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Improvement of Electrical Properties of CMC-PVA Doped with Various Contents of LiNO_3 as an Application for Hybrid Polymer Electrolytes

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Abstract

The present work was carried out with the development of hybrid polymer electrolytes (HPEs) by using carboxymethyl cellulose (CMC)–polyvinyl alcohol (PVA) doped with different contents of lithium nitrate (LiNO_3) for the determination of their structural and conduction properties. The structural analysis was conducted by using Fourier transform infra-red spectroscopy and showed the interaction between the blend host polymer and ionic dopant, which formed via the coordinating site of CMC-PVA and $\text{Li}^+\text{-NO}_3^-$. The complexes of CMC-PVA doped LiNO_3 led to an increment in ionic conductivity, as observed by electrical impedance spectroscopy, and the sample containing 20 wt% LiNO_3 obtained the highest ionic conductivity of $3.54 \times 10^{-3} \text{ S cm}^{-1}$ at room temperature. The ionic conductivity at different temperatures (from 303 K to 343 K) was measured and found to obey the Arrhenius rule. The activation energies of the HPEs were computed based on the Arrhenius equation and were inversely proportional to the ionic conductivity. The highest ionic conducting sample was fabricated into an electrical double layer capacitor (EDLC). Cyclic voltammetry (CV) was used to study the electrochemical performance of the EDLC. A high value of C_s was obtained at 2 mVs^{-1} due to the utilization of ions in the vacant sites of the electrode material.

Abstrak

Peningkatan Sifat Listrik CMC-PVA Didoping dengan berbagai Konten LiNO_3 sebagai Aplikasi untuk Elektrolit Polimer Hibrid. Karya ini dilakukan dengan pengembangan hibrida polimer elektrolit (HPE) dengan menggunakan karboksimetil selulosa (CMC) -polyvinyl alcohol (PVA) yang diolah dengan berbagai konten lithium nitrat (LiNO_3) untuk penentuan sifat struktural dan konduksi mereka. Analisis struktural telah dilakukan dengan menggunakan spektroskopi Fourier Transform Infra-red (FTIR) dan menunjukkan interaksi antara polimer host campuran dan dopan ion telah terjadi melalui situs koordinasi CMC-PVA dan $\text{Li}^+\text{-NO}_3^-$. Kompleks CMC-PVA yang didoping LiNO_3 menyebabkan peningkatan konduktivitas ionik yang diamati dengan spektroskopi impedansi listrik (EIS) dan ditemukan bahwa sampel yang mengandung 20 wt% LiNO_3 memperoleh konduktivitas ionik tertinggi dengan $3.54 \times 10^{-3} \text{ S cm}^{-1}$ pada suhu kamar. Konduktivitas ion pada suhu yang berbeda diukur dari 303 K hingga 343 K dan ditemukan mematuhi aturan Arrhenius. Energi aktivasi HPE dihitung berdasarkan persamaan Arrhenius dan ditunjukkan berbanding terbalik dengan konduktivitas ionik. Sampel konduktivitas ionik tertinggi dibuat menjadi kapasitor listrik lapisan ganda (EDLC). CV digunakan untuk mempelajari kinerja elektrokimia EDLC dan mendapatkan nilai C_s yang tinggi pada 2 mVs^{-1} karena pemanfaatan ion di situs kosong bahan elektroda.

Keywords: hybrid polymer electrolytes, ionic conductivity, lithium nitrate, structural

1. Introduction

Electrical double layer capacitors (EDLCs) are one of the main types of electrochemical supercapacitors that can store electrical energy. EDLC offers a long lifetime and provides a high power density [1]. For the development

of promising EDLCs, electrolytes have become the most significant constituent because they act as media for ion transportation [2]. Natural and synthetic polymers can be used as electrolytes because they provide various advantages to the environment. These polymers were examined broadly in the manufacture of electrochemical

devices, such as super capacitors, solar cells, batteries, fuel cells, and sensors [3]-[11]. Carboxymethyl cellulose (CMC), chitosan, and starch are commonly used in the development of polymer electrolytes due to their substantial amount of natural resources, whereas synthetic polymers, including polyethylene oxide, polyvinyl alcohol (PVA), and polyvinylidene fluoride (PVDF), have an extraordinary ionic conductivity [1],[12]-[31]. However, most single-polymer electrolytes possess a low ionic conductivity, as proven by Mallick *et al.*, who showed that the ionic conductivity of gum arabic reaches $1.5 \times 10^{-6} \text{ S cm}^{-1}$ [32]. Nevertheless, a possible way to overcome this issue has been identified, that is, by introducing hybrid polymer electrolytes (HPEs). HPEs provide a number of benefits in electrochemical devices. These benefits include high operating temperatures, safety, and energy density, ease of application, and flexible geometry [8]. Dry solid polymer electrolytes have received great attention in the market as safer electrolyte alternatives [33]. These electrolytes have been extensively used in various electrochemical device applications.

Several ways can be implemented to enhance the structural and ionic conductivity properties of polymer electrolytes. Polymer blending mixes two or more different polymers and lacks the formation of covalent bonding. Polymer blending is a popular technique because of its low-cost technology and simple process [34]. Conductance property in solid polymer electrolytes can be improved by employing this technique. In addition, polymer blending can improve the mechanical stability and structure of polymers. Thus, this technique is used to synthesize CMC/PVA as the dressing material because it enables the observation of the healing process. Moreover, this process produces a semi-transparent and pliable blend [12]. Furthermore, El Sayed *et al.* reported that the properties of PVA can be enhanced if blending method is implemented with natural polymers such as CMC [35]. However, a previous work conducted by Saadiah *et al.* reported that the CMC blend with PVA-based HPEs possesses a low conductivity of $9.12 \times 10^{-6} \text{ S cm}^{-1}$. [36]. Thus, an ionic dopant was introduced into the CMC-PVA polymer blend system to improve the ionic conductivity [24]. The presence of an ionic dopant can change the polymer texture by increasing the softness and flexibility of the polymer during the improvement of the membrane chemical and mechanical stability.

In a recent work, CMC was selected as the primary host polymer given the studies on its use for polymer electrolytes [13],[15],[18],[37],[38]. CMC was selected because of its good biocompatibility, biodegradable, and water-soluble polysaccharide characteristics. In addition, CMC is extensively used as an additive to increase the standard of manufacturing products, such as paper, cosmetics, and industrial textiles [39]. CMC is an anionic

polysaccharide that can be derived from the etherification between hydroxyl groups and sodium monochloroacetate in an aqueous solution [39]. From the reaction, CMC obtains two functional groups, namely, a hydroxyl group [40] and carboxylate anion (COO^-), that provide the reaction between ionic dopants in a polymer electrolyte system [17]. PVA is another secondary host polymer used in HPEs due to its hydrophilic properties in the polymer; such properties are due to the existence of a hydroxyl group [40] in the carbon chain backbone [36], which forms H-bonding with CMC during the blending method. PVA has also been used extensively in previous studies of polymer electrolytes [28],[37],[41]-[45]. PVA also has several characteristics that are advantageous for HPEs, such as non-toxicity, biocompatibility, and photostability in the presence of ultraviolet-visible light [46]. Lithium nitrate (LiNO_3) is selected as an ionic dopant because it can be easily handled at room temperature, and it acts as an active material when used with polymers with biodegradable characteristics [9]. LiNO_3 has been previously demonstrated as a good liquid electrolyte and has been incorporated into polymer electrolytes [45,47-50]. Therefore, in the present work, CMC and PVA were selected as host polymer blends and doped with LiNO_3 . The structural and ionic conductivity of HPEs were determined by using Fourier transform infra-red (FTIR) spectroscopy and electrical impedance spectroscopy (EIS). The highest ionic conductivity of CMC/PVA- LiNO_3 was used to develop an EDLC, and its electrochemical performance was determined by cyclic voltammetry (CV).

2. Experimental

Materials. The materials used in the experiment were CMC, PVA, lithium nitrate (LiNO_3), and distilled water.

Sample preparations. CMC and PVA (~85% hydrolysis) were acquired from Acros Organic Co. (molecular weight (M.W.): 90000) and Merck Co. (M.W. 70000). Referring to the work of Saadiah *et al.*, a favorable conducting ratio of 80/20 CMC/PVA was used in the present work [36]. The CMC was dissolved in distilled water until homogeneity, followed by the addition of PVA into the solution. The mixture was stirred until the solution was totally dissolved. Then, different contents of LiNO_3 in the range of 10 wt% to 30 wt% were added to the polymer blend system and stirred constantly until the solution became homogenous. The solution was casted into different petri dishes and dried in oven until film formation. The samples were left in a desiccator for further drying process. Then, the samples were retrieved and further dried at room temperature until the films have formed. Table 1 shows the sample designation and mass of the materials used in the sample preparation of HPE system:

Table 1. Designation of CMC/PVA-LiNO₃ HPEs

Designation	Mass of CMC (g)	Mass of PVA (g)	LiNO ₃ Concentration (wt%)	Mass of LiNO ₃ (g)
CPL1			0	0
CPL2	1.6	0.4	10	0.2222
CPL3			20	0.5000
CPL4			30	0.8571

Sample characterizations. For structural study, FTIR spectroscopy was employed to examine the interaction of functional groups in the HPEs. The prepared samples were characterized by Perkin Elmer analysis in the wavenumber range of 700 cm⁻¹ to 4000 cm⁻¹, and the sample was placed on a germanium crystal configured at 2 cm⁻¹ spectrum resolutions. The spectroscopy transformed the collected spectrum data from the interface pattern into the spectrum. Meanwhile, the ionic conductivity and electrical properties of the HPEs system were investigated using EIS. The impedance spectroscopy was performed via HIOKI 3532-50 LCR Hi-TESTER. Small disk samples of 2 cm diameter were used and flanked between two stainless steel electrolytes under spring pressure. The HPE system was tested from 303 K to 343 K with frequencies at the range of 50 MHz to 1 MHz. The ionic conductivities of the samples were identified by using Equation (1):

$$\sigma = \frac{\tau}{R_b A} \quad (1)$$

where t and A are the thickness and area of the samples, respectively, and R_b is the bulk resistance generated from the complex impedance spectroscopy. For the EDLC development, the electrode was prepared by using 80:10:10 ratio of activated carbon (AC BP20): super P: PVDF and with N-methyl-2-pyrrolidone as the solvent. The mixture was stirred until homogenous and spread on an aluminum foil by using the doctor blade technique. Next, the electrodes were dried in a vacuum oven for 24 h. Then, the sample with the highest ionic conductivity was fabricated into an EDLC coin cell and tested using CV. Autolab Potentiostat was used from 0 V to 1 V at different scan rates of 2, 4, 10, 20, and 40 mVs⁻¹ at room temperature. The specific capacitance (C_s) from the CV measurement was calculated by using the following:

$$C_s = \int_{V_1}^{V_2} \frac{I(V)dV}{2(V_2 - V_1)mv} \quad (2)$$

where $I(V)$ is the current, $(V_2 - V_1)$ is the potential window, m is the mass of active material, and v is the scan rate.

3. Results and Discussion

Structural studies. In the FTIR analysis, the interaction between the host polymer blend and ionic dopant can be observed based on the complexation in the FTIR spectra. Figure 1 shows the FTIR spectrum of pure LiNO₃. According to Jinisha *et al.*, for pure LiNO₃, strong absorption bands occur at 830, 1051, and 1374 cm⁻¹, which are represented by the out-of-plane deformation, symmetric stretching, and asymmetric stretching of NO₃⁻, respectively [9].

Figure 2 displays the spectrum of FTIR for CMC/PVA doped with different contents of LiNO₃. In the present work, the CMC/PVA-LiNO₃ sample showed a highlighted peak, which is believed to be where most interactions occurred. Figure 3 shows the FTIR spectra of the HPE system at focusing regions: a) 800–1200, b) 1200–1800, and c) 2700–3500 cm⁻¹. The peaks at 1056, 1320, 1584, 1730, 2935, and 3285 cm⁻¹ of CPL1 belonged to bending C-O-C, -OH bending, asymmetric COO⁻, C=O stretching, -CH stretching, and -OH stretching, respectively [40,51-55]. As can be observed in Figure 3(a), the peak intensity at a wavenumber of 822 cm⁻¹ for CPL3 decreased upon the addition of LiNO₃, confirming the interaction between CMC/PVA and LiNO₃. According to Kesavan *et al.*, increasing the LiNO₃ content into the system decreases the width of vibrational band, which is similar to the observed finding [56].

The vibrational band at 1052 cm⁻¹ was indicated by the bending C-O-C and known as an ether linkage. Ether linkage consists of an oxygen site that can create intermolecular H-bonding with another molecule [14]. Figure 3(a) shows that the band shifted from a low to a higher wavenumber with the addition of LiNO₃ content. This shifting can be explained by the interaction between the C-O-C functional group in the CMC/PVA and Li⁺ ion and the formed ion-dipole interaction [57]. Thus, this situation confirmed the interaction between the blended host polymer and ionic dopant.

Figure 3(b) shows the peaks at 1320 and 1584 cm⁻¹, which corresponded to the -OH bend and -COO⁻ asymmetric stretching, respectively. Both peaks slowly decreased with the addition of LiNO₃ exceeding 20 wt%. The vibrational band of -OH bending shifted from 1320 cm⁻¹ to 1323 cm⁻¹. This result can be explained by

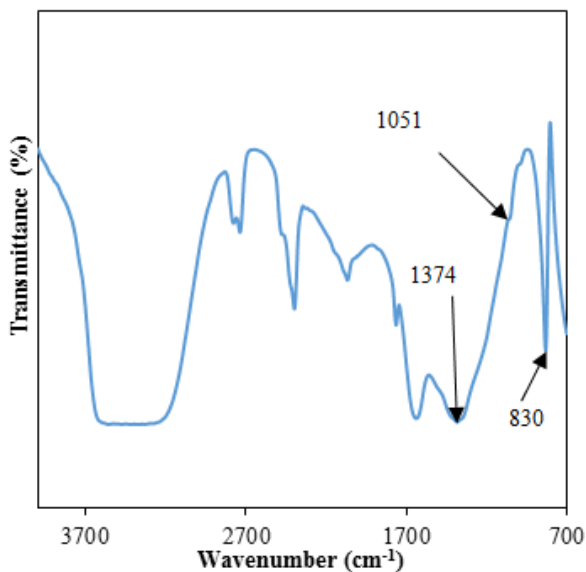


Figure 1. FTIR Spectrum of Pure LiNO₃

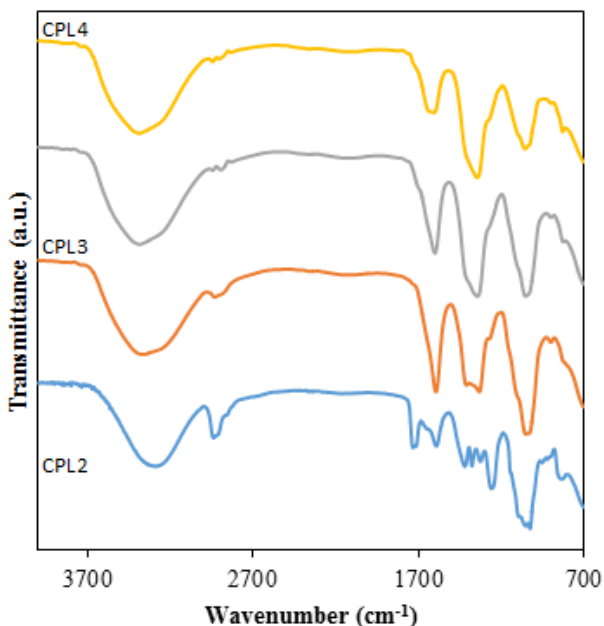


Figure 2. FTIR Spectra of CMC/PVA-LiNO₃ System

the interaction between the -OH functional group with Li⁺ that dissociated from LiNO₃, leading to the formation of Li-OH [12]. A shifting of the vibrational band from 1584 cm⁻¹ to 1601 cm⁻¹ was observed for asymmetric COO because the electron pairs of oxygen atoms can coordinate with the cation of the ionic dopant [47]. Figure 3(b) also reveals the appearance of a new peak at 1730 cm⁻¹, which belongs to the C=O stretching, and it disappeared when 10 wt% LiNO₃ was added to the system. However, a small hump reappeared for the CPL3 and CPL4 samples. The existence of stretching C=O in the system was due to the partially hydrolyzed

acetate group belonging to PVA [14]. The sudden appearance of C=O functional group at the peak occurred because of the dipole-dipole interaction with oxygen, which resulted in a highly electronegative atom [12]. In addition, the oxygen atom from C=O exhibited a strong intermolecular force with lithium ion, which indicates the complexation between the host polymers and ionic dopant [49].

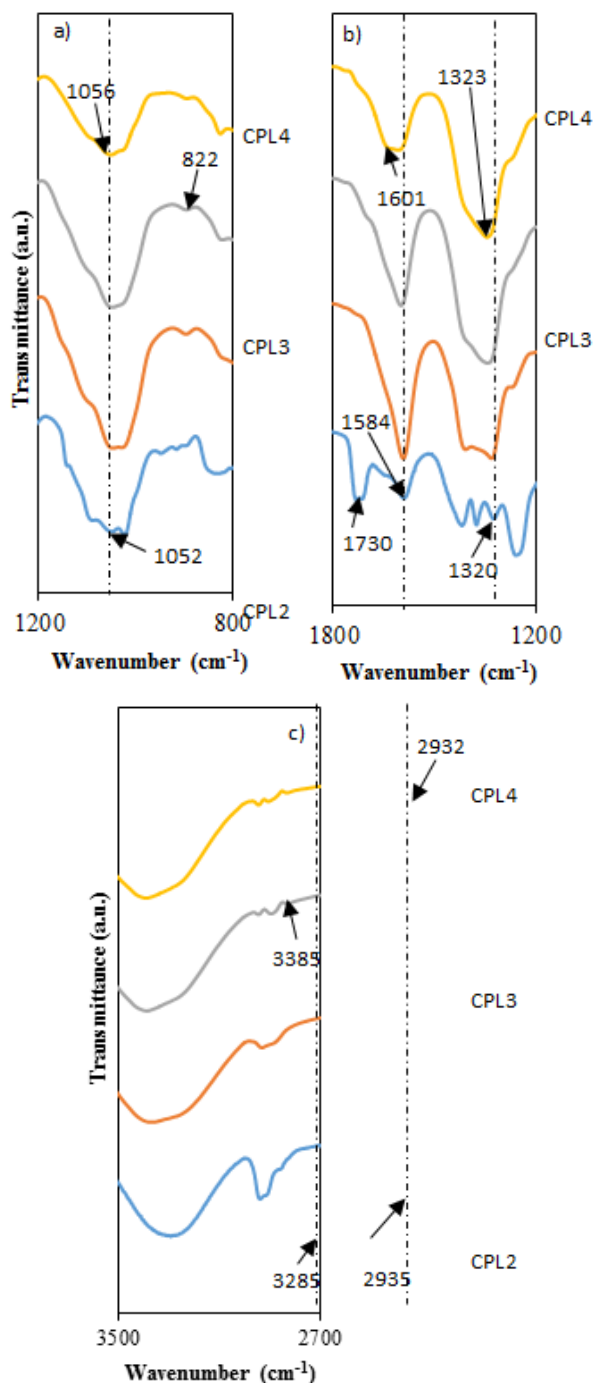


Figure 3. FTIR Spectrum of CMC/PVA-LiNO₃ with Various Contents of LiNO₃ at a) 800–1200, b) 1200–1800, and c) 2700–3500 cm⁻¹

Meanwhile, the band at a wavenumber of 2942 cm⁻¹ corresponded to -CH stretching (Figure 3(c)). During the addition of LiNO₃, the peak intensity decreased, and this result may be attributed to the incorporation of LiNO₃ into the CMC/PVA blending mixture, which caused changes in the band intensity of the system [58]. Furthermore, the peak at 3285 cm⁻¹, which belonged to -OH stretching, shifted to a higher wavenumber. This phenomenon might have been due to the decreased amount of Li⁺ that interacted with the -OH functional group. The result indicates the high formation of ion pairs and minimized coordination sites at the -OH functional group [47].

Ionic conductivity studies. Figure 4 shows the ionic conductivity of CMC/PVA-LiNO₃-based HPEs at room temperature. Figure 4 displays that the ionic conductivity increased with the increase in LiNO₃ content. This increment can be attributed to the dissociation of ions, which in turn increased the ionic mobility and charge carrier. CPL3 exhibited the highest ionic conductivity of 3.54 × 10⁻³ S cm⁻¹ compared with the other materials and showed that 20 wt% is the optimum amount of LiNO₃ that can be added to the HPEs. LiNO₃, which acted as ionic dopant, dissociated into Li⁺ and NO₃⁻ and led to a higher number of free ions (Li⁺). A similar observation have been reported by Samsudin *et al.*, that is, as the composition of LiNO₃ increased, more Li⁺ contributed to the system, which caused ion hopping in the system [59]. The increased number of free ions increased the ionic conductivity of the system [60].

However, beyond 20 wt% LiNO₃, the ionic conductivity decreased. This result was observed because excessive LiNO₃ can cause the accumulation of ions and hinder

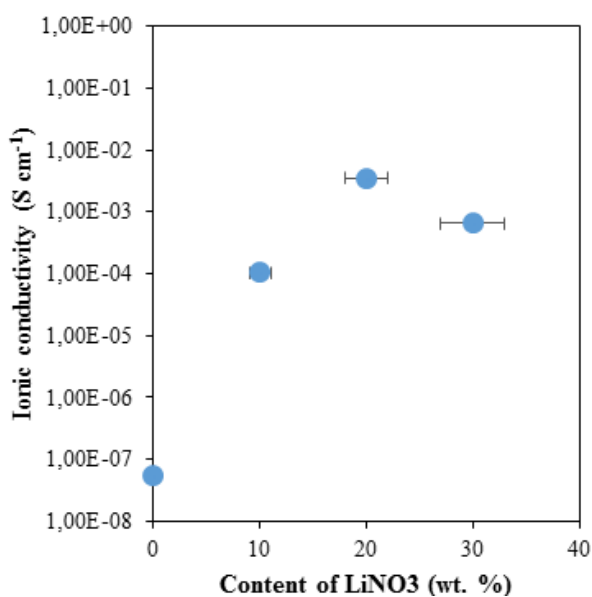


Figure 4. Ionic Conductivity of CMC/PVA-LiNO₃ at Room Temperature

their mobility, leading to a reduced ionic conductivity [39]. The high LiNO₃ content triggered ions to associate and form neutral aggregation, resulting in the decrement of ionic conductivity. In addition, the number of mobile ions in HPE was reduced [61].

In this research, the temperature dependence of ionic conductivity was studied in the temperature range of 303 K to 343 K. Based on Figure 5, when the temperature increased, the ionic conductivity increased, indicating the high mobile charge carrier produced in the system due to the sufficient energy provided for the ions to dissociate [15]. All HPE samples showed a correlation close to unity, and such result can be associated to the high number of ions and mobility of ions in the HPEs. Therefore, this situation resulted in the increased ionic conductivity due to the decrease in viscosity and increase in chain flexibility [31].

The HPE samples obeyed the Arrhenius behavior as the regression values dependence are close to unity (R²~1) according to the equation below:

$$\sigma = \sigma_o \exp\left(\frac{E_a}{kT}\right) \tag{3}$$

where σ_o is the pre-exponential factor, E_a is the activation energy, and k is the Boltzmann constant. The E_a value was calculated by using Equation (3) and plotting the values in Figure 5. The lowest activation energy was 3.29 × 10⁻² eV, which belonged to the most conducting sample in the HPE system (CPL3).

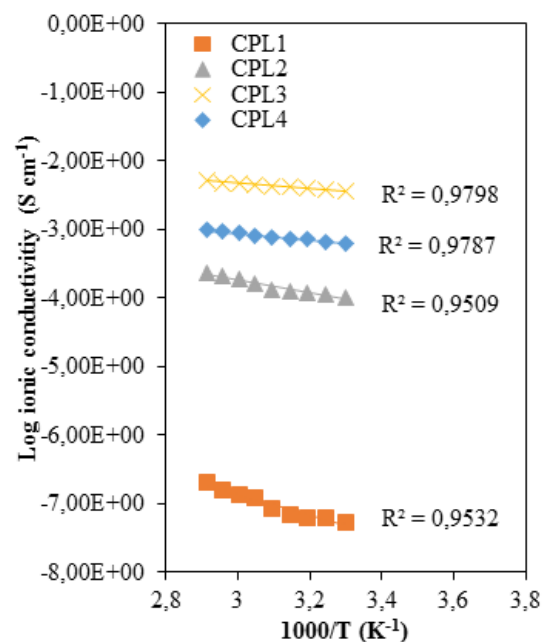


Figure 5. Temperature Dependence of CMC/PVA-LiNO₃ from 303 K to 343 K

Figure 6 shows that the activation energy decreased as the ionic dopant increased. The lowest activation energy was a low energy barrier for ion transport and enabled the easy passage of ions present in the HPEs through the barrier. Meanwhile, CPL1 achieved the highest activation energy, indicating that ions need a high energy to hop from one site to another.

Electrochemical characterizations. The highest ionic conducting sample, CPL3, was fabricated into an EDLC, and the electrochemical performance of the EDLC was tested at different scan rates. The charge storage at the electrode–electrolyte interface in the EDLC was identified by using CV analysis [26]. Figure 7 shows the CV profiles of the most conducting sample at room temperature. The CV profile showed an almost rectangular shape that became smaller with the decrease in the scan rate. This

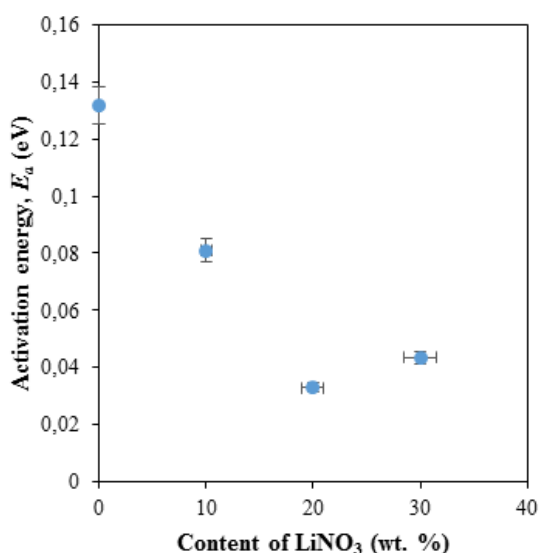


Figure 6. Activation Energy of CMC/PVA-LiNO₃ HPEs

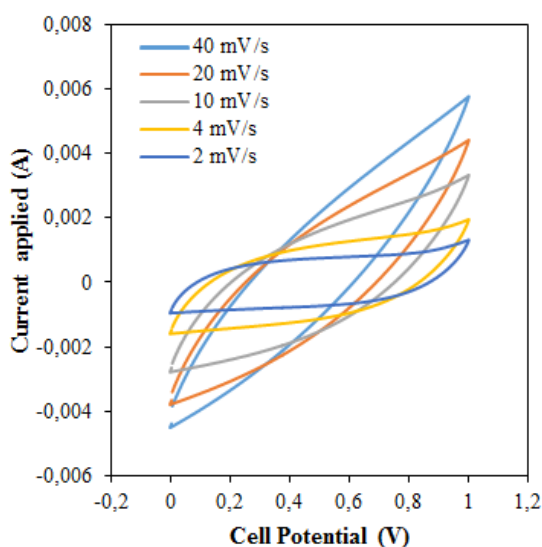


Figure 7. CV Profiles at Various Scan Rates of CPL3

Table 2. Specific Capacitance at Different Scan Rates by using CV

Scan rate (mV s ⁻¹)	Specific capacitance (F/g)
2	737.07
4	537.45
10	281.78
20	150.75
40	74.95

finding was due to the charge transfer resistance that occurred in the EDLC when different scan rates were used.

The specific capacitance, C_s was calculated by using Equation (2), and the results were tabulated in Table 2. As the scan rates increased, the C_s decreased, possibly due to the increased energy loss and low stored charge on the electrode surface of the EDLC cell. The value of C_s was high at the lowest scan rates because of the utilization of ions on the empty sites in the active electrode material. Hence, the ions had sufficient time to diffuse on particular empty sites [23].

4. Conclusions

CMC/PVA doped with various contents of LiNO₃ was prepared by using a casting method. The structural and ionic conductivity of CMC/PVA-LiNO₃ were studied by using FTIR spectroscopy and EIS. The interaction between the host polymer and ionic dopant was explained by the appearance of peaks, shift in wavenumber, and reduction of peak intensity in IR spectroscopy. Meanwhile, the highest ionic conductivity obtained belonged to the sample containing 20 wt% LiNO₃, which also showed the lowest activation energy. The temperature dependence of ionic conductivity for all the HPE samples obeyed the Arrhenius behavior. Therefore, the ionic conductivity of HPEs is dependent on the number of mobile charge carrier. The highest ionic conducting sample fabricated into EDLC exhibited a high value of C_s at 2 mVs⁻¹ due to the utilization of ions in the vacant sites of the electrode material.

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