regression analysis shows that increased LiTFSI corresponds to increased conductivity, however increased LiBOB corresponds to decreased conductivity

**Index Terms**— Electrolyte polymer membranes, Ionic conductivity, LiBOB, LiTFSI, PVDF-HFP, and Solution casting,

## 1 INTRODUCTION

RESEARCH on electrolyte polymer membranes as one of battery components is currently in high gear along with the increasing uses of portable electronic devices. Polymer-based solid electrolyte materials (electrolyte polymer membranes) possess many advantages over other solid electrolyte materials. These advantages include, but are not limited to, flexibility to fit any design, ease of fabrication, and mechanical strength. Even though liquid electrolyte materials have higher ionic conductivity, but solid electrolyte replacement for liquid one is a very important endeavor for safety reason [1].

Wright *et al.* were the first to report on solid polymer electrolyte and showed that polyethylene oxide (PEO) polymer had the capacity to conduct electricity at room temperature [2]. Electrolyte polymer membrane is formed with a polymer matrix containing alkaline metal ions as the source of electrical charge. Ions with low masses, such as Lithium ion ( $\text{Li}^+$ ), would easily exchange charges. These charge exchanges occur as a result of segmental relaxation, in which charges move from one segment to the next in the polymer matrix. This event takes place more effectively when the polymer matrix is in amorphous phase [3], because at this

phase there are more empty spaces, which allow for ionic movements, than when the matrix is in crystalline phase. The disadvantage of the solid electrolyte polymer is its lower ionic conductivity, which is only  $10^{-8}$  to  $10^{-5}$  S.  $\text{cm}^{-1}$  at room temperature, compared to that of the liquid one, whereas the requirement for battery application is for the electrolyte polymer membranes to have ionic conductivity of  $\geq 10^{-4}$  S.  $\text{cm}^{-1}$  [4]. Therefore, ionic conductivity becomes a significant characteristic in the development of this material. Extensive endeavors have been directed towards the development of this material. To increase the conductivity of the polymer, attempts have been carried out to modify the types of polymer by plasticization and composite formation by adding inorganic oxide materials as fillers [5].

Tripathi *et al.* [6] combined PVDF-HFP and PMMA, added NaI, and used solution casting method. They produced a matrix with a conductivity of  $1,67 \times 10^{-2}$  (S/cm). Santhi *et al.* [7] showed the recent results that a conductivity of  $1.3 \times 10^{-3}$  S/cm. They utilized a combination of LiTFSI and  $\text{LiNO}_3$  with the Electro spinning method, PVDF-HFP polymer and  $\text{SiO}_2$  as ceramic filler.

In a previous study [8], an electrolyte polymer was prepared with the solution casting method, using PVDF-HFP as the polymer, LiBOB as the electrolyte salt, and  $\text{TiO}_2$  as the ceramic filler. The results showed that 2%  $\text{TiO}_2$  in these mixtures could increase conductivity, however it was below the required value for an electrolyte polymer membranes.

Various attempts to improve conductivity have been carried out by previous researchers, and as such, this study focuses on the modifications of the types of added electrolyte salts. This salts modification study is based on that previously performed by Chen *et al.* (2013). The study showed that electrolytes with a base component of LiTFSI and an addition of LiBOB in Li/LiFePO<sub>4</sub> half cells could reach more than 1000 cycles at high temperature of 60°C, whereas electrolytes with a

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base component of LiPF<sub>6</sub> could not reach 1000 cycles. The mixture of LiTFSI and LiBOB salts also had the capacity to reduce corrosion on the battery electrodes[9].

The Lithium salts used to make the electrolyte solution in this study are in the forms of LiBOB (Lithium bis(oxalato) borate) and LiTFSI (Lithium bis (trifluoromethanesulfonyl) imide) mixed with TiO<sub>2</sub> (Titanium(IV)oxide). The polymer used is PVDF-HFP (Polyvinylidene fluoride-co-hexafluoropropylene) dissolved in DMAC to produce a polymer solution for the separator, which will be mixed with the electrolyte solution to form the electrolyte membrane. PVDF possesses high porosity and electrolyte absorption. PVDF use as polymer host matrix is more stable against hard chemicals and high temperature, whereas HFP co-polymer possesses the highest ionic conductivity compared to other mixtures [10].

The objective of this study is to determine the effects of LiTFSI and LiBOB compositions as electrolyte salt mixtures on the conductivity of electrolyte polymer membrane. The additions of these salt mixtures are expected to increase the conductivity of membrane

## 2 EXPERIMENTAL

### 2.1 Materials and Equipment

The materials used in this study consist of Polyvinylidene fluoride co-hexafluoropropylene (PVDF-HFP) from Sigma Aldrich as polymer, lithium bis(oxalate) borat (LiBOB) and LiTFSI (Lithium bis-trifluoromethanesulfonimide) from Sigma Aldrich as Lithium salts, and titanium oxide (TiO<sub>2</sub>) from Merck as filler. The solvent is NN-dimethylacetamide (DMAC) from Merck. The equipment used in the synthesis process includes magnetic stirrer, beakers, pipettes, and other glass wares including glass board. The apparatus for characterization includes LCR meter, XRD, and SEM.

### 2.2 Method

For There were two stages in generating PVDF-HFP-based electrolyte polymer membrane, which were production and characterization. The production used a weight ratio of 70% PVDF-HFP as polymer and 2% TiO<sub>2</sub> as filler, which was similar to the previous study [8]. In this study, there were eight (8) trials with varying concentrations of LiTFSI and LiBOB electrolyte salts, as shown in Table 1. The first trial was designated as sample A, second trial as sample B, and so on, until 8<sup>th</sup> trial as sample H. This study was intended to determine the effects of electrolyte salt concentrations on conductivity, therefore, only EIS test was performed on sample H (XRD and SEM characterization is not conducted on this sample).

#### 2.2.1 Production of electrolyte polymer membrane

Electrolyte polymer membrane was produced with solution casting method. The first stage in the membrane production was preparation of two types of slurry. Solution I contained a mixture of LiBOB and LiTFSI electrolyte salts with TiO<sub>2</sub> filler and in DMAC solvent, whereas solution II contained PVDF-HFP in DMAC solvent. The process of mixing and dissolving took place on a hot plate set at 60-70° C and was assisted with a magnetic stirrer set at 240 rpm. The two solutions were then

TABLE 1  
SAMPLE COMPOSITION

No	Sample code	Electrolyte salt composition	
		LiTFSI (w%)	LiBOB (w%)
1	A	0	28
2	B	5	23
3	C	10	18
4	D	14	14
5	E	18	10
6	F	23	5
7	G	28	0
8	H	0	0

mixed with the same procedure to form slurry for four hours. The resulting slurry was cast using solution casting method on a glass board and dried up at room temperature for ± 48 hours. The dry samples were stored in a desiccator or dry box before further uses and analyses.

#### 2.2.2 Characterization of electrolyte polymer membrane

Electrolyte polymer membrane was characterized for ionic conductivity, diffraction patterns, and morphology. The ionic conductivity analyses were performed with electrochemical impedance spectroscopy (EIS) using an LCR meter (HIOKI 3532150 chemical impedance meter). The diffraction patterns are evaluated using X - Ray diffraction (XRD) Rigaku (Smart Lab) at 2θ angle of 0 - 60°. The morphology of the membranes was analyzed with SEM (Jeol JED 350) at 2500x magnification. The data analyses employed polynomial regression to determine the correlation between electrolyte salt concentrations and conductivity.

#### 2.3 Conductivity Measurements

The ionic conductivity of electrolyte polymer membranes was determined from the measurement of impedance using Electrochemical Impedance Spectroscopy (EIS). The impedance was measured at the frequency interval of 100,000 Hz – 0.1 Hz. The data from the resistance measurements comprise of real part (R') and imaginary part (R''), which form Nyquist plots. Electrolyte polymer membrane resistance, R<sub>b</sub>, can be obtained from Nyquist plots. These plots of electrolyte polymer membranes from the various compositions of LiTFSI and LiBOB will form a half ellipsoid or semicircle. The initial point for the ellipsoidal pattern represents the electrolyte resistance (R<sub>e</sub>). The distance between R<sub>e</sub> to the final point represents the charge-transfer resistance (R<sub>ct</sub>). The width between R<sub>e</sub> and R<sub>ct</sub> determines the ionic conductivity of the battery, therefore the value of R<sub>b</sub> can be determined from this measurement. The ionic conductivity of electrolyte polymer membranes can be determined using the following formula:

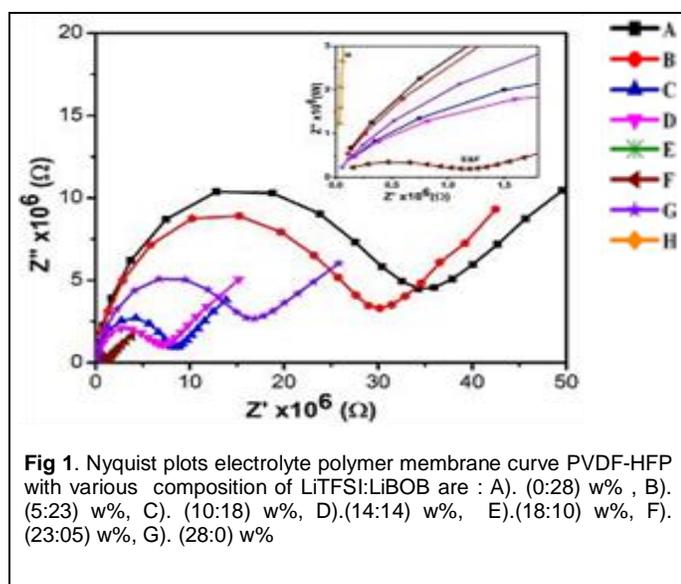
$$\sigma = t / (R_b \times A) \dots\dots\dots (1)$$

Where *t* is polymer membrane thickness (cm), *R<sub>b</sub>* is polymer membrane resistance (Ohm), and *A* is sample surface area (cm<sup>2</sup>).

### 3 RESULTS AND DISCUSSIONS

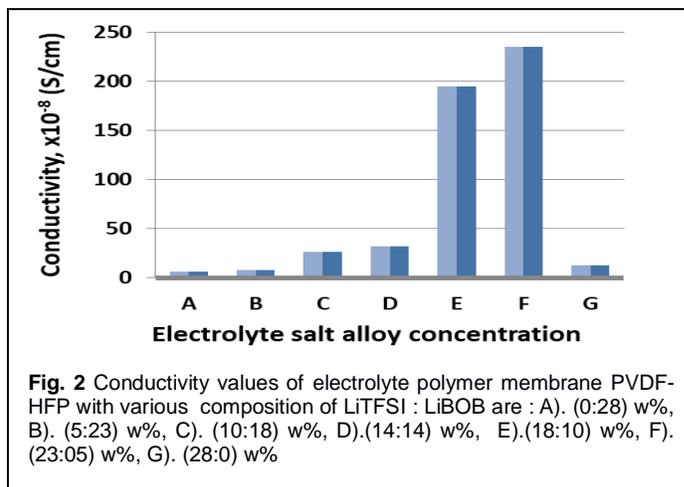
#### 3.1 Analyses of Electrochemical Impedance Spectroscopy (EIS)

Fig. 1 shows the Nyquist plots for electrolytes polymer membranes at various compositions of LiTFSI and LiBOB, which form a half ellipsoidal curve or semicircle. Based on Equation (1), the wider the semicircle, the lower the ionic conductivity because of an increased resistance. From the same figure, sample A (LiTFSI 5 w% and LiBOB 28 w%) is represented by the largest curve, which means that sample A exhibits the lowest ionic conductivity. On the other hand, sample F (LiTFSI 23 w% and LiBOB 5 w%) exhibits the highest ionic conductivity, as shown by the smallest curve, whereas sample H does not yield a semicircle pattern because it does not contain LiTFSI and LiBOB. LiTFSI and LiBOB salts play the roles of controlling the lithium ion transport medium.



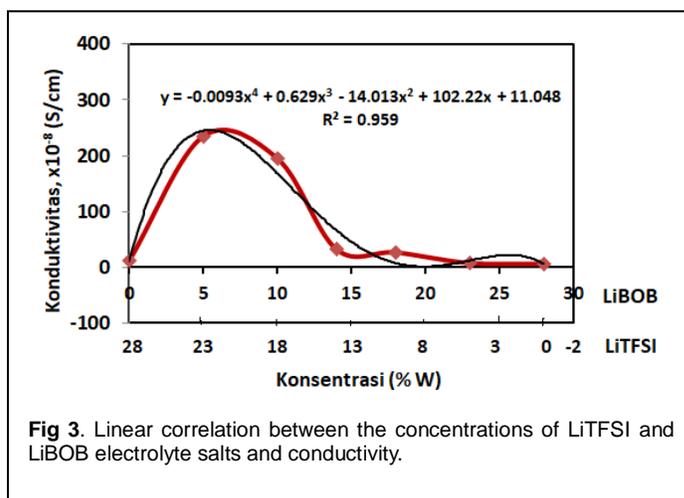
**Fig 1.** Nyquist plots electrolyte polymer membrane curve PVDF-HFP with various composition of LiTFSI:LiBOB are : A). (0:28) w% , B). (5:23) w%, C). (10:18) w%, D).(14:14) w%, E).(18:10) w%, F). (23:05) w%, G). (28:0) w%

The presence of these salts, therefore, will affect the conductivity of the samples. Using equation (1), the conductivity values for all samples can be determined as presented in Fig. 2. The different values show influence of the compositions of LiTFSI and LiBOB added. From Fig. 2 it is known that sample F, with the compositions of 23 w% LiTFSI and 5 w% LiBOB, exhibits the highest conductivity ( $2.35 \times 10^{-6}$  S/cm). This value is not significantly different from that of previous study by [8]. Tripathi *et al.* [10] developed their research by using a combination of PVDF-HFP and PMMA with the addition of NaI and yielded a conductivity of  $1.67 \times 10^{-2}$  (S/cm). Shanthy *et al.* [9] were able to yield a conductivity of  $1.3 \times 10^{-3}$  S/cm, by using the combinations of LiTFSI and  $\text{LiNO}_3$  salts on PVDF- HFP polymer and  $\text{SiO}_2$  filler processed with Electro spinning.



**Fig. 2** Conductivity values of electrolyte polymer membrane PVDF-HFP with various composition of LiTFSI : LiBOB are : A). (0:28) w%, B). (5:23) w%, C). (10:18) w%, D).(14:14) w%, E).(18:10) w%, F). (23:05) w%, G). (28:0) w%

Method [7]. By comparing our current study with those previous ones, we can see that the combinations of electrolyte salts, combinations of polymer types, ceramic filler types, and membrane production methods have significant effects on the resulting conductivity



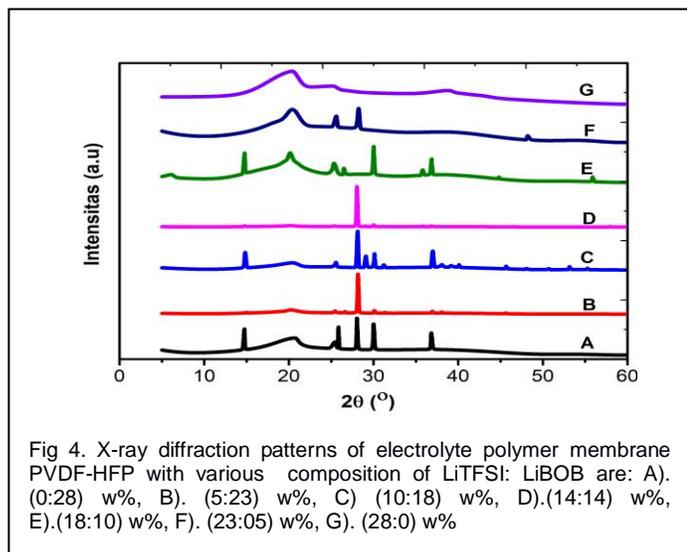
**Fig 3.** Linear correlation between the concentrations of LiTFSI and LiBOB electrolyte salts and conductivity.

Polynomial Order 4 is used to analyze and to determine the correlation between the concentrations of LiTFSI and LiBOB electrolyte salts and conductivity ( $\sigma$ ). In order 4 Polynomial of equation there are 2 independent variables controlled and 2 variables are not fully understood yet. The two independent variables controlled were LiBOB and LiTFSI concentration, while two variables which are not fully understood yet are the interaction between LiBOB - LiTFSI and an interaction between the two salts with the PVDF HFP polymer. This interaction needs further study. The addition of LiTFSI and LiBOB produce  $y = -0.0093x^4 + 0.629x^3 - 14.013x^2 + 102.22x + 11.048$  with  $R^2 = 0.959$ , where  $y$  represents conductivity and  $x$  represents the concentration of electrolyte salts. Furthermore, Fig. 3 shows that increased LiTFSI correspond to increased conductivity, however increased LiBOB corresponds to decreased conductivity. From the same diagram, the highest conductivity is shown to have the concentrations of electrolyte salts at LiTFSI 23w% and LiBOB 5w%. The absence of LiTFSI and LiBOB combinations are also shown to decrease

conductivity. This result confirms the findings of Chen *et al.* (2014), in which addition of LiBOB at a low concentration to LiTFSI-based electrolyte increased the conductivity of Lithium battery. They also demonstrated that the addition of LiBOB to LiTFSI-based electrolyte inhibited the corrosion on the current collectors that were made from aluminum and became more stable at high temperature than LiPF<sub>6</sub> based electrolyte. In reality, the membrane resistance ( $R_b$ ) resulting from EIS tests shows that the concentrations of LiTFSI and LiBOB electrolyte salt combinations influence the conductivity. In general, electrical ionic activity of electrolyte membranes is influenced by two factors, which are the concentrations of ions carrying the charges and mobility of those ions [10]. This result is supported by analyses of the crystal structure using XRD and morphological structure using SEM

### 3.2. Analyses of X-Ray Diffraction (XRD)

Fig. 4 shows the X-Ray diffraction patterns of electrolyte polymer membranes from various combinations of LiTFSI and LiBOB compositions. The formation of sharp peaks provides information on the characters of the samples, crystal structures, crystal orientations, crystal sizes, crystallinity, amorphous nature, and material phase transformation. The material formation, whether amorphous or crystalline, can be observed by analyzing one or two peaks of the diffraction pattern.

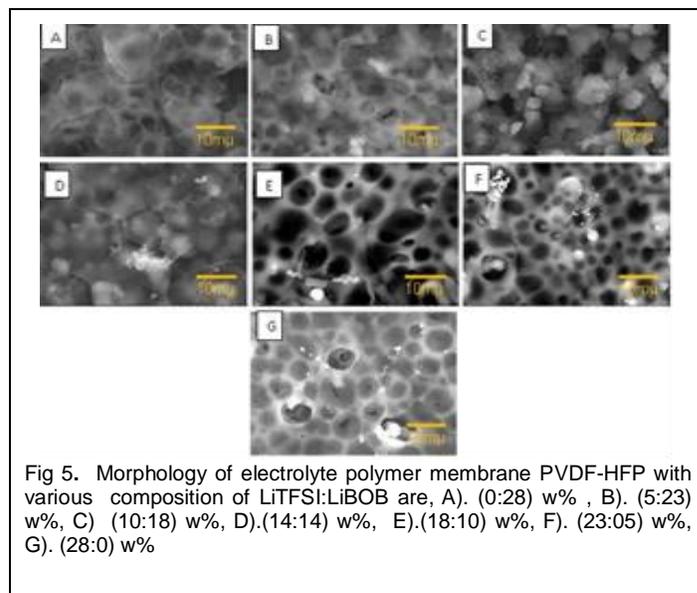


Hodge *et al.* (1996) observed that there was a correlation between the intensity of the X-Ray diffraction patterns and the degree of crystallinity. The decrease of intensity and disappearance of X-Ray diffraction patterns indicated an increase of amorphous [11]. Amorphous in polymer increases conductivity [4]. Charge transfer becomes more effective when the polymer matrix is in an amorphous phase. In an amorphous polymer matrix, there are numerous empty spaces that allow more effective movements of ions compared to those in a crystalline polymer matrix [4]. The peaks of X-Ray diffraction patterns for PVDF-HFP observed at 2θ angles of 20.4° and 38° indicate a semi-crystalline structure or co-existence of crystal and amorphous mixture [6]. According to the current references, when the peaks of X-Ray diffraction

patterns of LiTFSI electrolyte salt observed at 2θ angles of 14.11°, 15.91°, 18.61°, 18.91°, and 21.41°, the LiTFSI salt shows crystalline characters. This salt can be dispersed in a polymer matrix and all peaks that show the crystalline character will disappear and be replaced with one at 2θ angle of 14.11° [12]. In Fig. 4A, the peaks of X-Ray diffraction for the samples containing PVDF-HFP, TiO<sub>2</sub> and LiBOB can be found at 2θ angles of 14.7°, 20.1°, 25.2°, 28°, 31.2° and 36.8°. The addition of 5w% LiTFSI increases the intensity at 2θ angles of 25.2°, 29°, 36.8° and results in the appearance of new peaks after 2θ angle of 40° with low intensities. This phenomenon shows that reactions with LiTFSI have taken place. The addition of electrolyte salt combination at the same concentration causes a drastic increase of intensity at 2θ angle of 28°, as shown in Fig. 4D. Increase of LiTFSI and decrease of LiBOB, as shown in Fig. 4F, cause a decrease in intensity and disappearance of several peaks of X-Ray diffraction, which indicates an increase of amorphous character and a decrease of crystalline one. Sample F, therefore, with the composition of 23w% LiTFSI and 5% LiBOB, shows the highest conductivity at 2.35 × 10<sup>-6</sup> S/cm.

### 3.3 Analyses of Scanning Electron Microscopy (SEM)

The morphological structures of the samples were analysis using SEM. The results of electron microscopy scanning of the electrolyte polymer membrane surfaces at 2500x magnification are presented in Fig. 5. The SEM observation shows that all samples have undergone compatible mixing among polymer, salts and filler.



All samples have structure resembling porous honeybee hive, whether open or closed by chunks or grains and thin layers. The pores that are formed have the diameter of ± 2-5 μm. Aravindan *et al.* [13] developed their research using the same polymer with Al(OH)<sub>3</sub> filler yielded a membrane with pore size of ±0,265 μm. The pore size on commercially separator commonly used in Lithium Ion battery cell is < 1mm. Sample A (without the addition of LiTFSI) shows that some pores that have been formed are still thinly covered with layers (Fig. 5A). The addition of LiTFSI salt at 5% of weight causes coagulation of the thin layer, which in turn occludes those pores.

Increasing the concentration of LiTFSI and decreasing the concentration of LiBOB cause the opening of some of those pores, while other pores are still occluded by chunks previously formed, as shown in Fig. 5C. This blockade inhibits ionic movement, and manifests in low conductivity. The addition of LiTFSI and LiBOB salt mixture at the same concentration causes the breakdown of the thin layer covering the pores and ejection of chunks or grains blocking the pores. The addition of increased LiTFSI concentration causes the clearance of the pores from chunks or grains previously formed, and therefore ionic movement increases and results in sample F having the highest conductivity. Meanwhile, in sample G (without LiBOB) conductivity decreases. This phenomenon is caused by the formation of thin layers that cover most pores, as shown in Fig. 5G.

#### 4 CONCLUSION

Based on the results in this study, conclusions can be drawn that electrolyte polymer membrane PVDF-HFP was successfully produced with the combinations of LiTFSI-LiBOB electrolyte salts. The conductivity of electrolyte polymer membranes with the highest conductivity value obtained for sample F containing LiTFSI 25w% and LiBOB 5w% is  $2.35 \times 10^{-6}$  S/cm. This result is supported with analyses of XRD and SEM showing the sample to have the lowest crystallinity, which increases ionic movement due to the formation of pores on the membrane surface at the size of  $\pm 2-5 \mu\text{m}$ . The addition of LiTFSI and LiBOB produce  $y = -0.0093x^4 + 0.629x^3 - 14.013x^2 + 102.22x + 11.048$  with  $R^2 = 0.959$ , where  $y$  represents conductivity and  $x$  represents the concentration of electrolyte salts. From the 4th order polynomial regression equation show that increased LiTFSI corresponds to increased conductivity, however increased LiBOB corresponds to decreased conductivity..

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