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Impact of $LiClO_4$ Concentration on the Electrical and Dielectric Properties of Corn Starch-PVA Solid Polymer Electrolytes as Potential Application for Electric Double-Layer Supercapacitors

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Electrolytes are the critical determinant of the performance and efficiency of Electric Double-Layer Capacitors (EDLCs). The current study addresses the analysis of solid polymer electrolytes for electrical and dielectric characteristics which were prepared from corn starch–polyvinyl alcohol (CS–PVA) blends doped with varying concentrations of lithium perchlorate, $LiClO_4$. In this regard, systematically evaluating the main parameters such as ionic conductivity, dielectric behavior and electrochemical stability is of utmost importance. These results showed that the enhancement of $LiClO_4$ concentration drastically changed the electrical properties of CS–PVA blends. Samples were prepared and were tested. The mixture with 70 wt.% $LiClO_4$ had the maximum ionic conductivity with a value of 2.58×10^{-3} S/cm at room temperature. This improvement has been attributed to the higher mobility of mobile carrier ions as well as to better segmental motion inside the polymer matrix. Dielectric analysis revealed that the sample containing a higher $LiClO_4$ addition displayed a lower relaxation time, as well as an increased dielectric constant, indicating improved ion mobility inside the polymer matrix. The loss tangent values suggested an increased segmental motion of polymer chains with increasing content of $LiClO_4$ in turn affecting the charge transport. Linear sweep voltammetry (LSV) was carried out to verify the electrochemical stability of this optimized electrolyte, which can sustain a potential window of 2.93 V and the measured ion transference number (TNM) achieved 0.87 showing that ionic conduction is still the main mechanism in this case. The results demonstrate the feasibility of using $LiClO_4$ doped CS–PVA SPE as a promising biodegradable material for EDLCs applications with extensive highlights on its great electrochemical properties needed for ion transportability, high conductivity and good electrochemical stability.

Keywords: Conductivity; dielectric permittivity; corn starch; polyvinyl alcohol

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One of the promising technologies being investigated for an efficient energy storing device over the last few years is electric double-layer capacitors (EDLCs). The electrolyte plays an essential role in EDLCs, as it enables the electric double layer to be formed on the interface between two electrodes and allows the movement of ions back-and-forth between the two. EDLCs operate through a non-Faradaic mechanism by storing charge as ions dwelling on the electrode-electrolyte interface, and not by electron transfer [1, 2].

Recently, many researchers have investigated solid polymer electrolytes (SPEs) because they contribute to the excellent mechanical stability and safety requirements, as well as the excellent contact at each interface between SPEs and electrodes in EDLCs. The ideal EDLCs electrolyte is required to possess a high ionic conductivity, wide electrochemical stability window, rapid ion diffusion mobility dynamics and simultaneously low cost-effective large-scale production with environmental sustainability. Polyvinyl alcohol (PVA) is one of the most investigated polymers for SPEs since it has a semicrystalline structure, nontoxicity and mechanical robustness that also favors charge storage [3]. Corn starch, a naturally abundant and biodegradable polymer, is composed of two glucose-based components—amylose and amylopectin—linked by α-glycosidic bonds [4]. Corn starch when blended with PVA, interacts mainly in terms of hydrogen bonding, where the OH groups are responsible for it. In addition to the functional groups that introduce hydroxyl groups and thus also polarity through lone pairs on the oxygen atoms, these functional groups are likely to improve their suitability for use in electrochemical systems primarily by forming solid-solid contacts between the components.

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The selected conductive salt is another factor that can affect SPE performance. Typically, lithium perchlorate ($LiClO_4$) is employed due to its low molar mass of the lithium cation, bulky perchlorate anion, allowing for high ion mobility and better conductivity [5]. Furthermore, its high energy of dissociation and solubility make it an attractive candidate in polymer-based systems. It was an energetic study [6], experiments regarding this matter proved that $LiClO_4$ provides perfect conductivity and a wide electrochemical stability window, making it a good candidate for EDLCs.

On the other hand, for its biodegradability and renewable character, corn starch was chosen as the main polymer matrix in this work because of its ability to serve as an eco-friendly substitute for synthetic polymers. Although biodegradable matters, for example starch, often have the regretfully low ionic conductance, and conventional SPEs are potentially sustainable problems concerning long-term surroundings repercussions in prolonged use [7]. Overcoming the trade-off can be key to developing green energy materials. To tackle these problems, it focuses on the effect of $LiClO_4$ concentration in CS-PVA blend over its conductivity and stability. Although some studies have been done regarding these electrolytes, to the best of knowledge, there have not been any studies concerning the electrical and dielectric characteristics of $LiClO_4$-doped CS–PVA solid polymer electrolytes done before. The present study fills this gap by providing a comprehensive assessment of the basic properties of cell behavior in terms of impedance, ionic conductivity, dielectric response, modulus behavior, and linear sweep voltammetry from both cathode and anode sides, and also with transference number. These findings could lead to more efficient, biodegradable polymer electrolytes that are inherently earth-abundant, and point towards strategies for future energy storage device design.

METHODOLOGY

Materials

The key materials used in this experiment were corn starch, PVA, glycerol (Aldrich, 99.0% purity), 0.1 M sodium hydroxide (NaOH, Aldrich, 97.0% purity), 0.1 M hydrochloric acid (HCl, Aldrich, 37.0%), and lithium perchlorate ($LiClO_4$, Across, 99.0% purity).

Preparation of the Blend CS- PVA BPEs

The blend films were prepared using the solution casting method. Various weight percentages of $LiClO_4$ (10, 30, 50, 70 wt%) were added to the blend solution to create different formulations. First, $LiClO_4$ was dissolved in 20 mL of methanol. Meanwhile, the corn starch and PVA blend was prepared by adding 10 wt% of each to a solution containing 0.1 M HCl, 0.1 M NaOH, and glycerol. Each $LiClO_4$ solution was then mixed with the blend solution. The resulting mixture was stirred with a magnetic agitator at 80 °C for 24 hours to ensure uniformity. Afterward, the homogeneous blend was poured into a petri dish and allowed to dry through slow evaporation at room temperature.

Characterization of the Blend CS- PVA BPEs

The impedance, dielectric permittivity, modulus, and conductivity of all films were measured at room temperature (27°C) using an Agilent 4284A Precision LCR meter, interfaced with a computer and controlled via software. The measurements were taken over a frequency range of 0.1 Hz to 100 kHz. The samples, cut into dimensions of 2 cm x 1.5 cm, were placed between stainless-steel electrodes to ensure optimal contact. The Nyquist plot for complex impedance (Z*) was analyzed, with both the real part (Z') and imaginary part (Z'') assessed. Bulk resistance (R_b) was extracted from the Nyquist plot along the real axis.

To evaluate the ionic and electronic transport properties of the $LiClO_4$-CS-PVA blend, which exhibited the highest conductivity at room temperature, Transference Number Measurements (TNM) were performed using Wagner's DC Polarization Method. In this method, a fixed DC voltage of 1.5 V was applied across the electrolyte, and the DC current was monitored over time. Stainless-steel electrodes were used as blocking electrodes to prevent ion flow, allowing only electrons to pass through the external circuit. The transference numbers of ions ($t_{1_{on}}$) and electrons (t_e) were subsequently calculated.

The electrochemical stability window of all samples was assessed at room temperature (27°C) using linear sweep voltammetry (LSV). The breakdown voltage was determined at a sweep rate of 50 mV/s, with data recorded over a potential range of 0 to 5 V.

RESULTS AND DISCUSSION

Impedance and Conductivity

The electrochemical impedance spectroscopy (EIS) technique was utilized to determine the direct current conductivity σ_{dc} and ion transport behavior of the prepared solid polymer electrolyte (SPE) samples. One critical aspect of the traditional Nyquist plot ($-Z_i$ versus Z_r) is to obtain the bulk resistance (R_b) for calculating σ_{dc}, however, it additionally contributes to understanding the mechanism of ion transport and behavior of charge carries i.e., ionic or electronic nature [8]. Figure 1 shows Nyquist plots ($-Z_i$ versus Z_r) of CS-PVA BPEs with the various concentrations (wt.%) of $LiClO_4$ at room temperature. A sample with a concentration of 10 wt.% $LiClO_4$ shows an incomplete semicircle at high frequency which

illustrates the rollover phenomenon of the lack of ion transporting within the bulk electrolyte which strongly contributes by resistive and capacitive elements in the polymer network, respectively. The appearance of a spike at low frequencies indicates surface charge accumulation at electrode interfaces, which describes the processes of double layer formation and electrode polarization for systems with blocking edges [9], [10]. When the $LiClO_4$ concentration increases above 10 wt.%, the Nyquist plot changes from having a semicircle to just forming a linear spike. This transition indicates the resistance of charge transfer at the interface of electrode–electrolyte is very low or even neglected. In lower concentrations, the semicircle is due to the resistance present during the charge transfer and in forming the electrical double layer. Above a certain salt concentration, the system becomes fully galvanostatic and the electrochemical reaction is easily reached, that point at which charge transfer no longer limits the process. As a result, the semicircle shrinks or disappears completely. The resulting spike, frequently oblique at 45° or perhaps also vertical, reflects the fact that diffusion-related phenomena / capacitive behavior has now assumed an overwhelming role in the impedance response (i.e. ion transport or interfacial charging process semi-determines the magnitude of the impedance response), rather than the kinetics of electron transfer. The bulk resistance (R_b) steadily declined as the $LiClO_4$ concentration rose up to 70 wt.%. At this point, the samples became quite brittle and fragile structurally, often failing and fracturing during routine tests and measurements due to their delicate nature. These values of R_b for the samples lead us to compute by means of Eq. 1 σ_{dc}, i.e., direct current (DC) conductivity:

$$\sigma_{dc} = \frac{t}{R_b A} \quad (1)$$

where, t is the thickness and A is the contact area of the sample [11], [12]. A key aspect in spontaneously evaluating electrolyte materials is the ionic conductivity. The dependency of conductivity on concentration in terms (wt.%) of $LiClO_4$ for various compositions added into the CS-PVA matrix is shown in Figure 2. The figure indicates that the conductivity increases gradually with the increase in concentration of $LiClO_4$ and significantly achieves a maximum value of 2.70×10^{-3} S cm^{-1} at 70 wt.% $LiClO_4$ concentration. The increase in ionic conductivity may be due to the complex interaction of Li^+ ions with hydroxyl groups having oxygen atoms inside both PVA and corn starch. Such interactions result in the creation of charge transfer complexes that serve as hopping sites for Li+ ions to be easily transported within the CS–PVA matrix. As for corn starch and PVA, they have hydroxyl groups (–OH) along their polymer chains, which might take up lithium ions. These hydroxyl groups act as Lewis bases, which bond strongly to Li^+ ions and hold them in place within the polymer network. This interaction serves to increase the thermodynamic stability of the lithium ions in the matrix and simultaneously to suppress ion-trapping sites, which facilitates their eventual agglomeration. One representation of the improvements is the formation of more effective ion-conducting pathways, specifically among the amorphous areas in the polymer blend leading to better ionic conductivity.

Such behavior is consistent with the prevailing understanding that electrical conductivity in ionically conducting materials is controlled by two principle factors, charge carrier number and mobility, respectively [13]. This relationship is given by Eq.2:

$$\sigma = \sum_i n_i q_i \mu_i \quad (2)$$

where n_i, q_i,and μ_i are the charge carrier's concentration (i.e., number of charge), electron charge, and the ion mobility (where i refers to the identity of the ion), respectively. From Eq. 2, it is evident that increasing the concentration of charge carriers (n_i) and/or enhancing the mobility of ionic species in the system will result in a higher ionic conductivity (σ). It is well-established that conductivity is directly proportional to the number of charge carriers, the charge associated with the ions, and the mobility of the ions. Previous studies have shown that salt dissociation can be facilitated either by using volatile solvents or by selecting suitable polymers that are compatible with cations, allowing them to dissociate from their corresponding anions. According to research conducted by Aziz et al. in 2019, a polymer blend of corn starch and polyethylene oxide achieved an impressive DC conductivity of 7.34×10^{-4} S cm^{-1} when incorporating 40 wt.% by of $LiClO_4$ salt and this polymer blend electrolyte system demonstrated good performance in an EDLCs [14]. The present study recorded an even higher conductivity value of 2.58×10^{-3} S cm^{-1} for a CS-PVA blend polymer electrolyte doped with the concentration of 70 wt.% $LiClO_4$. This excellent outcome further suggests that the CS-PVA BPEs system exhibits enormous potential for development in applications utilizing EDLCs, where a high degree of ionic conductivity is crucial for optimized charge accumulation and swift ion transport within the device.

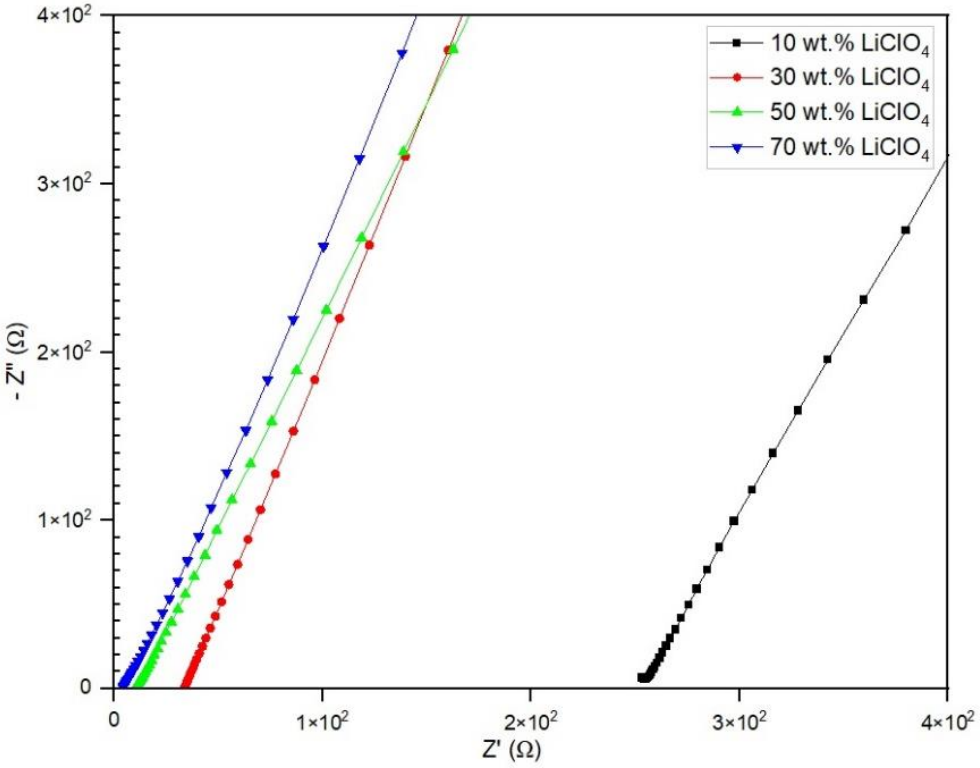

Figure 1. Electrochemical impedance spectroscopy (EIS) curve of different concentrations (wt. %) of $LiClO_4$ in CS- PVA blend-based polymer electrolyte at room temperature.

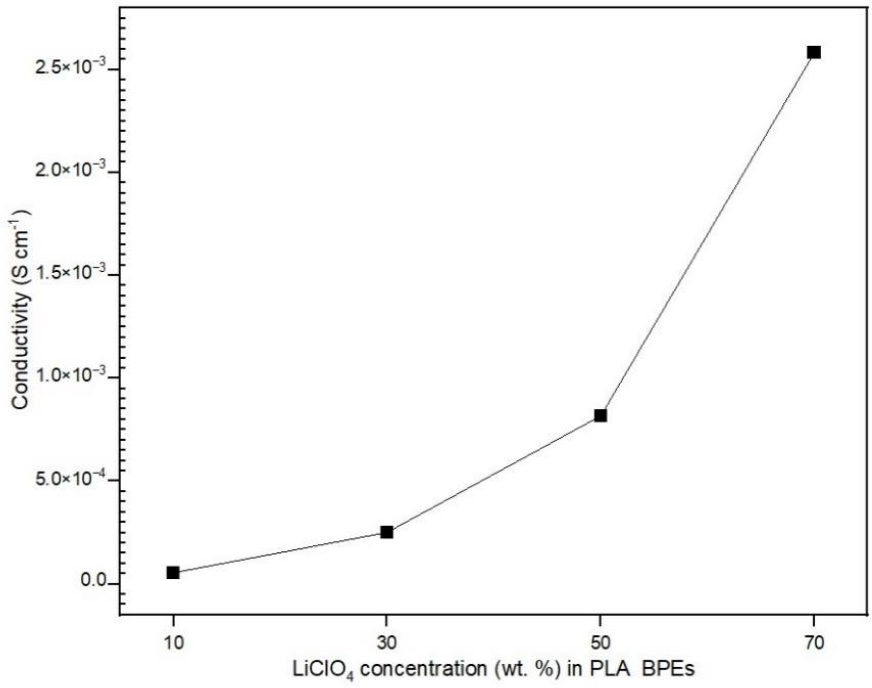

Figure 2. Conductivity at different concentration (wt. %) of $LiClO_4$ in CS- PVA blend-based polymer electrolyte at room temperature.

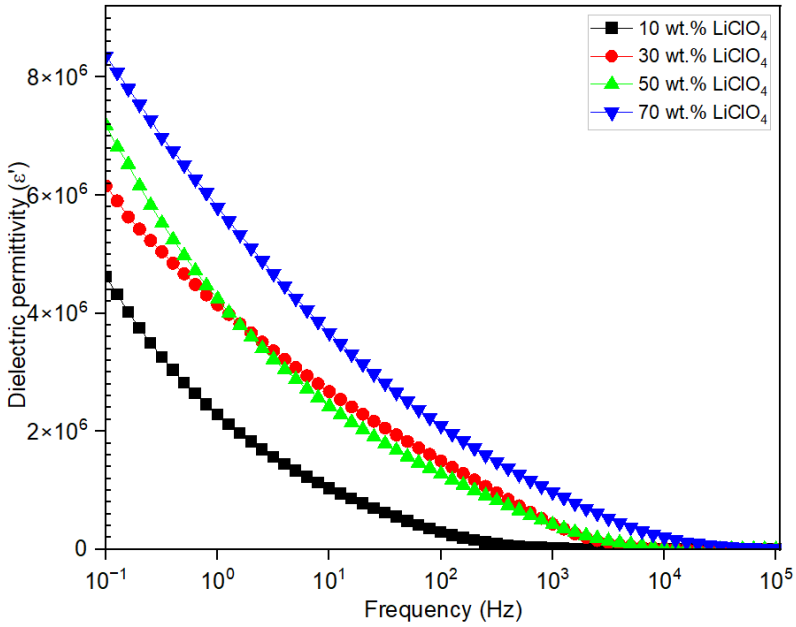

Figure 3. Dielectric permittivity (ε') at different concentrations (wt. %) of $LiClO_4$ in CS- PVA blend-based polymer electrolyte at room temperature.

Dielectric Study Analysis

Dielectric measurements are essential for understanding the ion-polymer interactions and conduction mechanisms in polymer electrolyte systems. The dielectric parameters are linked to the relaxation processes, which include structural relaxation and conductivity relaxation. Figure 3 shows a higher value of ε′ in the low-frequency region, which is associated with interfacial polarization. This is likely due to the increased charge carrier concentration at the electrode-electrolyte interface, rather than reflecting the bulk properties of the dielectric, as typically observed in solid polymer electrolytes (SPEs). At very low frequencies, ions have enough time to accumulate at the interface, leading to a high dielectric constant. As the frequency increases, the dielectric constant decreases non-linearly and approaches a steady state, known as the limiting permittivity (ε∞), around 10 kHz, due to the system's inability to keep up with the rapid reversal of the electric field.

The high dielectric constant observed at low frequencies is primarily attributed to the Maxwell-Wagner effect, which involves ionic conduction and electrode polarization due to ion accumulation at the electrode-electrolyte interface [15]. As the frequency increases, the dielectric constant decreases and reaches a constant value at higher frequencies. This is because the periodic reversal of the electric field occurs too quickly for ions to diffuse efficiently in the direction of the field, and most ions remain in the bulk of the sample, leading to a reduction in the dielectric constant [16].

These results suggest that the CS-PVA-$LiClO_4$ polymer blend electrolyte system exhibits a non-Debye type of behavior, where the space charge regions in relation to frequency are explained by ion diffusion. Additionally, the dielectric constant increases with the $LiClO_4$ content. The higher dielectric constant observed for the CS-PVA-$LiClO_4$ blend electrolyte with 70 wt.% $LiClO_4$ salt can be attributed to the enhanced charge carrier density in the space charge accumulation region, which increases the equivalent capacitance value [16].

Tangen Loss Analysis

The loss tangent (tan δ) parameter is commonly used to describe the dielectric relaxation process in solid polymer electrolytes (SPEs). The loss tangent versus frequency plot, shown in Figure 4, illustrates this behavior. Tan δ is defined as the ratio of the imaginary part of the permittivity to the real part, or alternatively, the ratio of energy loss to energy stored [17]. This figure shows a single relaxation peak for the sample with 10 wt% $LiClO_4$, indicating ionic conduction within the system. The graph also reveals that as the salt concentration increases, the relaxation peak shifts towards higher frequencies. This shift occurs because the peak moves out of the measured frequency range.

In the low-frequency region, the increase in loss is primarily attributed to the dominance of the Ohmic component over the capacitive element [17], [18]. The presence of a maxima at a single frequency corresponds to the perfect matching between the frequency of the electric field and the molecular rotation frequency. This resonance condition leads to maximum power transfer to the dipoles in the system, resulting in the maximum heat generation.

In the high-frequency range, the capacitive component becomes more prominent, while the Ohmic part becomes frequency-independent. As the frequency increases, the capacitive component grows, and the shift in the relaxation peak to higher frequencies with increasing $LiClO_4$ concentration in the CS-PVA blend polymer electrolyte suggests faster ion dynamics. This is due to a decrease in relaxation time, indicating more efficient ion movement from one coordinating site to another [18].

Linear Sweep Voltammetry (LSV) Analysis

A critical characteristic to assess before device production is the Linear Sweep Voltammetry (LSV), which helps determine the operational voltage range of energy storage devices. Figure 5 presents the LSV trends, showing that as the concentration of $LiClO_4$ in the CS-PVA BPEs increases, the breakdown voltage decreases. However, all samples exhibited a minimal anodic current peak at voltages above 3 V. This indicates that the electrolyte films have a breakdown voltage greater than 3 V, making them suitable for use in supercapacitors. Given that the breakdown voltage for energy devices should be at least 1.0 V [19], these CS-PVA BPEs are appropriate for such applications. The optimized concentrations of $LiClO_4$ enhance ion transport by creating pathways for ion migration, ensuring a more uniform ion distribution and minimizing the formation of localized high electric fields. This result is significant for electrochemical applications. The observed increase in current at higher voltages is attributed to decomposition occurring at the inert electrode interface. The anodic decomposition voltage of the polymer electrolyte is defined as the potential at which a rapid rise in current is observed, continuing to increase as the voltage is swept. This initial current surge is associated with the decomposition of the electrolyte [20]. Overall, the electrochemical stability of all CS-PVA BPEs is confirmed by these findings.

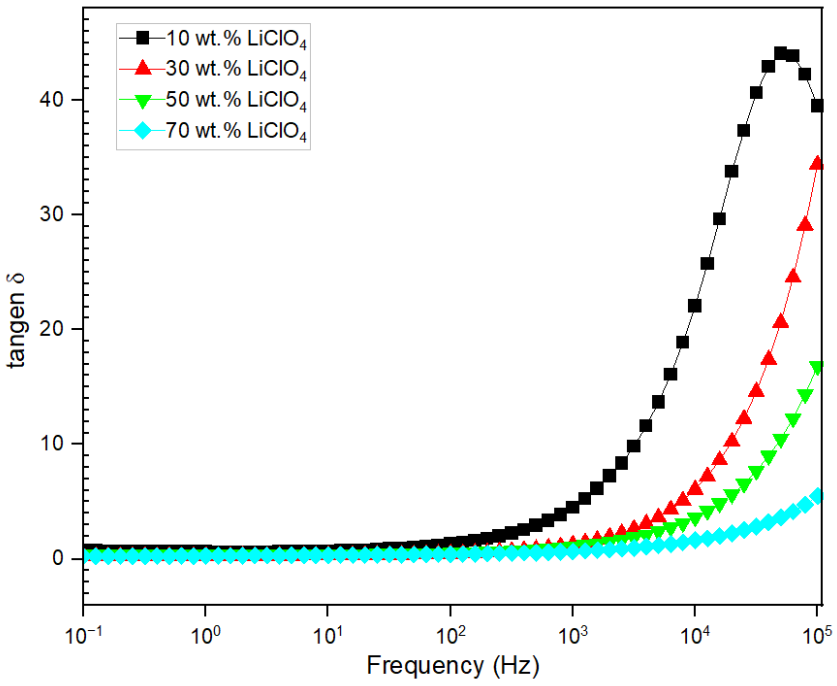

Figure 4. Tangent δ at different concentrations (wt. %) of $LiClO_4$ in CS- PVA blend-based polymer electrolyte at room temperature.

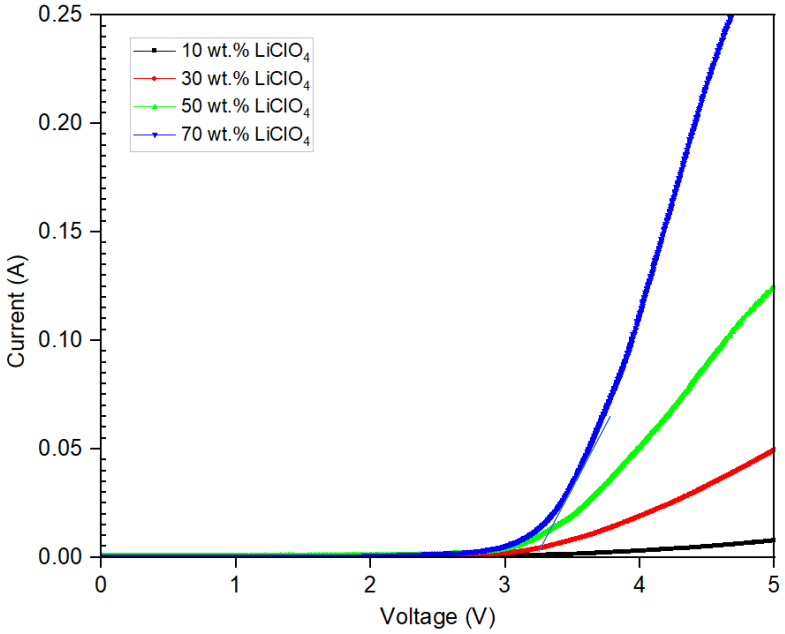

Figure 5. Potential stability of the system with different concentrations (wt.%) of $LiClO_4$ in PLA.

TNM Measurement

The contribution of charged species, whether ions or electrons, involved in the conduction process within electrolytes can be identified through transference number measurements. Wagner's DC polarization technique is used to determine the total ionic transference number for polymer electrolytes with stainless-steel electrodes. This method helps categorize the primary charge carrier species present in the electrolytes. When the transference number value approaches unity, it indicates that ions are the dominant charge carriers in the system. Figure 6 shows the current versus time curve for the optimal sample, which contains 70 wt% $LiClO_4$ in CS-PVA BPEs, during the polarization process. Since most charge carriers are ions, the initial current gradually decreases, as seen in the figure. This is due to the balancing of mobile ion diffusion, which eventually reaches a steady state. At the plateau region, very few ions contribute to current flow, as the stainless-steel electrodes act as barriers, preventing further ion movement. The total ionic transference number (t_{ion}) of the polymer electrolytes is calculated by

$$t_{ion} = \frac{I_i - I_O}{I_i} \quad (3)$$

where I_i and I_o represent the initial and steady currents, respectively. The value of t_{ion}, which is 0.875, confirms the dominant role of ions in the total conductivity of the polymer electrolyte. The transference numbers for both electrons and ions were determined using the following relationships:

$$t_{ion} = 1 - t_e \quad (4)$$

The ionic transference number is denoted as t_{ion}, while the electron transference number is denoted as t_e. The initial current is represented by I_i, and the steady-state current is denoted as I_{ss}. Using the equations outlined previously, the calculated values for t_e and t_{ion} are 0.125 and 0.875, respectively.

In theory, once the polarization is complete in the prepared electrolyte film, it should become highly resistive, and ideally (for a pure ionic conductor electrolyte), no current should be recorded. However, the small amount of current observed in this study may be attributed to the release of a minimal number of electrons during the applied potential, or due to complexation between the various constituents of the polymer electrolyte (PE), or even the presence of impurities within the PE. These factors can contribute to the electronic current observed after full polarization [19].

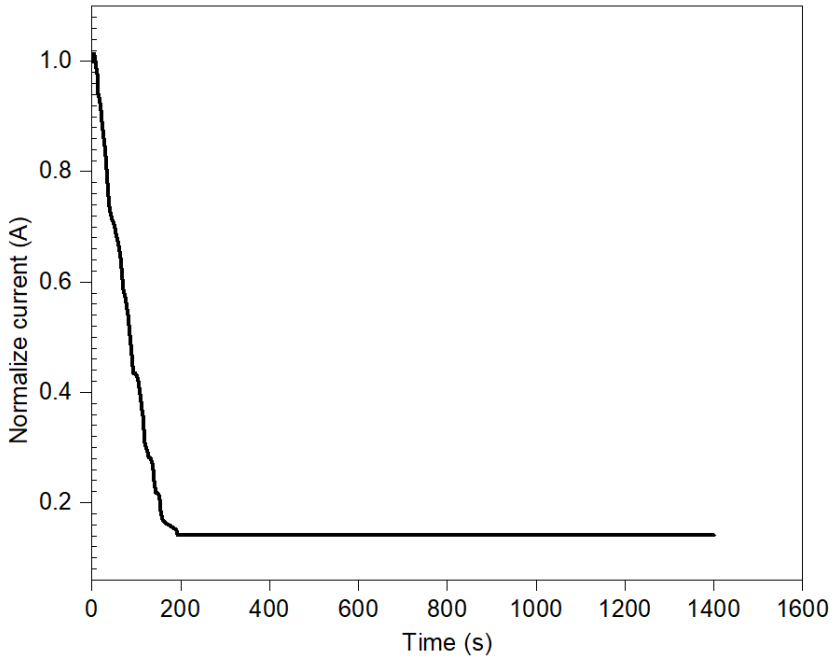

Figure 6. Transfer number for the optimum $LiClO_4$ in CS-PVA BPEs.

CONCLUSION

CS-PVA blend-based polymer electrolytes (BPEs) with varying concentrations of $LiClO_4$ have been successfully synthesized using the solution casting technique. The results demonstrate that CS-PVA blend-based solid polymer electrolytes (SPEs) incorporating $LiClO_4$ show significant potential for use in electrical double-layer capacitors (EDLCs). Optimization of the $LiClO_4$ concentration improves both ionic conductivity and ion mobility, with the optimal concentration found to be 70 wt.% in the samples. By utilizing renewable materials, this research offers an eco-friendly alternative to petroleum-based plastics, contributing to waste reduction and promoting sustainable energy solutions.

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