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Synthesis and Ion Transport Studies of a New Potassium Ion-Conducting Nanocomposite Polymer Electrolyte

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Abstract

The pursuit of safe, efficient, and sustainable energy storage solutions has intensified research on nanocomposite polymer electrolytes, especially for next-generation battery technologies. This work reports the synthesis and ion transport characteristics of potassium ion-conducting nanocomposite polymer electrolytes (NCPEs), emphasizing their structural, electrical, and electrochemical performance. To enhance ionic conductivity and mechanical strength, polymer matrices were reinforced with nanoscale ceramic fillers. A new potassium ion-conducting NCPEs: (1-x)[70PEO:30KCl] + x SiO_2 (where 0<x<20 wt.%) were synthesized via a hot-pressing method. The polymer electrolyte films combined polymer hosts with potassium salts and incorporated nano-sized ceramic filler SiO_2 to disrupt polymer crystallinity and facilitate improved ion transport pathways. The optimal composition, consisting of 95% [70PEO:30KCl] and 5% SiO_2, demonstrated the highest room-temperature ionic conductivity of approximately 6.3×10^{-5} $S\cdot cm^{-1}$. Key electrical parameters including ionic conductivity (σ), ionic mobility (μ), mobile ion concentration (n), and ionic transference number (t_{ion}□) were thoroughly evaluated. Structural and thermal analyses of the NCPEs were conducted using scanning electron microscopy (SEM), differential scanning calorimetry (DSC), and thermogravimetric analysis (TGA). The optimized nanocomposite displayed enhanced ionic conduction, favorable ion transport characteristics, and improved thermal stability. These findings underscore the promise of potassium-based NCPEs as safer and more abundant alternatives to lithium-based electrolytes for solid-state energy storage applications.

Keywords: DSC, hot-press method, ionic conductivity, nanocomposite polymer electrolytes, Sem, TGA

INTRODUCTION

In recent years, there has been an increasing demand for Advanced solid polymer electrolytes (SPEs) are increasingly demanded for next-generation energy storage like all-solid-state batteries. These systems promise enhanced safety, higher energy density, and longer lifecycles compared to liquid electrolyte-based systems. Among ion-conducting systems, potassium-ion based polymer electrolytes draw interest due to potassium's abundance, low cost, high redox potential (−2.93 V vs SHE), and large ionic radius offering favorable diffusion dynamics in polymer matrices [1,2]. Over three decades, significant attention focused on developing ion-conducting nanocomposite polymer electrolytes (NCPEs). These materials offer advantages over traditional polymer electrolytes, including lightweight construction, ease of handling, high energy density, volumetric stability, lack of solvents, leak-proof behavior and broad electrochemical stability [3–9]. NCPEs

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facilitate fabricating flexible, compact, thin-film batteries in various shapes, enabling intimate electrode-electrolyte interfaces. Beyond batteries, they find applications in displays, capacitors, portable power, sensors, and fuel cells. Polyethylene oxide (PEO)-based electrolytes are widely used in electrochemical devices. However, pure PEO has low room-temperature ionic conductivity (~10^{-9} $S \cdot cm^{-1}$). Conductivity is enhanced by incorporating salts, resulting in solid polymer electrolytes (SPEs) [10–14]. Further improvements in SPE properties are achievable by dispersing nanoscale fillers like SiO_2, TiO_2, or ZnS [15,16]. Potassium salts offer advantages over lithium counterparts, including better stability under ambient conditions, greater abundance, and lower cost.

In the context of Potassium-ion conduction research is in its infancy compared to lithium and sodium systems. Developing potassium-conductive nanocomposite polymer electrolytes (K-NCPEs) holds promise due to compatibility with aluminum current collectors and inexpensive materials [17]. Potassium salts like $KClO_4$, KPF_6, or KCF_3SO_3 offer dissociation characteristics when paired with polymer hosts and nanofillers [18].

Typically, SPE, and NCPE films are fabricated using casting or sol-gel methods [5]. Recently, solvent-free hot-pressing techniques emerged, offering benefits over traditional fabrication approaches [19–22].

In this context We report the synthesis and characterization of potassium-ion conducting nanocomposite polymer electrolytes with composition (1–x) [70PEO:30KCl] + x SiO_2, where 0<x<20 wt.%. Materials were prepared using a hot-pressing technique, ensuring uniform filler dispersion and mechanical integrity. Investigations assessed the effect of SiO_2 content on ion transport properties.

MATERIALS AND METHODS

The Starting materials for synthesizing hot-pressed nanocomposite polymer electrolytes (NCPEs) with composition (1-x)[70PEO:30KCl] + x SiO_2 (0<x<20 wt.%) included poly(ethylene oxide) (PEO) with molecular weight 10^5 (Aldrich, USA), potassium chloride (KCl) purity above 98% (Merck, India), and silica nanoparticles (SiO_2) of 8 nm size and 99.8% purity (Sigma, USA). NCPE films were prepared using a hot-press technique. Detailed procedures for this method appear in our previous publications [21,22]. Materials were characterized and interactions analyzed through differential scanning calorimetry (DSC; Perkin Elmer), scanning electron microscopy (SEM; JEOL JXA-8100, Japan), and thermogravimetric analysis (TGA; SDT Universal). Ionic conductivity (σ) measurements used the formula:

$$\sigma = \frac{l}{R_b.A}[S.cm^{-1}] \quad (1)$$

where R_b represents bulk resistance, l is thickness, and A denotes cross-sectional area. R_b was measured at 5 kHz using an LCR instrument (HIOKI 3520–01, Japan). Ionic mobility was calculated using the relation:

$$\mu = \frac{d^2}{V.\tau}[cm^2V^{-1}s^{-1}] \quad (2)$$

where d is thickness, V is DC voltage, and τ is time of flight. Time of flight τ was measured using the transient ionic current (TIC) method with a DC polarization setup and an x-y-t recorder (Graphtec WX 2300–1L, Japan) [23]. Mobile ion concentration (n) used conductivity (σ) and mobility (μ) values per the formula:

$$\sigma = n.\,q.\,\mu\,[cm^{-3}] \quad (3)$$

where q is charge. Ionic transference number (t_{io}□) was evaluated using dc polarization with the following equation:

$$t_{ion} = 1 - \frac{I_e}{I_T} \quad (4)$$

where I_e represents electronic current, and I_T is total current in the cell [SS / NCPE OCC / SS]. To evaluate activation energy (E_a) for the SPE host and NCPEs, conductivity measurements were performed across various temperatures.

RESULTS AND DISCUSSION

Figure 1 shows how the ionic conductivity of nanocomposite polymer electrolytes (NCPEs) with composition (1-x)[70PEO:30KCl] + x SiO_2 (0<x<20 wt.%) changes as nano-sized SiO_2 varies. Ionic conductivity increases with SiO_2 content, showing two peaks at 5 wt.% and 13 wt.% SiO_2, with values of 6.3×10^{-5} S·cm^{-1} and 3.1×10^{-5} S·cm^{-1}, respectively. Highest conductivity occurs at 5 wt.% SiO_2. Similar dual maxima were reported by other researchers [5,24]. These peaks correspond to different percolation thresholds associated with K^+ and Cl^- species. Improvement in ionic conductivity is linked to Lewis acid-base interactions between components [5]. Enhancement is also attributed to transient inter- and intra-molecular cross-links among PEO chains [25,26]. Two types of transient cross-linking occur: (a) polymer segment cross-linking mediated by cation interactions, and (b) cross-linking involving both cations and anions, as illustrated in Figure 2.

Figure 3 illustrates plots of log(μ) and log(n) as functions of SiO_2 content (x) for NCPE films with composition (1–x)[70PEO:30KCl] + xSiO_2. Trends in mobility (μ) and mobile ion concentration (n) follow the log(σ) behavior. Highest mobility of 2.08×10^{-2} cm^2·V^{-1}·s^{-1} and mobile ion concentration of 4.52×10^{15} cm^{-3} were recorded at x = 5 wt% at room temperature. Findings suggest room temperature conductivity improvement is due to enhancements in both μ and n. Table 1 presents conductivity (σ), mobility (μ), concentration (n) and transference number (t_{io}□) for the SPE host and optimized NCPE composition, confirming that μ and n contribute to increased conductivity in the NCPE.

Figure 4 displays SEM images showing surface morphology of pure PEO, SPE host (70PEO:30KCl) and NCPE with 5 wt% SiO_2. The NCPE sample exhibits a smooth and homogeneous surface, reflecting higher amorphous content due to interaction between polymer, salt, and SiO_2 filler. In contrast, pure PEO reveals a rougher surface texture, consistent with higher crystalline content [27–30].

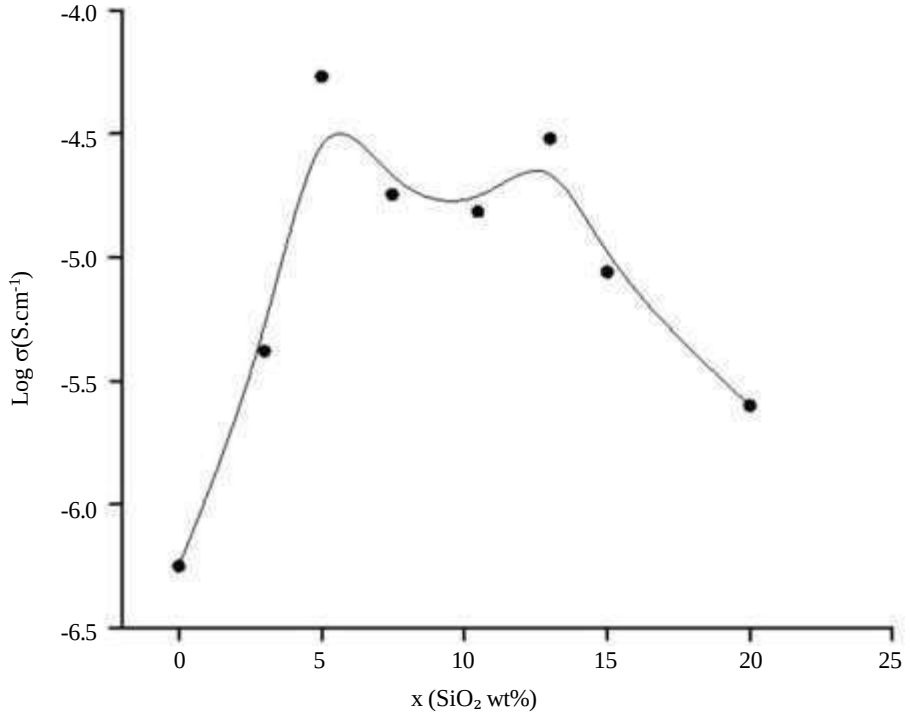

Figure 1. Compositional dependent conductivity plot of NCPE films: (1-x) [70PEO:30KCl] + x SiO_2.

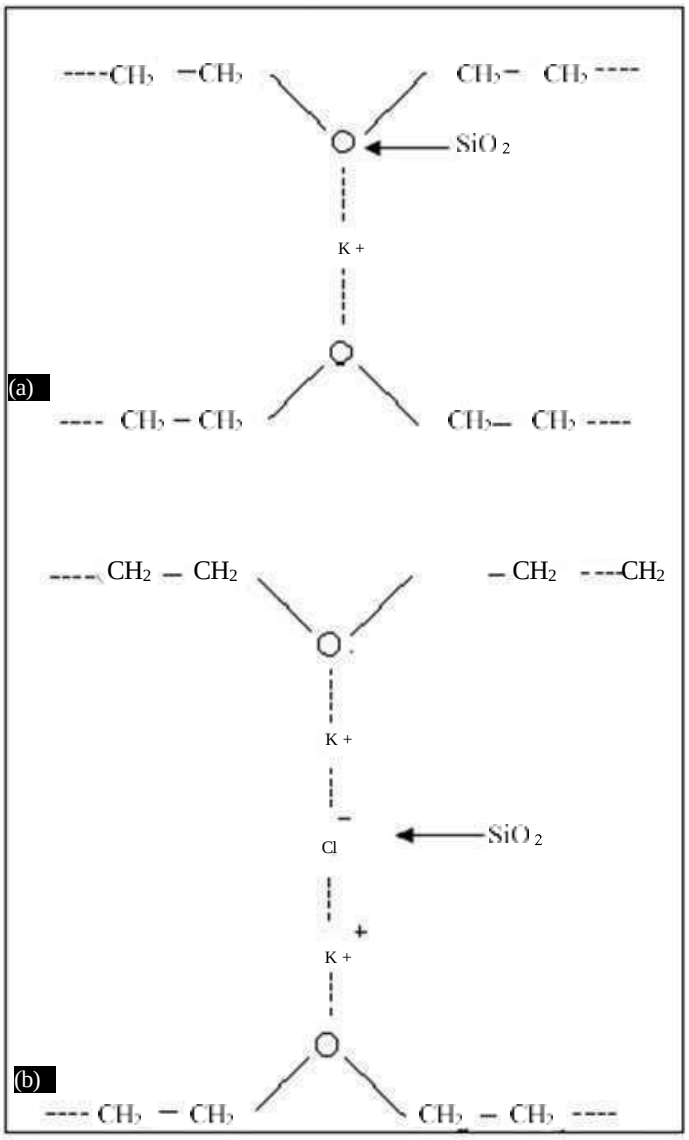

Figure 2. Schematic illustration of two types of cross-linking mechanisms in NCPEs: (a) cross-linking between polymer chains through cation coordination, and (b) cross-linking involving both cation and anion interactions with polymer segments.

Figure 5 presents differential scanning calorimetry (DSC) curves for pure PEO, SPE host (70PEO:30KCl) and NCPE composition: 95[70PEO:30KCl] + 5 wt% SiO_2. All samples exhibit broad endothermic peaks corresponding to melting temperatures (Tm), indicating thermal transitions associated with crystalline regions of the polymer matrix.

The slight decrease in melting point observed in both the SPE host and the NCPE system indicates an increase in amorphous content, suggesting successful formation of the nanocomposite polymer electrolyte (NCPE).

Table 1. Some important ionic parameter values of SPE host and NCPE OCC.

Systems	**σ (Scm^{-1})**	**μ (cm^{2}V^{-1}s^{-1})**	**n (cm^{-3})**	**t_{ion}**	**E_a (eV)**
SPE Host: [70PEO:30KCl]	5.6×10^{-7}	3.16×10^{-3}	1.09×10^{15}	0.95	0.39
NCPE OCC: 95[70PEO:30KCl] + 5 SiO_2	6.3×10^{-5}	2.08×10^{-2}	4.52×10^{15}	0.95	0.24

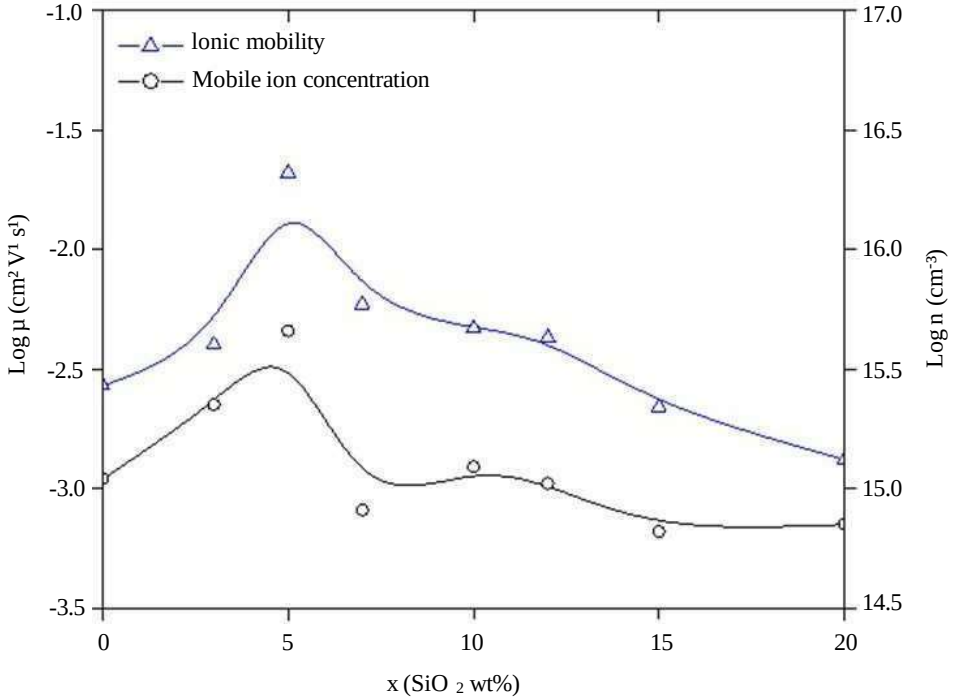

Figure 3. Compositional mobility and mobile ion concentration plots of NCPEs: (1-x) [70PEO:30KCl] + x SiO_2.

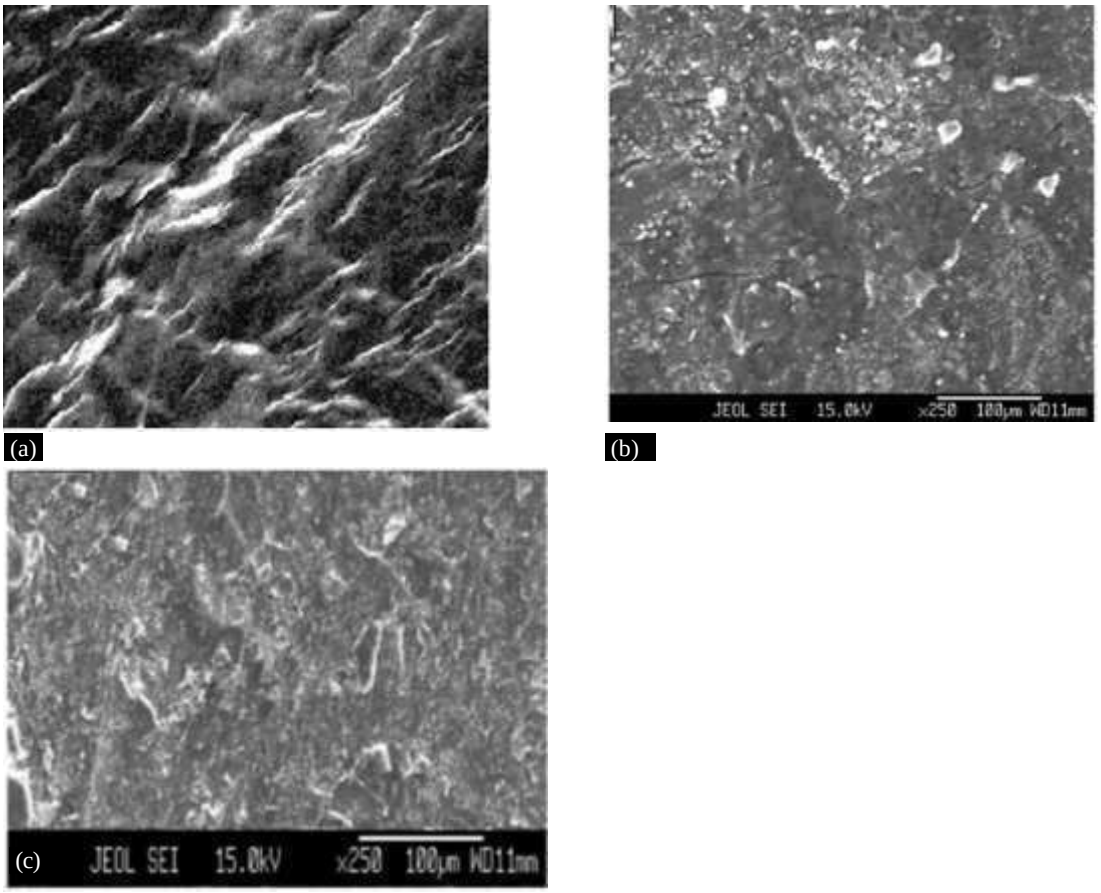

Figure 4. SEM Images: (a) Pure PEO [18], (b) SPE Host: 70PEO:30KCl and (c) NCPE OCC: 95 [70PEO:30KCl] + 5 SiO_2.

Figure 6 displays thermogravimetric analysis (TGA) curves for pure PEO, SPE host (70PEO:30KCl), and NCPE OCC: 95[70PEO:30KCl] + 5 wt% SiO_2. Weight loss recorded for pure PEO is approximately 95%, significantly higher than the SPE host (~73%) and the NCPE (~65%). This reduction reflects improved thermal stability. Enhancement in resistance with SiO_2 addition is consistent with increased amorphicity and confirms successful NCPE formation.

To assess the ionic nature, the ionic transference number (t_{io}□) was determined using DC polarization. Figure 7 presents current versus time for the cell [SS / NCPE / SS]. A transference number of approximately 0.95 was obtained, indicating dominant charge carriers are K^+ ions (~95%), with minimal Cl^- contribution (~5%). The value close to unity confirms predominantly ionic conduction behavior, making this NCPE system suitable for application in solid-state electrochemical devices.

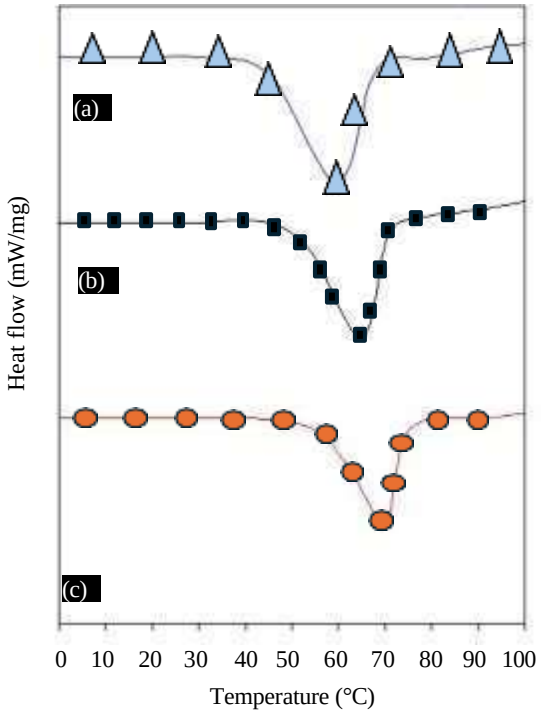

Figure 5. DSC Thermograms: (a) Pure PEO, (b) SPE Host: (70PEO:30KCl) and (c) NCPE OCC: 95 [70PEO:30KCl] + 5 SiO_2.

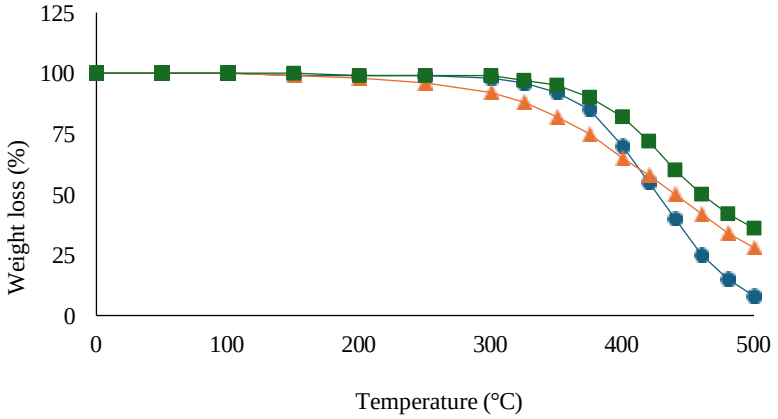

Figure 6. TGA curves: (a) Pure PEO(●), (b) SPE host: (70PEO:30KCl) (▲) and (c) NCPE OCC: 95 [70PEO:30KCl] + 5 SiO_2(■).

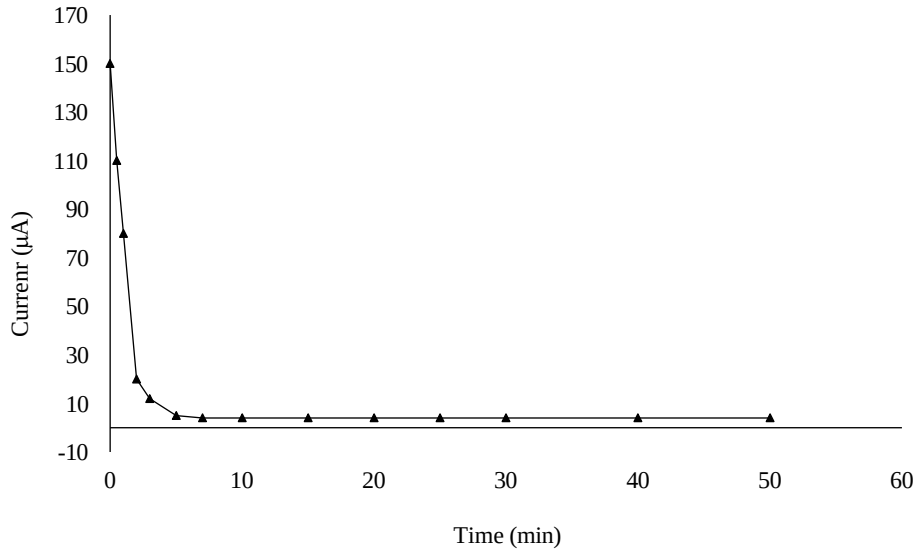

Figure 7. Current vs time' plot for the cell: SS / NCPE OCC/ SS.

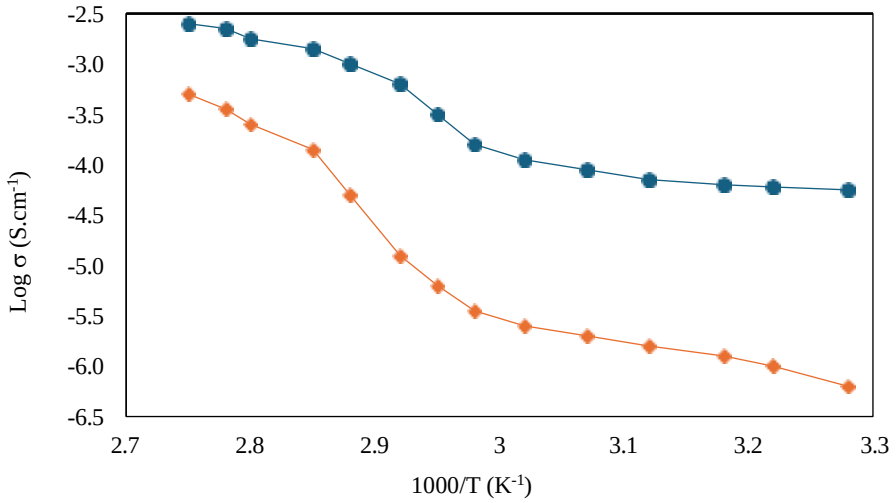

Figure 8. Log σ-1/T' Plots for SPE Host: (70PEO:30KCl) (◆) and NCPE OCC: 95 [70PEO:30KCl] + 5 SiO_2 (●).

Figure 8 presents temperature-dependent conductivity for the SPE system (70PEO:30KCl) and NCPE OCC: 95 [70PEO:30KCl] + 5 SiO_2. Data reveal conductivity decline with increasing inverse temperature (1/T), suggesting thermally assisted conduction. Rise in conductivity is attributed to a hopping mechanism, where ions move between coordination sites aided by structural flexibility. As temperature increases, polymer chains experience motions facilitating segmental movement. This promotes transport, leading to improved conductivity. The linear trend reflects Arrhenius behavior, expressed using these equations:

SPE host: $\sigma (T) = 4.5 \times 10^{-3} \exp (-0.39/ kT)$ [S·cm^{-1}] (5)

NCPE OCC: $\sigma (T) = 2.3 \times 10^{-2} \exp (-0.24/ kT)$ [S·cm^{-1}] (6)

Activation energies, 0.39 eV and 0.24 eV, correspond to the systems. Lower activation energy for NCPE suggests higher amorphous character enhancing flexibility and faster ionic transport. This reduced energy barrier implies efficient mobility within the material, making the system a promising candidate for electrochemical devices.

CONCLUSIONS

A novel potassium ion-conducting nanocomposite polymer electrolyte (NCPE): 95 [70PEO:30KCl] + 5 SiO_2 exhibiting ionic conductivity of 6.3×10^{-5} $S\cdot cm^{-1}$ has been developed. Incorporation of SiO_2 nanoparticles despite being insulating resulted in a two-order-of-magnitude improvement in conductivity. This enhancement is attributed to both increased ionic mobility (μ) and higher concentration of mobile ions (n). Structural and thermal analyses confirm effective polymer-salt interaction. Characterization suggests the system predominantly conducts via ionic transport, with 95% of K^+ ions contributing. The material exhibited excellent performance, including a high ionic transference number of 0.95, indicating ion conduction is the dominant mechanism. This t_{io}□ value, along with conductivity, thermal stability and integrity, highlights the suitability of the electrolyte for use in advanced solid-state batteries.

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