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Short Report

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Effect of Nano sized Alumina on Poly (ethyl methacrylate) (PEMA) based Electrolyte Nanocomposite and its application in dye sensitized solar cell (DSSC)

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Abstract: Poly (ethyl methacrylate) (PEMA) based solid polymer composite electrolyte structural and ionic conductivity characteristics were modified by the incorporation of Alumina (Al₂O₃) nanoparticles. PEMA in addition with 40% wt. potassium iodide (KI) and ethylene carbonate (EC) having 60% wt., has the highest ionic conductivity of 4.65×10^{-5} S/cm when employed the solution casting technique. Nanosized Alumina (Al₂O₃) was incorporated with PEMA + KI 40% wt. + EC 60% wt. sample to get maximum conductivity sample. The highest ionic conductivity 2.65×10^{-3} S/cm, was attained at 7% weight percentage of Alumina Nanoparticles (Al₂O₃). The highest conductive sample's surface morphology was examined using SEM, its amorphicity and crystalline structure was investigated using XRD, and confirmation of composite formation was done through Fourier transform infrared (FTIR) technique. Differential scanning calorimetry (DSC) analysis was used to find out the glass transition (T_g) temperature of maximum conducting sample of polymer composite. The doctor blade method was employed to develop the dye sensitized solar cell (DSSC), and it had been observed that, under 1 sun condition, the energy conversion efficiency was 2.09%, having parameters fill factor was 79.77%, the short circuit current density was 3.82 A/cm² and the open circuit voltage was 0.686V.

Keywords: PEMA, EC, Polymer Electrolyte, ionic conductivity, Alumina, DSSC, DSC.

1. Introduction

The rapid growth of portable electronics, renewable energy technologies, and advanced electrochemical devices has intensified the demand for safe, efficient, and environmentally sustainable energy-storage materials. Conventional liquid electrolytes, although characterized by high ionic conductivity, often suffer from several limitations such as leakage, volatility, flammability, and poor mechanical stability. These shortcomings have encouraged researchers to explore alternative electrolyte systems capable of delivering improved safety and long-term operational stability. Among the available alternatives, solid polymer electrolytes (SPEs) have emerged as attractive candidates because of their flexibility, lightweight nature, ease of processing, excellent film-forming ability, and compatibility with a wide range of electrochemical devices including lithium-ion batteries, fuel cells, supercapacitors, and dye-sensitized solar cells (DSSCs) (Manuel Stephan and Nahm 2006).

Polymer electrolytes generally consist of a polymer host matrix and an ionic salt dissolved within the polymer framework. The polymer provides mechanical integrity, whereas the dissolved salt supplies mobile ions responsible for ionic conduction. The effectiveness of ion transport in polymer electrolytes is strongly influenced by the structural characteristics of the host polymer. In particular, the degree of crystallinity plays a crucial role in determining ionic conductivity. Highly crystalline polymers restrict chain mobility and impede ion migration, whereas amorphous regions facilitate segmental motion of polymer chains, thereby promoting ion transport. Consequently, enhancing the amorphous phase of polymer electrolytes has become one of the most widely adopted strategies for improving their electrochemical performance.

Many polymers, including PMMA, PEO, PVA, PVC, PEMA, and PVdF, among others, were examined by researchers around the Globe (Zhang et al. 2008). Degree of Crystallinity in polymers can be modified by several approaches, like by adding plasticizers, fillers, salt, and nanofillers..(Gray 1997; Bower 2003). Higher ionic conductivities are found in polymer electrolytes with increased amorphousness, as reported by Berthier et al. (Berthier et al. 1983). Salt/ion transfer happens readily if the polymer composite material has an amorphous structure. Researchers have reported the effectiveness of EC, DMC, PC, and other plasticizers in promoting amorphousness (Chen et al. 2002; Sachdeva et al. 2017). As, by the addition of these plasticizers, the polymer electrolyte tends to become more amorphous,(Kalaighan and Kang 2006; Reddy et al. 2007) which further raises the glass transition temperature, enhances ion pair movements, and increase the ionic conductivity of the polymer composite electrolyte. Addition of any type of plasticizer in polymer composites decreases the mechanical strength of the polymer composite material. To increase the mechanical strength, thermal conductivity, ionic conductivity, and stability of the amorphous nature of the polymer electrolyte various different types of nanoparticles like titanium dioxide (TiO₂), carbon nanotubes (CNT), tellurium dioxide (TeO₂), aluminium oxide (Al₂O₃), Zinc oxide (ZnO), aluminium oxide (Al₂O₃) and silicon dioxide (SiO₂) (Kalaighan and Kang 2006; Ibrahim and Johan 2011; Klongkan and Pumchusak 2015) have been incorporated in the plasticised Polymer Electrolyte.

The polymer used in the present research work was PEMA. It was first time studied and discovered by researchers Han et al. and Fahmy et al. PEMA polymer contains carboxylic groups (-COOH), which facilitate ion movement in polymer composites by forming channels for interconnection. Compared to other polymers, PEMA is more semicrystalline, has a higher ionic conductivity and used in various applications(Rawal et al. 2024; Elsharkawy et al. 2024). Polymer electrolyte was investigated using various characterization techniques such as complex impedance spectroscopy (CIS), X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, Scanning electron microscopy (SEM), Differential scanning calorimetry (DSC) etc.(Pradhan et al. 2005; Amaral et al. 2015; Saxena et al. 2021.; Agrawal et al. 2004) To increase the conductivity of polymer PEMA, potassium iodide (KI) was added as salt; similarly, ethylene carbonate (EC) was added as a plasticizer to increase the amorphousness of polymer composite. Solvent Tetrahydrofuran (THF) was used as a common solvent in which these materials (PEMA, KI, EC and Al₂O₃) easily dissolved. The mechanical strength property and ionic conductivity characteristics of the polymer composite matrix improved by incorporation of Alumina nanocomposite material. Many methods including tape-casting, screen printing, solution casting, spin coating, hot pressing, and others, have been reported in the literature for producing solid polymer electrolyte films (Ngai et al. 2016).

2. Materials and Methods

2.1. Materials and Approaches

Polymer Poly ethyl methacrylate (Santa Cruz, USA) (PEMA) as polymer, potassium iodide (KI) (Loba Chemie Pvt. Ltd, India) as salt and ethylene carbonate (EC) (Sigma Aldrich, USA); as plasticizer, Al₂O₃ Nanoparticles (Sigma Aldrich, USA); were used. Solution Casting Method was used for synthesis of Composite in the present investigation. 0.5 gm of PEMA was dissolved in THF through stirring for four hours of stirring with a magnetic stirrer, a viscous solution obtained. Then, weight percentage variations of salt (KI) was added into solution, such as 10%, 20%, 30%, 40%, 50%, 60%, 70%, 80%, and 90% weight of PEMA polymer. After that solution was stirring with a magnetic stirrer for 4 hours to obtain uniformly viscous solution. To create thin films, each sample was now placed within Petri dishes and allowed to dry at room temperature. Complex impedance spectroscopy characterization technique used to measure the ionic conductivity of each sample. Subsequently, EC weight percentages variation was added to the maximum conducting sample obtained previously, including 10%, 20%, 30%, 40%, 50%, 60%, 70%, 80%, and 90% weight. Every variation's solution was stirred for up to 4 hours, resulting in a uniformly viscous solution. Subsequently, several weight percentages of Al₂O₃ nanoparticles were added to the maximum

conducting solution, ranging from 3% to 17% of the total weight. Every variation's solution was stirred for up to 12 hours, resulting in a uniformly viscous solution. To create thin films, each sample was now placed within Petri dishes and allowed to dry at room temperature. The complete process shown in fig. 1.

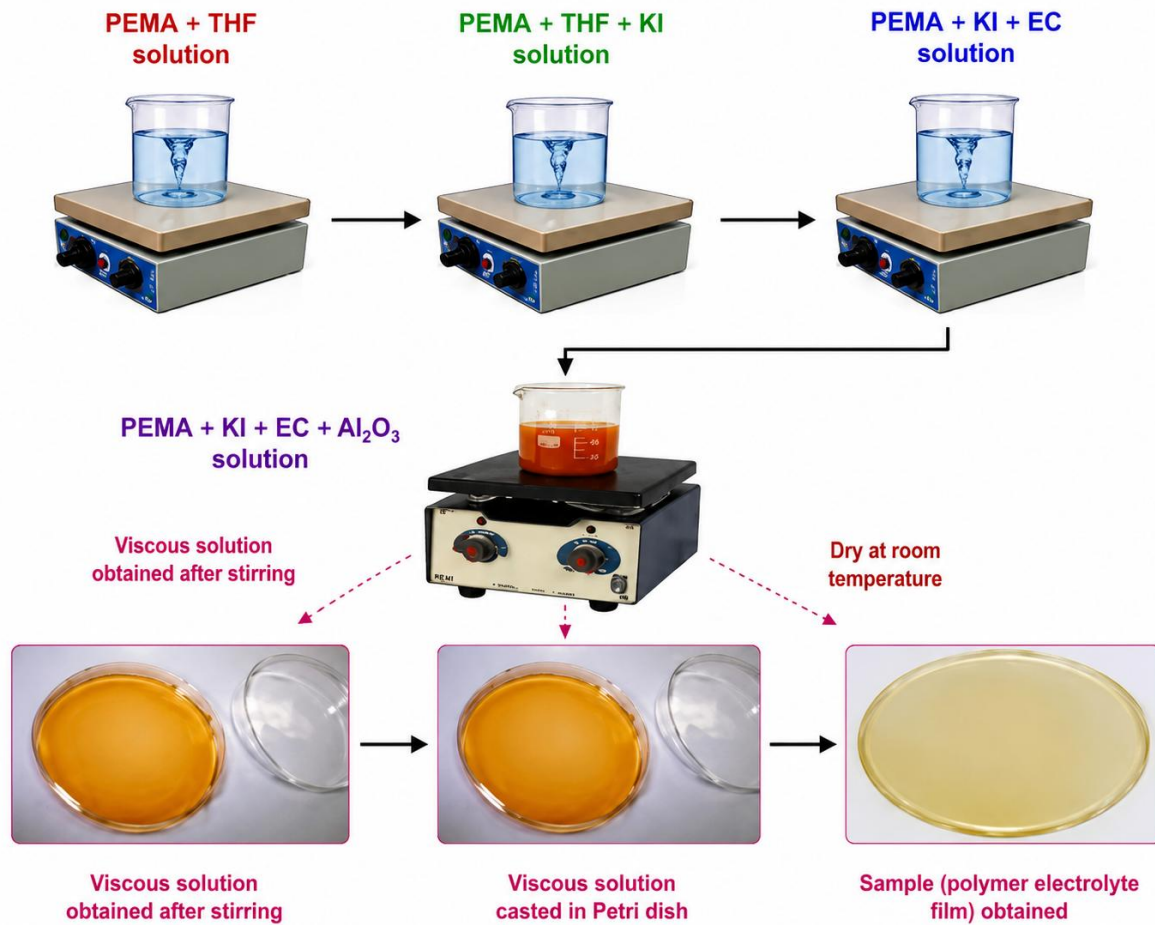


Fig. 1 Synthesis of Polymer nanocomposite material.

3. Results and discussion

3.1. Sample preparation and visual observations

During the laboratory work various samples were prepared using solution casting techniques. Some sample images are shown in fig. 2.

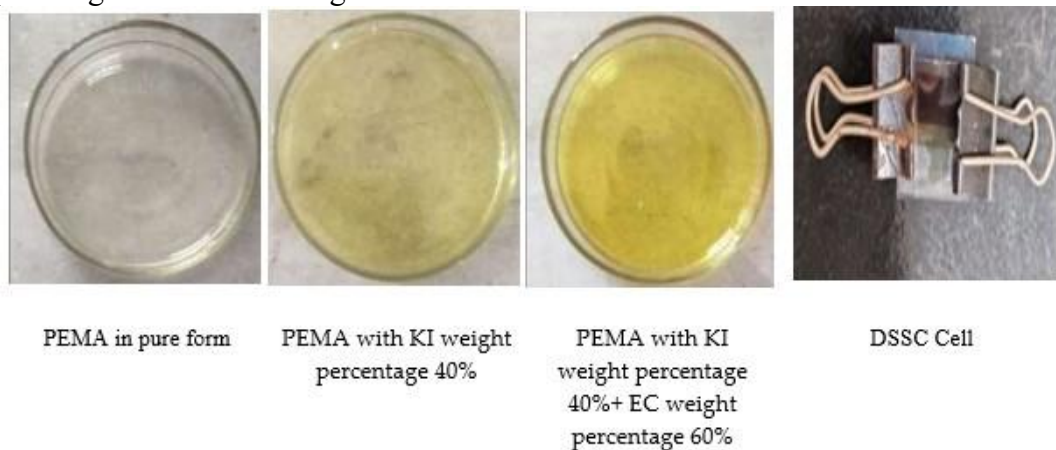


Fig. 2 Sample images.

3.2. Complex Impedance Spectroscopy

Ionic conductivity of maximum conducting samples were evaluated using following formula

$$\sigma = \frac{GI}{A}$$

Where ionic conductivity represent by σ , area by A, width by I, and G ($G=1/R_b$, R_b is bulk resistance) is conductance of sample. Nyquist graph or cole-cole was used to calculate the value of bulk resistance (R_b) that was relationship between real and imaginary axis. Using an HIKOKI 3522-50 LCR Hi Tester, USA, the produced polymer composite film was evaluated by putting a sample between stainless steel electrodes at frequencies ranging from 50 Hz to 100 KHz. Using graph between real and imaginary axis i.e. Nyquist plot, bulk resistance value was calculated. When different amounts of KI were added to PEMA polymer, 3.80×10^{-6} S/cm was the greatest conductivity that was achieved at 40% weight percentage of KI. EC was dissolved to the polymer composite in several amounts to increase its mechanical strength, and at 60% weight percentage, the maximum conductivity of 4.65×10^{-5} S/cm was attained. After some variation the conductivity did not increase because there were no more ions available for conduction. Adding Alumina nanoparticles to a plasticized PEMA-based sample further enhanced the ionic conductivity; at 7% weight percentage, the maximum conductivity was 2.65×10^{-3} S/cm as shown in fig. 3. Comparison of ionic conductivity of various polymer electrolyte composite shown in table 1.

Table 1 Comparison of Maximum Conductivity of various obtained polymer composites

Polymer Electrolyte composite	Ionic Conductivity (S/cm)
PEMA+ KI weight percentage 40%	3.80×10^{-6}
PEMA+ KI weight percentage 40% + EC weight percentage 60%	4.65×10^{-5}
PEMA+ KI weight percentage 40% + EC weight percentage 60%. + Al ₂ O ₃ weight percentage 7%	2.65×10^{-3}

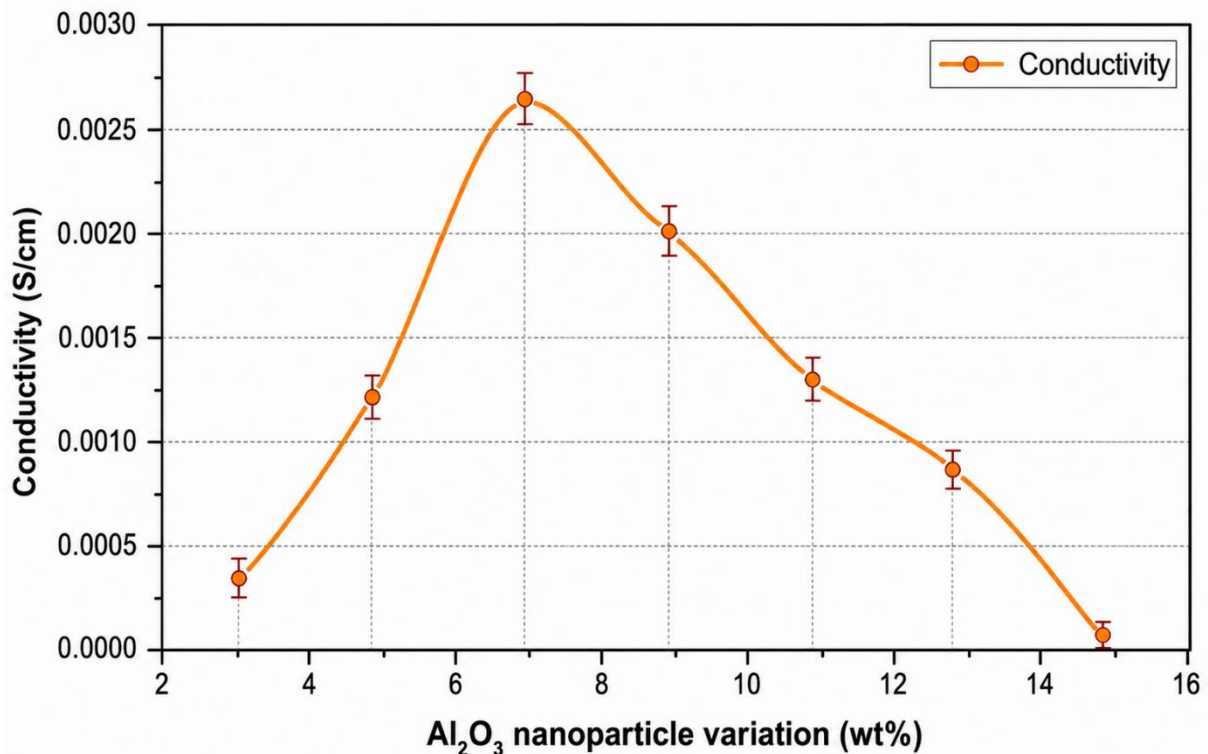


Fig. 3 Conductivity of polymer nanocomposite at different variations of Al₂O₃.

3.3. SEM

Scanning electron microscopy (Hitachi S-570, USA) was used to determine the morphology of solid polymer electrolyte. Fig. 4 displays the surface morphology of various obtained samples having maximum conductivity. PEMA was crystalline, indicating comparatively low ionic conductivity.

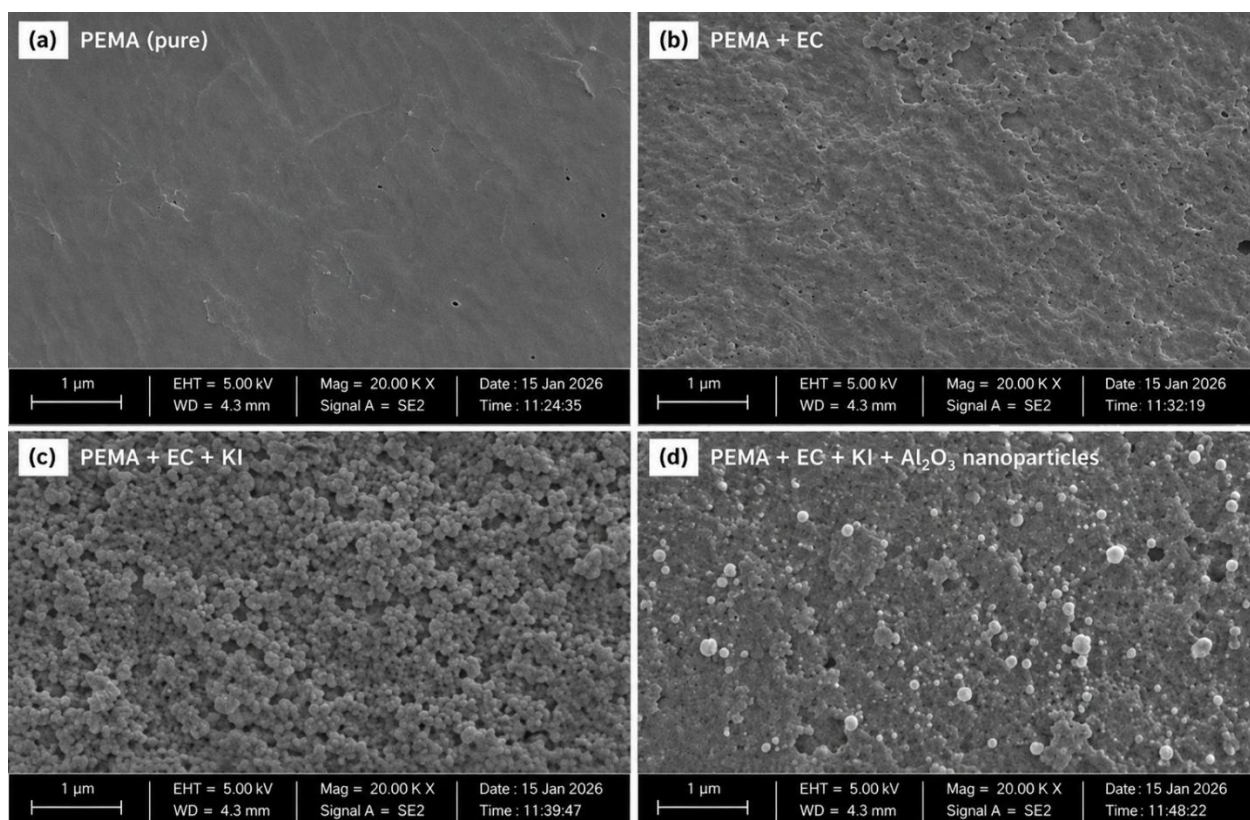


Fig. 4 Morphology comparison of different polymer composite.

The SEM micrograph of pure PEMA exhibits a relatively smooth, dense, and homogeneous surface with very few surface defects or pores. The compact morphology indicates the semi-amorphous nature of the polymer matrix and the absence of any filler or salt particles. Such a compact structure offers limited pathways for ion transport, resulting in comparatively low ionic conductivity.

After the incorporation of ethylene carbonate (EC), the surface becomes slightly rougher and more flexible with the appearance of shallow depressions and irregular features. EC acts as a plasticizer, reducing the crystallinity of PEMA and increasing the amorphous phase. The increased free volume enhances polymer chain mobility, thereby facilitating ion migration.

The addition of potassium iodide (KI) results in a more granular and uniformly distributed morphology. The salt is well dispersed throughout the polymer-plasticizer matrix without significant phase separation, indicating good compatibility among the constituents. The increased amorphous nature and higher concentration of mobile K^+ and I^- ions provide continuous conduction pathways, leading to a significant improvement in ionic conductivity.

The SEM image of the Al_2O_3 nanocomposite electrolyte reveals uniformly dispersed bright nanoparticles embedded within the polymer matrix. The nanoparticles are distributed homogeneously with only slight agglomeration, indicating effective interaction between Al_2O_3 and the polymer-salt system. The incorporation of Al_2O_3 disrupts the residual crystalline regions of PEMA, enhances the amorphous content, and promotes salt dissociation through Lewis acid–base interactions. Consequently, additional ion transport pathways are created, resulting in enhanced ionic conductivity. The homogeneous dispersion of nanoparticles also contributes to improved mechanical stability of the electrolyte film..

3.4. FTIR Analysis

The FTIR characterization approach was used to study information about the function group and composite polymer structure. The FTIR spectra of the several materials was investigated using Perkin-Elmer spectrophotometer (model 833) shown in Fig. 5. The vibrational band found at wavenumber 2995 cm^{-1} and 2935 cm^{-1} was attributed to the symmetric and asymmetric C-H stretching of the methylene group contained in PEMA. FTIR spectra were displayed between 500-

4000 cm^{-1} wavenumber. Because THF, the solvent utilized, was hygroscopic, a single bond group of -OH and -OOH was produced at wave numbers of 3000 cm^{-1} to 3500 cm^{-1} . Various vibrational bands observed during FTIR analysis shown in table 2.

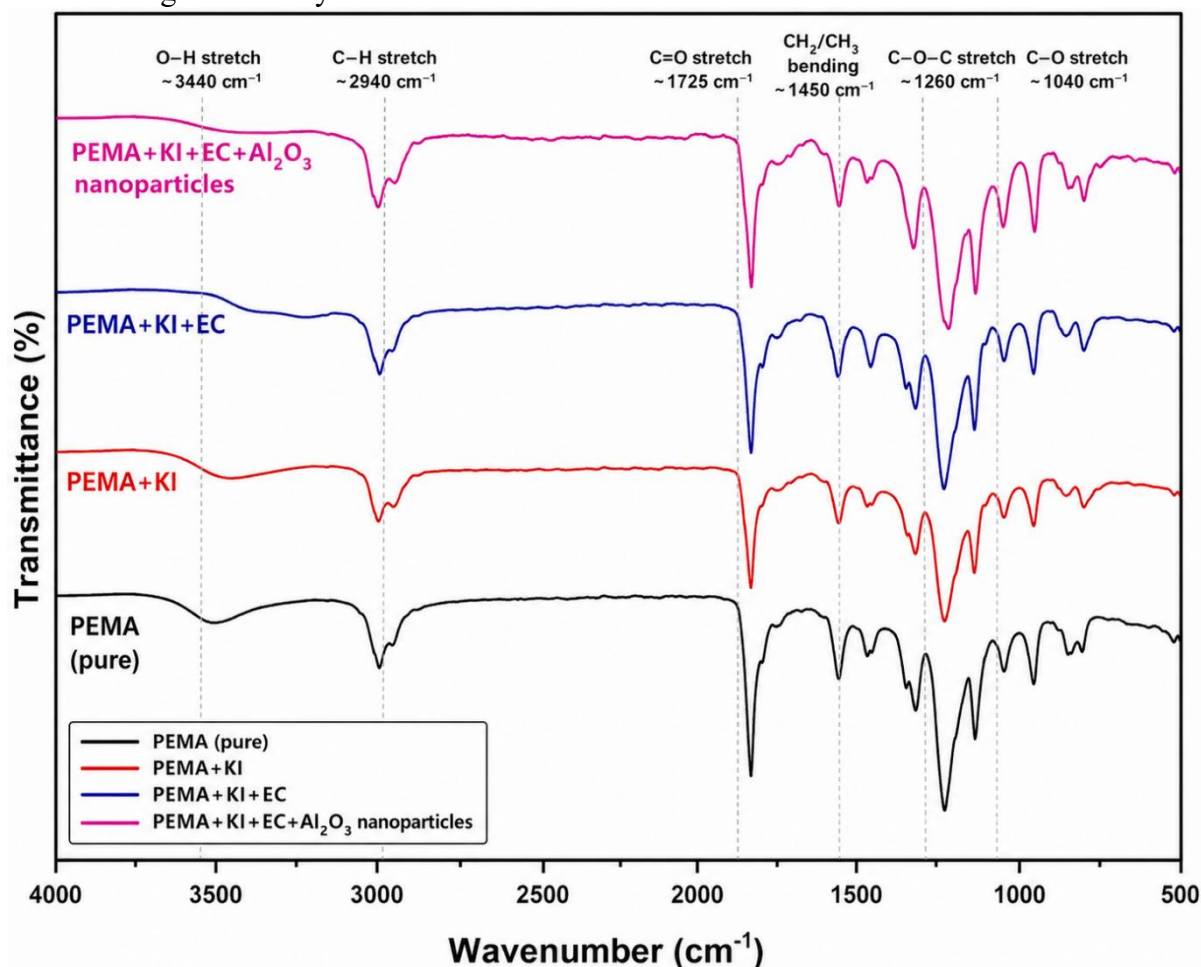


Fig. 5 FTIR analysis comparison of different polymer composite

Due to the addition of Al_2O_3 some additional peaks of Al_2O_3 nanomaterial were observed at 600 cm^{-1} and 750 cm^{-1} .

Table 2 Different vibrational band analysis using FTIR

Materials	Wavenumber (cm^{-1})	Band assignment
PEMA	843,965	CH_2 twisting
	1100	anti-symmetric C-O-C stretching
	1242,1280	asymmetric CH_2 twisting
	2700-2800	asymmetric C-H stretching
EC	720	C=O bending (Pradhan et al. 2005)
	1750	C=O stretching bands
	1805	C=O stretching bands
Al_2O_3	650,750	C=O bending
	1825,1850	C=O stretching band

The FTIR spectrum of pure PEMA shows characteristic bands at 3430–3450 cm^{-1} (O–H stretching due to absorbed moisture), 2950–2990 cm^{-1} (C–H stretching), 1720–1735 cm^{-1} (C=O stretching of the ester group), 1450–1380 cm^{-1} (CH_2/CH_3 bending), and 1260–1140 cm^{-1} (C–O–C stretching).

After incorporation of **KI**, slight shifts and intensity changes in the C=O and C–O–C bands indicate coordination of K^+ ions with the oxygen atoms of PEMA, confirming polymer–salt interactions. Addition of **EC** causes further band broadening and shifts toward lower wavenumbers, indicating enhanced interactions and increased polymer-chain flexibility and amorphous character.

For **PEMA–KI–EC–Al₂O₃**, further changes in the C=O and C–O–C bands confirm interactions between Al₂O₃ nanoparticles and the polymer electrolyte through Lewis acid–base interactions. The absence of new characteristic bands indicates that no new chemical compound is formed. Al₂O₃ primarily modifies the polymer–salt environment, promotes salt dissociation, increases the amorphous fraction, and facilitates ion transport, thereby contributing to enhanced ionic conductivity.

3.5. XRD Analysis

The crystalline or amorphous nature of the polymer composite electrolyte is determined using the XRD method. Shimadzu diffractometer (XRD 6100, USA) was used to record the X-ray diffraction study. An XRD diffractometer with an operating voltage of 40 KV and an electrical current of 30 mA was used to meet the criteria. The XRD investigation presented in Fig. 6 revealed the following observations, including the crystalline form of Pure PEMA (Fig. 6). A prominent peak appeared in the pure PEMA composite when salt was added, indicating that the crystalline area of the pure PEMA had been disturbed. This resulted in a drop in both the intensity and degree of crystallinity of the PEMA. The lack of amorphousness in this composite prevented ions from moving freely throughout the components.

EC plasticizer was added to the polymer composite material to enhance its amorphous nature, as depicted in figure 6. It was now noticed that the composite, which is depicted in black in fig. 6, was more amorphous. It was found that adding Al₂O₃ to the polymer composite removed sharp peaks. The mechanical strength characteristics of the polymer composite decreases with addition of EC; however, this strength can be increased by incorporating nano composite Al₂O₃ into the red-coloured polymer matrix. The sample was found to be more amorphous than the PEMA+KI+EC variant sample.

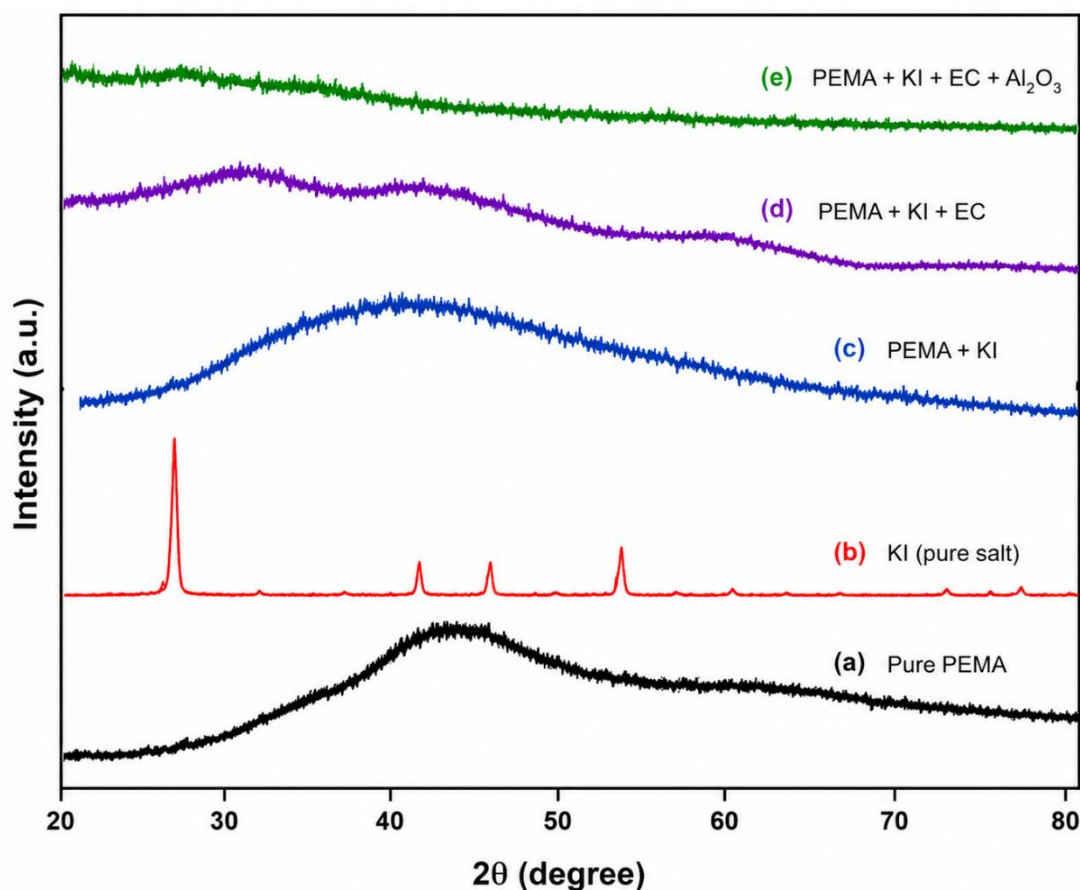


Fig. 6 XRD analysis comparison of polymer composite PEMA+KI+EC, PEMA+KI+EC+Al₂O₃

4. Differential Scanning Calorimetry

Glass transition temperature of the maximum conducting sample was studied in this work using PerkinElmer DSC 800. If the sample's glass transition temperature decrease future means sample was more amorphous so that conductivity of the sample increase (Turnbull and Cohen 1961). In this study a single peak occurrence, its significance that proper potassium iodide salt (KI), ethylene carbonate (EC) and Alumina (Al₂O₃) materials has good compatibility with polymer PEMA(Saxena et al. 2021).

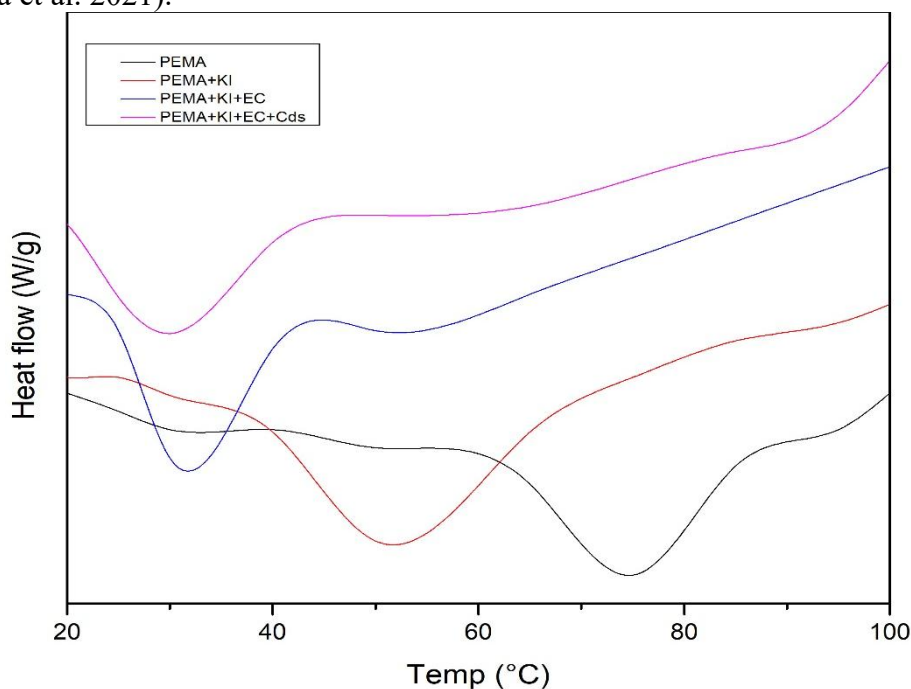


Fig. 7 Glass transition temperature of various maximum conducting sample.

PEMA polymer has 73°C glass transition temperature(Anuar et al. 2012) and its value was decreased when potassium iodide salt was added. To make the sample more conductive and amorphous EC was added and further the glass transition temperature reduce its significance that sample was more amorphous rather than previous sample(Qian et al. 2019; Dudowicz et al. 2005). After that maximum conductive sample incorporated with nanomaterial Al₂O₃ and DSC analysis indicate that glass transition temperature further decreased shown in fig. 7. Table 3 shows various glass transition temperature values of different samples.

Table 3 Various glass transition temperature values of different samples.

Materials	Glass Transition Temperature
PEMA	73°C
PEMA+40%KI	52°C
PEMA+40%KI+60%EC	30°C
PEMA+40%KI+60%EC +7%Al ₂ O ₃	22°C

5. Current and Voltage characteristics

Using a Keithley 2400 source meter and Sun simulator, the current and voltage characteristics of DSSC (Dye-Sensitized Solar Cell) have been determined under 1 sun condition. Iodine was added to PEMA+KI + EC + Al₂O₃ matrix with maximum ionic conducting value to generate redox couple. The DSSC's performance is determined using the following formula.

$$FF = \frac{V_{max} J_{max}}{V_{oc} J_{sc}}$$

$$\eta = \frac{V_{oc} J_{sc}}{P_{in}} FF$$

FF stands for fill factor, η photon to electron conversion efficiency, V_{oc} is open circuit voltage (V), V_{max} maximum voltage, J_{max} (mA/cm²) current density and I_{sc} short circuit current. The current and voltage relationship are shown in fig. 8. To calculate fill factor and efficiency of DSSC cell various parameters observed and these parameters are shown in table 4.

Table 4 Observed parameters to calculate fill factor and efficiency

I_{sc} (mA)	V_{oc} (V)	J_{sc} (mA/cm ²)	FF	η
1.91	0.686V	3.82	79.77	2.09

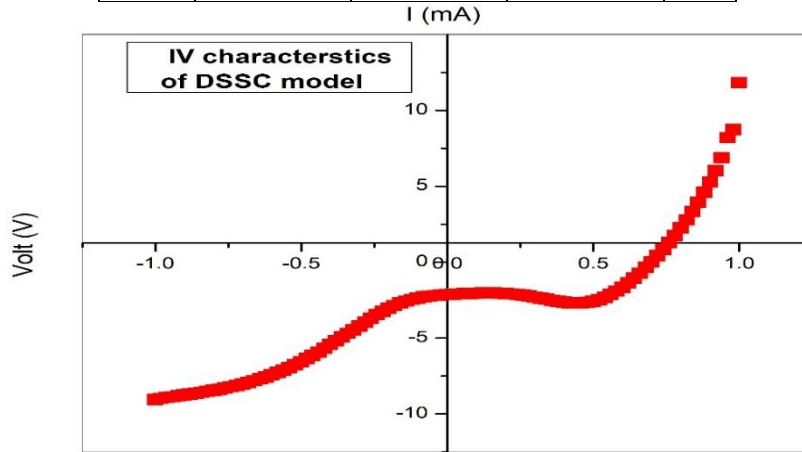


Fig. 8 I-V characteristics of DSSC solar cell

At 1 sun condition the prepared DSSC cell model showed an overall efficiency of 2.09%. having parameters short circuit current density J_{sc} = 3.82mA/cm², V_{oc} = 0.686V and fill factor=79.77%.

6. Conclusion

The highest conductivity of solid polymer composite (PEMA+KI) is 3.80×10^{-6} S/cm at 40% weight percentage of KI, EC was added to polymer composites at different weight percentages to increase their ionic conductivity and make composite more amorphous by reducing crystallinity. At 60% wt. percentage of EC, the maximum conductivity was 4.65×10^{-5} S/cm. Al₂O₃ was added to the polymer composite at different weight percentages to increase its ionic conductivity. At 7%, weight percentage the maximum conductive sample obtained having 2.65×10^{-3} S/cm. The polymer electrolyte composite crystalline or amorphous was analysed by using FTIR analysis, XRD analysis and further it was investigated using DSC analysis. To investigate the homogeneous mixing of polymer with different components, SEM analysis was employed. The DSSC was developed using the maximum conducting sample, and its efficiency was determined 2.09% under 1 sun condition having fill factor 79.77 and an open circuit voltage is 0.686V.

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