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Limiting Molar Conductivity Dynamics of κ_{car}/PEO/$NaClO_4$ Aqueous Electrolyte System at Different Temperature

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The limiting molar conductivity behaviour (Λ_o) of κ_{car}/PEO/$NaClO_4$ blend liquid electrolyte systems in aqueous solution at 20 and 25 ºC was studied. The ionic conductivity (κ) of the electrolyte system was measured using Mettler Toledo Seven Compact S230 AC conductivity meter. The κ of the electrolyte was measured at a range of salt concentration, C_{salt} (10^{-5}-10^{-8}) and at different polymer concentration, C_{poly} (0.001-0.003 g cm^{-3}). The data analysis showed a directly proportion relation between Λ_o and C_{poly}, which indicates a decrease in total salt dissociation (α) and consequently, an increase in the formation of ion pairing occurs due to the increase in salt dissociation and less dynamic volume. In addition, a direct relation between Λ_o and temperature was also observed. An increase in temperature causes a decrease in the total ion pairing while increase the ion movement.

Keywords: Molar conductivity, Carrageenan, Polyethylene oxide, Sodium perchlorate, Electrolyte.

INTRODUCTION

Over the past few decades, studies in the field of secondary batteries has attracted huge attention from scientists all around the world [1]. Intensive researches have been carried out to obtain an optimum green energy resource derived from natural resources. Natural base polymer electrolyte systems are always the main subject of discussion in secondary battery systems over synthetic polymer electrolyte systems [2]. This is mainly because they are environmentally friendly and thus, they give minimum impact to the environment. In addition, natural polymers are biodegradable, hence, they do not contribute to an increase in the environmental waste. The employment of natural polymers can be a solution to solve global warming, pollution, shortage of petroleum and price fluctuation issues [3].

Polymer electrolyte is macromolecules with ability to transport charged species such as ions or protons [4] and it is prepared by doping a polymer host with inorganic salt [3]. Traditionally, acting as host, polymer electrolyte is used in the formation of synthetic polymers such as polyethylene oxide (PEO) [3], polyacrylic acid (PAA) [5] and polyvinyl alcohol (PVA) [6]. However, it has several disadvantages due to the fact that they are synthetic, high-priced, possesses mixability issue and difficult to degrade [7]. Therefore, polysaccharide has been extensively explored to reduce the manufacturing cost and pollution [2]. Regarding the effectiveness of polysaccharides like cellulose, agar, and chitosan as polymer electrolyte, a vast number of research papers are published [3]. Other than its similarity in structure and properties, therefore, in this study the kappa carrageenan (κ_{car}) is used to examine the ionic conductivity (κ) behaviour.

kappa-Carrageenan (κ_{car}) is a linear hydrophilic polysaccharide polymer formed by alternate units of D-galactose and 3,6-anhydrogalactose bonded by α-1,3 and β-1,4-glycosidic linkage [8]. The κ_{car} is extracted from several seaweeds from *Eucheuma cottonii* which is commonly found in Indonesia, Malaysia and Philippines [9]. Aside from being used as thickening, gelling and stabilizing agents in food products, κ_{car} is also used in drugs, pharmaceuticals and cosmetics [9]. There are three main types of carrageenan that have been reported in literature which are kappa (κ), iota (ι) and lambda (Λ) [10]. These three types of carrageenan differ in the number and position of half-ester sulphate group. The half-ester sulphate group in carrageenan is strongly anionic since it is negatively charged. The conformation of carrageenan in the polymer chain as well as the associated cation will determine the physical properties. For instance, κ and ι carrageenan bearing ester sulphate content of 25-30 % and 28-30 % respectively are in the form of gel in presence of potassium and calcium ions, however, Λ-carrageenan which contains 32-39 % ester sulphate content does not form a stable gel [11] since the number of ester sulphate is inversely proportional to the gel strength and solubility temperature [12]. The formation of film of carrageenan is possible *via* self-aggregation due to the significant number of sulfonic groups in its helical structures. The film produced possesses good tensile strength, gelling ability and transparency. It is also reported that among the three types of carrageenan, κ_{car} is non-toxic [13], thus, based on these reasons, kappa-Carrageenan (κ_{car}) was chosen in this research over other types of carrageenan.

In polymer electrolyte systems made up of κ_{car} as polymer host, the ions are able to move through its hydrogen-bonded network and the hydroxyl groups present in the polymer structure enables the formation of coordinate bonds with cations or protons, which might enhance ion mobility in the electrolyte system. However, naturally, κ_{car} has low κ (10^{-7} S cm^{-1}) [14], therefore, an alteration has to be done to enhance its κ. This can be accomplished by controlling the crystallinity of κ_{car} in solid polymer electrolyte (SPE) system since κ is greatly influenced by the degree of crystallinity of polymer [15]. It was reported that the κ increases with the decrease in crystallinity due to the expansion of free volume for ion transport [16]. According to Rhodes [15], ion movement mostly takes place in the amorphous phase while the crystalline phase, which has ordered arrangement and limited free volume hinder ion transport.

Polyethylene oxide (PEO) is commonly chosen as a polymer substrate due to its stable mechanical properties. It can form sturdy and flexible films, in turn hinder electrolyte leakage and secure excellent electrode contact [17]. Hence, PEO is used in this research to increase the stability of polymer electrolyte, furthermore, PEO is soluble with various solvents. This feature is crucial in making polymer blends in order to obtain a well-mixed and stable polymer blend. Although it is widely acknowledged that $NaClO_4$ will act as a strong electrolyte in aqueous solution, the addition of polymer prompts inquiry into whether the α will decrease or remain unchange. The salt dissociation (α) in the presence of polymer will elucidate the total free moving ions in the electrolyte, thereby influencing ionic conductivity (κ). The α might decrease due to ion pairing since the presence of polymer reduce the hydrodynamic volume.

In this work, the conductivity behaviour of κ_{car}/PEO/$NaClO_4$ polymer blend in liquid polymer electrolyte (LPE) system was examined and the findings can be an advantage for SPE system. The data obtained in this research can help build the SPE system with destabilize crystallinity and better ions move-ment for enhanced conductivity [18,19]. The κ_{car} and PEO polymer blend investigated in this work is in liquid phase. The solvent used in this polymer blend is water since both polymers are soluble in water and homogeneous [20,21]. Until now, there are few studies on the conductivity behaviour of κ_{car} in solution. Consequently, the information that is obtained from this study might be used as an added value and will contribute to knowledge advancement for a better understanding of ionic conductivity behaviour, especially in the amorphous phase.

The data on the ionic conductivity (κ)of LPE system in dilute condition can provide insight on the interaction between ions and polymers in amorphous regions of solid polymer electrolyte system [22]. However, the effect of polymer concentration (C_{poly}) on κ and α in polymer electrolyte membrane has been a secluded topic and unconfirmed theory. Therefore, the determination of κ and Λ_o for dilute LPE systems at various salt concentrations, C_{salt} could act as a method to understand the effect of C_{poly} on salt dissociation (α) [23].

Surprisingly, none of the researchers report on the molar conductivity behaviour (Λ_o) values. Moreover, no prior research was reported in determining the κ and Λ_o of κ_{car} and PEO blend. Therefore, in this study, we aim to determine the Λ_o behaviour of κ_{car}/PEO/$NaClO_4$ blend in liquid electrolyte system at 20 ºC and 25 ºC for C_{salt} (10^{-5}-10^{-8}) at polymer concentration, C_{poly} (0.001-0.003 g cm^{-3}).

EXPERIMENTAL

kappa-Carrageenan (κ_{car}) with > 99% purity (M_w = 788.647 $gmol^{-1}$) and PEO with > 99% purity (M_w = 100 kg mol^{-1}) were purchased from Sigma-Aldrich Chemical Company, USA. Anhydrous sodium perchlorate, $NaClO_4$ (M_w = 122.5 g mol^{-1}) with purity > 99 % was purchased from Merck, USA and dried in oven for 48 h at 100 ºC and vacuum condition to remove any traces of water prior to being dissolved in solvent. The solvent used was deionized water, which was prepared by pumping the water into a water deionizing system (Sartorious, Germany) producing 18 × 10^{-6} megohm at 25 ºC.

Conductivity: The ionic conductivity (κ) of the systems was measured using the Mettler Toledo Seven Compact S230 AC conductivity meter (Schwerzenbach, Switzerland). The probe used was Inlab 741, which possessed 0.001-500 µS cm^{-1} measurement range and 0-70 ºC temperature range. The conductivity meter's cell constant was established using an automated calibration process conducted on a daily basis. The aqueous solution of KCl provided by Mettler Toledo (Schwerzenbach, Switzerland) was used to calibrate the conductivity meter. The buffer solution was available in concentrations of 0.1, 0.01 and 0.005 mol L^{-1}, with associated κ values of 12.88 mS cm^{-1}, 1413 µS cm^{-1} and 84 µS cm^{-1}, respectively. The calibration procedure was conducted while the conductivity meter was linked to an Inlab® 741 electrode. After successfully calibrating the conductivity meter, electrolytic conductivity measurements were conducted on sample solutions at 25 ºC and 20 ºC. Each measurement was conducted at a minimum of three times (triplicate analysis) and later the mean values of ionic conductivity (κ) were reported, ensuring a coefficient of variation of less than 5%.

Sample preparation: The purification process was performed using precipitated method. In brief, 1.0 g of κ_{car} powder was dissolved in 99 g of distilled water. The mixture was allowed to swell and stirred for 48 h at 50 ºC under fume hood to form a homogeneous solution and later the solution was filtered. Rotary evaporator was used to extract the solvent and leave 1/3 residue of κ_{car} mixed solvent for the next process. The residue then underwent re-precipitation with *n*-hexane by the ratio of residue 1:5 *n*-hexane. The precipitate was then dried in oven under 50 ºC for 24 h. Similarly, purification of PEO followed the same procedure.

Preparation of $NaClO_4$ solution: An amount of sodium perchlorate salts was dissolved in 100 mL of deionized water and stirred at 50 ºC for 24 h. The stock solution was then diluted into a series of concentrations as tabulated in Table-1.

TABLE-1
VARIOUS C_{salt} UPON DILUTION WITH WATER

Initial C_{salt}, M_1 (mol cm^{-3})	Initial volume, V_1 (cm^3)	Final C_{salt}, M_2 (mol cm^{-3})	Final volume, V_2 (cm^3)
1.56×10^{-4}	10	3.12×10^{-5}	50
5.95×10^{-5}	10	1.19×10^{-5}	50
1.64×10^{-5}	10	3.28×10^{-6}	50
8.20×10^{-6}	10	1.64×10^{-6}	50
4.10×10^{-6}	10	8.20×10^{-7}	50
2.66×10^{-6}	10	5.32×10^{-7}	50
3.34×10^{-7}	10	6.67×10^{-8}	50
5.70×10^{-8}	10	1.14×10^{-8}	50

Preparation of polymer blend (κ_{car}/PEO/$NaClO_4$) in deionized water: The polymer blend was prepared with wt % of 50:50 ratios according to Table-2. The mixture was dissolved in 500 mL deionized water and stirred at 50 ºC for 48 h. These stock solutions were used to dissolve a known amount of $NaClO_4$ and stirred at 50 ºC for 24 h before being diluted into a series of concentrations. It was observed that phase separation in polymer blend will cause a significant problem that restricts the performance [24]. Hence, the 50:50 ratio polymer blend formed was carefully observed and mixed until a transparent solution with no phase separation was obtained. The solution appears homogeneous and the addition of salt does not cause any polymer salt-aggregation.

TABLE-2
VARIOUS C_{poly} IN WATER

C_{poly} ($\times 10^{-3}$) (g cm^{-3})	Mass of κ_{car} (g)	Mass of PEO (g)	Volume of deionized water (cm^3)
1.0	0.250	0.250	500
1.5	0.375	0.375	500
2.0	0.500	0.500	500
2.5	0.625	0.625	500
3.0	0.750	0.750	500

RESULTS AND DISCUSSION

Determination of Λ_o of $NaClO_4$ in deionized water after power law at 20 ºC and 25 ºC: As a result of strong thermodynamic hydration interaction between salt and water, $NaClO_4$ undergoes full dissociation, yielding a high concentration of free moving ions, particularly at dilute condition where the C_{salt} is less than 1.0×10^{-4} mol cm^{-3} as shown in eqn. 1 [25]:

$$NaClO_4\ (aq.) \longrightarrow Na^+\ (aq.) + ClO_4^-\ (aq.) \tag{1}$$

However, as the concentration increases above 1.0×10^{-4} mol cm^{-3} the possibility of ion pairing may happen. At higher concentration, the total free moving ions in the electrolyte decreases, which will cause a significant change in the ionic conductivity (κ).

The κ of the salt solution at various concentrations has been measured and the Λ_o at infinite dilution was determined after Kohlrausch's equation (eqn. 2).

$$\Lambda = \Lambda_o - KC_{salt}^{1/2} \tag{2}$$

The relationship between temperature and Λ_o is typically described by Kohlrausch's law of independent migration of ions. According to this law, the increase in kinetic energy of ions leading to greater mobility and faster migration of ions in the solution. As temperature rises, the ions move more freely, resulting in higher κ (Fig. 1) [26]. By comparing the Λ_o value of $NaClO_4$ salt at 25 ºC obtained from experiment (119.9 S cm^2 mol^{-1}) to the theoretical value (117.42 S cm^2 mol^{-1}) [27], it suggests that the measurement of κ was carried out in this research is reliable and accurate. The difference between the experimental to the theoretical value obtained is only 2.11%.

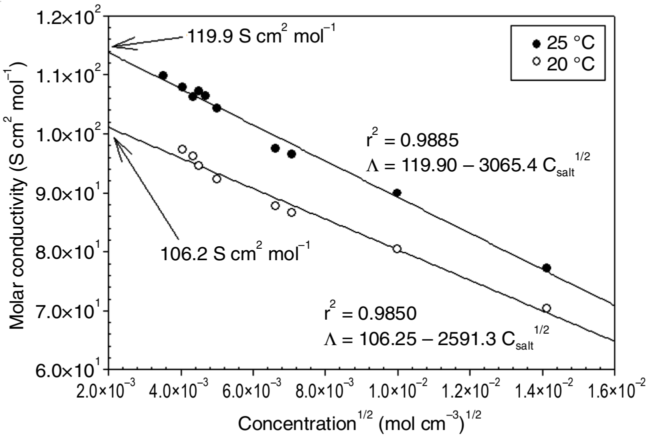

Fig. 1. Λ against $C_{salt}^{1/2}$ for $NaClO_4$ salt in aqueous solution

Kohlrausch's equation is widely used and accepted in determining the Λ_o for strong electrolyte, however, when approaching infinite dilution where the C_{salt} is zero, the Kohlrausch's equation is flawed since the extrapolation is deceptive. Λ_o is a κ value at $C_{salt} \approx 0$ mol cm^{-3}. The higher the dilution, the κ data measured will give more precise Λ_o value. However, the instrument such as conductivity meter has measurement limitation. Therefore, an alternative approach as suggested by Hanibah [25] named as Power law will be applied in order to obtain the Λ_o.

Power law is a double logarithmic plot of κ against C_{salt}.

$$\log \kappa = K' + \gamma \log C_{salt} \tag{3}$$

Eliminating log from eqn. 3 will yield eqn. 4:

$$\kappa = K'C_{salt}^{\gamma} \tag{4}$$

Since,

$$\Lambda = \frac{\kappa}{C_{salt}} \tag{5}$$

Hence, dividing eqn. 4 with C_{salt} yield eqn. 6:

$$\Lambda = K'(C_{salt})^{\gamma-1} \quad (6)$$

K′ denotes as the average ion mobility and it is a constant value over a range of C_{salt}. Hence, Λ_o of a system can be calculated at a selected C_{salt} known as reference salt concentration C_{ref}.

$$\Lambda_o = K'\gamma C_{ref}^{\gamma-1} \quad (7)$$

By substituting Λ_o, K′ and γ into eqn. 7, we can obtain C_{ref} value. It is obtained that the C_{ref} value for $NaClO_4$ at 25 ºC is approximately two times lower than the lowest C_{salt} value that follows linearity in Power law graph as suggested by Hanibah [25]. Therefore, a backwards calculation is done to determine the Λ_o of a system. The lowest C_{salt} value will be obtained from Power law graph, then, will be further convert into C_{ref} respectively and substituted into eqn. 7 to obtain the Λ_o.

From Power law plot (Fig. 2) for $NaClO_4$ (aq.) for 25 ºC,

$$\log\kappa = 1.3484 + 0.8536\ (C_{salt}) \quad r^2 = 0.9982 \quad (8)$$

$$\kappa = 22.30\ C_{salt}^{0.8536} \quad (9)$$

Rearranging according to eqn. 6 gives,

$$\Lambda = 22.30\ C_{salt}^{-0.1464} \quad (10)$$

From eqn. 9, K′ = 22.30 and γ = 0.8536.

Substituting K′, γ and Λ_o of $NaClO_4$ (aq.) at 25 ºC from literature which is 117.42 S cm^2 mol^{-1} into eqn. 7 gives the value of C_{ref}:

$$117.42 = (22.30)\ (0.8536)\ (C_{ref})^{-0.1464} \quad (11)$$

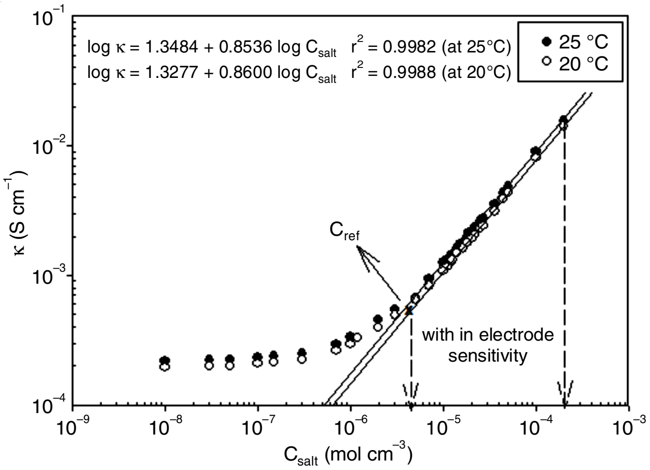

Fig. 2. Log κ against log C_{salt} for $NaClO_4$ salt in aqueous solution

From the above calculation, C_{ref} obtained is estimated at 4.00 × 10^{-6} mol cm^{-3}. Since C_{ref} is approximately half the lowest C_{salt} value that follows linearity in Power law, the lowest C_{salt} that follows linearity should be approximately 8.00 × 10^{-6} mol cm^{-3}. By referring to Fig. 2, it gives the lowest C_{salt} that follows linearity a value of 7.0 × 10^{-6} mol cm^{-3}. This value is within the acceptable range when compared to the calculated value. Therefore, it is suggested that Power law method is reliable and accurate to determine the Λ_o for this electrolyte system.

Now, by adopting the same method, C_{ref} value as estimated is recalculated for $NaClO_4$ (aq.) at 20 ºC according to eqn. 7.

From Fig. 2, C_{ref} is 7.0 × 10^{-6} mol cm^{-3}, K′ = 21.2667 and γ = 0.8600 and

$$\log\kappa = 1.3277 + 0.8600\ \log\ (C_{salt}) \quad r^2 = 0.9988 \quad (12)$$

$$\kappa = 21.2677\ C_{salt}^{0.8600} \quad (13)$$

Rearranging according to eqn. 6 gives,

$$\Lambda = 21.2677(C_{salt})^{-0.1400} \quad (14)$$

Substituting K′, γ and C_{ref} into eqn. 7 gives the value of Λ_o for $NaClO_4$ (aq.) at 20 ºC:

$$\Lambda_o = (21.2677)(0.8600)(3.50\times10^{-6})^{-0.14} \quad (15)$$

$$\Lambda_o = 106.18\ \text{S cm}^2\ \text{mol}^{-1}$$

The Λ_o for $NaClO_4$ (aq.) at 20 ºC was estimated using Power law and comparable to the value obtained using Kohlrausch's law (with 0.02 S cm^2 mol^{-1} difference), therefore, this shows the workability of Power law. The κ data from this experiment for strong electrolyte system fit well into Power law.

Determination of Λ_o of polymer blend of κ_{car}/PEO/$NaClO_4$ in deionized water after Power law at 25 ºC and 20 ºC: The blending of polymer was widely explored since the blending method can produce better flexibility and performance [24]. The 50:50 ratio is chosen since equal ratio of properties is desired in this liquid polymer electrolyte (LPE). Power law has also been applied to determine the Λ_o of the polymer blends at different C_{poly}. The regression function for the polymer blends at 20 ºC and 25 ºC are tabulated in Table-3.

Effect of salt concentration: It is observed from Fig. 2 that the κ increases linearly as the C_{salt} increases. Hence, according to Arrhenius law of free ions, it completely dissociates in water and produce individual ions due to strong thermodynamic interaction between water and $NaClO_4$. Nonetheless, it is also observed that the number of ions is not always proportional to the C_{salt} [28]. As the C_{salt} increases, the number of ions available per unit volume increases and consequently, more ions can move around in the electrolyte [29]. The charge transport of Na^+ ions is enhanced, resulting in the separation of Na^+ ions from their coordinating oxygen ions to a neighbouring site [30].

TABLE-3

SUMMARY OF THE Λ_o for κ_{car}/PEO/$NaClO_4$ POLYMER BLEND AT DIFFERENT C_{poly} AT TEMPERATURE 25 AND 20 °C

C_{poly} (× 10^{-3}) (g cm^{-3})	Linear regression function		Correlation (r^2)		Λ_o after power law (S cm^2 mol^{-1})	
	25 °C	20 °C	25 °C	20 °C	25 °C	20 °C
1.0	$\Lambda = 12.14\ (C_{salt})^{-0.2182}$	$\Lambda = 12.21\ (C_{salt})^{-0.2076}$	0.9985	0.9992	173.60	153.66
1.5	$\Lambda = 11.30\ (C_{salt})^{-0.2262}$	$\Lambda = 11.74\ (C_{salt})^{-0.2109}$	0.9996	0.9989	189.89	163.41
2.0	$\Lambda = 8.06\ (C_{salt})^{-0.2608}$	$\Lambda = 9.38\ (C_{salt})^{-0.2353}$	0.9981	0.999	192.15	164.75
2.5	$\Lambda = 7.42\ (C_{salt})^{-0.2680}$	$\Lambda = 8.85\ (C_{salt})^{-0.2410}$	0.9983	0.9993	192.99	178.39
3.0	$\Lambda = 5.64\ (C_{salt})^{-0.2982}$	$\Lambda = 5.50\ (C_{salt})^{-0.2901}$	0.9964	0.9981	196.88	186.24

Upon dilution, the κ deviates from linearity at certain dilute concentration. It is believed that the irregularities in κ obtained in this region is due to the limitation of conductivity meter. The fluctuation in κ observed is beyond the sensitivity capability of the probe. However, it is believed that this region will not affect our calculations and interpretations of the graph [25].

Effect of polymer concentration: Based on Table-3, the Λ_o increases as the C_{poly} increases for both temperatures. According to eqn. 16, Λ_o is inversely proportional to α [31].

$$\alpha = \frac{\Lambda}{\Lambda_o} \tag{16}$$

Thus, as C_{poly} increases, the α decreases, which is agreed by Szczκsna-Chrzan *et al*. [32] as observed in their findings. This might be caused by the increases in viscosity of the solution as C_{poly} increases as agreed by Walden's rule (eqn. 17).

$$K = \Lambda\eta \tag{17}$$

where K is a constant specific to temperature, Λ is molar conductivity and η is the viscosity.

Viscosity is the resistance of a fluid to flow. Hence, the increase in viscosity upon the increase in C_{poly} will decrease the κ since it restricts the movement of ions in response to the applied electric field [28]. It suggests that high viscosity controlled ion movement occurrs in the electrolyte system [32]. Furthermore, the increase in viscosity reduces the volume of free space in the solution. Hence, the ions are pushed together and results in the formation of ion-pairs [25].

Effect of temperature: Based on Fig. 2, the κ increases as the temperature increases. The κ of a system at 25 ºC is higher than 20 ºC. An increase in temperature weakens the dielectric constant of a solvent and lowering the viscosity of electrolyte [28]. A solvent especially those with low dielectric constant allows ions of one charge from associated with other ions of opposite charge. There is strong supporting evidence in literature [33] that supports the fact that Na^+ ion will pair with their counterions, ClO_4^- ion in concentrated solutions or in solvents with low dielectric current. An increase in temperature will weaken the dielectric constant by disrupting the orientation of solvent molecules to the applied electric field, predictively, κ will decrease due to the increase in ion association [28]. However, the increase in temperature also weakens ion pairing. This is due to the increase in thermal motions at high temperature, which makes the ions spread out over a larger area [34]. This implies the disruption of coordination between ions due to intense thermal motions. It seems that the increase in temperature breaks the ion pairing more efficiently than promoting ion pairing. Besides, as the temperature increases, the viscosity of electrolyte decreases [28]. Hence, the ions can move more freely and increase total ion transfer.

Conclusion

The utilization of Power law in this study was validated, demonstrating its effectiveness in determining the Λ_o of the LPE systems. The double logarithmic plot of κ against C_{salt} gives a linear graph with high linear correlation, r^2 value and the calculation performed results in data with high precision when compared to the theoretical value. The κ and Λ_o were measured for κ_{car}/PEO/$NaClO_4$ blend liquid electrolyte systems in aqueous solution at 25 ºC and 20 ºC. The data shows linear correlation between C_{salt} and κ, indicating an increase in total number of free ions as C_{salt} increases. Other than that, the decrease in α is observed as C_{poly} increases while an increase in temperature disrupts ion pairing, consequently increase the ion movement. The experimental results demonstrate the feasibility of employment of $NaClO_4$ salt in polymer electrolyte systems, displaying in a significant increase in κ as C_{salt} increases. However, the usage of κ_{car}/PEO is not advisable as it inhibits ion transfer. Moreover, it is more prominent as C_{poly} increases because ion pairing increases due to an increase in average viscosity of solution. Despite the achievement of this research, the sensitivity limitation of conductivity meter may impose constrains in these findings. Hence, for future research, more refine instrumentations should be explored to expand the understanding regarding this topic. By addressing these challenges, we can continue to advance the field of renewable energy and contribute to the global transition toward a more sustainable and environmental friendly energy landscape.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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