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## Original Article

## Ionic liquid (1-ethyl-3-methyltricynomethanide) doped polymer electrolyte (polyvinyl alcohol) for sustainable energy devices



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## ABSTRACT

Polyvinyl alcohol (PVA) polymer mixed with different weight ratios of 1-ethyl-3-methyltricynomethanide (EMIm-TCM) ranging from 0 to 30 wt% were used to develop solid polymer electrolytes (SPEs). The structural, electrical, and electrochemical properties of the SPEs films were extensively studied. Introducing ionic liquid (IL) into PVA's structure improved its mechanical properties, flexibility, and conductivity, increasing application potential in energy devices. The maximal ionic conductivity of  $3.85 \times 10^{-4} \text{ Scm}^{-1}$  was achieved by adding 30 wt% IL. Structural and morphology analysis was performed. Obtained materials exhibited enhanced amorphous nature, and the FT-IR spectrum showed perfect complexation, supported by our XRD pattern. The ionic transference number measurement ( $t_{\text{ion}}$ ) confirms that ionic liquid doped polymer electrolyte (ILDPE) is predominantly ionic. Several devices were tested. ILDPE film sandwiched Electric double-layer capacitor (EDLC) and Dye-Sensitized Solar Cell (DSSC) showed a favorable response.

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## 1. Introduction

All nations are boosting renewable energy generation to reduce carbon emissions and counteract climate change. Therefore, advanced energy storage systems are in high demand. Renewable energy cannot be produced equally across time. Solar energy cannot be generated at night, while wind and tidal power are climate dependent. As a result, the requirement for efficient energy storage systems that enables adequate energy storage and usage is caused by dynamic renewable energy development [1].

**Abbreviations:** PVA, Polyvinyl Alcohol; SPE, Solid Polymer Electrolyte; IL, Ionic Liquid; EDLC, Electric Double Layer Capacitor; DSSC, Dye Sensitized Solar Cell.

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Furthermore, the ionic transport properties and structural stability are essential and responsible for energy storage devices' efficient outputs.

Organic salts comprising ionic liquids are generally liquid (solvent-free) at ambient temperature. They are made up entirely of ions and are regarded as 'green' materials with intriguing qualities. They include being an excellent solvent for various organic and inorganic chemicals, highly polar yet non-coordinating, non-volatile, and with adjustable solubility and miscibility. Previous research has shown that one of the most advantageous techniques is to advance safety energy concurrently, including ILs in the electrolyte system enhanced device performance [2]. There are now several ILs of cation and anion combinations available. Still, imidazolium cation-based-ILs are of particular interest because of their high conductivity, which has been related to matching the imidazolium ring's cationic core [3]. There are several advantages to using ILs as EDLC electrolytes. They have a high electrochemical stability window (up to 6 V) and a high built-in capacitance, making

them suitable for high-energy electrochemical devices. Because of their incombustibility and low vapor pressure, they are less vulnerable to high temperatures. ILs, on the other hand, are immobile liquids. Using a liquid electrolyte has many practical and environmental disadvantages, including flaming concerns and the danger of leakage [4,5].

ILDPE have recently been prepared using various polymers such as poly (ethylene oxide) (PEO), poly (methyl methacrylate) (PMMA), poly (acrylonitrile) (PAN), and poly (vinylidene fluoride) (PVDF), polyvinyl alcohol (PVA) [6–8]. Each polymer has several advantages as well as disadvantages. Early investigations of PEO-based polymer electrolytes revealed poor ionic conductivity due to strong polymer crystallinity and limited polymer chain mobility. PMMA as a matrix in gel electrolytes demonstrated that PMMA has minimal ionic conductivity and no effect on gel electrolytes' electrochemical stability. Although PAN and PVDF-based gel electrolytes allow the creation of homogeneous gel films, PAN is inert in ionic transport processes. PVDF has a high dielectric constant, allowing for more charge carriers. Despite this, most of the polymers above exhibit limited ionic conductivity, high crystalline structure, and low interfacial chemical stability. Above all, pure PVA polymer and PVA nanocomposites have been actively researched among the mentioned polymeric hosts because of their exceptional features and possible uses in numerous science and technology domains. PVA possesses a semicrystalline structure owing to its hydroxyl groups (Fig. 1), which absorb a considerable quantity of water and hence the possibility of ionic conductivity enhancement [9].

The development of novel energy storage devices is critical owing to the limits of ionic conductivity enhancement by doping salts in polyether matrix. ILDPPE plays a dual role in the polymer electrolyte area, i.e., plasticizer and ionic source provider. In this communication, we report a new polymer electrolyte system doped with low-viscosity ionic liquid (18 cP). Using the same ILDPPE, we have developed dual energy devices, i.e., EDLC and DSSC, which shows that ILDPPE could be a novel approach to creating highly efficient energy devices [7–10].

## 2. Experimental

Polyvinyl alcohol (PVA) [Sigma Aldrich], 1-Ethyl-3-methyl-imidazolium tricyanomethanide (EMIm-TCM) [TCI Japan], and Dimethyl sulfoxide (DMSO) [TCI Japan].

The solution casting technique is followed to prepare ILDPPEs using PVA polymer as host and ILs as dopants. First, PVA is optimized at a specific temperature in DMSO solvent to prepare the homogenous solution followed by continuous stirring (24 h) with different (wt%) of IL. Several solutions (ranging from 0–40 wt%) were added using a micropipette and rigorously stirred for 24 h until the completely complexed solution was obtained. These complex solutions were finally poured into poly-propylene petri dishes to achieve mechanically stable solid electrolyte films (Fig. 2). We have observed brittle film after 40 wt% of IL concentration. Hence, we have only fixed our study to 40 wt% IL concentration, showing stable, flexible, and mechanically durable films (Fig. 2.)

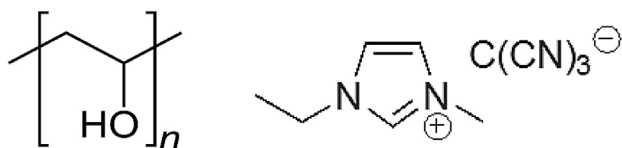


Fig. 1. Polyvinyl Alcohol and EMIm-TCM.

EDLC device was fabricated using activated porous carbon derived from bio-polymer, as reported in the literature [11]. Conducting graphite sheets of  $1 \times 1 \text{ cm}^2$  were used as a current collector, and a solid polymer electrolyte was sandwiched between the two electrodes (Fig. 3). In a standard procedure to develop DSSC, we have already maximized each component, as reported in the literature [12].

PVA and IL-doped PVA solid polymer electrolyte films were characterized using several techniques such as Electrochemical Impedance Spectroscopy (EIS), Ionic transference number ( $t_{ion}$ ), and electrochemical stability window (ESW) utilizing a CH instrument workstation (model 604D, USA). Polarized optical micrographs (POM) were created using a polarized optical microscope (Motic model no. BA310 Pol, Carlsbad, California, USA) at 100x magnifications. At the same time, FT-IR spectroscopy was performed using PerkinElmer Spectrum Version 10.4.00 in the 4000–400  $\text{cm}^{-1}$  range. To assess the electrochemical performance of the fabricated EDLCs, cyclic voltammetry (CV), low-frequency impedance spectroscopy, and galvanostatic charge/discharge tests were performed on the CH electrochemical workstation (604D Austin, Texas, USA). DSSC performance has been evaluated using Solar Simulator (Enlitech, model no–SS–F5–3A, Taiwan).

## 3. Results and discussion

### 3.1. Electrical characterizations (ionic conductivity, $t_{ion}$ , LSV)

All ILDPPEs made of ionic salt that dissolves in a polymer under the solvent medium shows both resistive and capacitive behavior as expressed by the real ( $Z'$ ) and imaginary ( $Z''$ ) components of the complex impedance ( $Z^*$ ) shown by:

$$Z^* = Z' + iZ'' \quad (1)$$

From the Impedance Spectroscopy in these ILDPPEs (Fig. 4(i)) [13], it appears a hike in the impedance curve, which is due to the movement of the ionic species at the electrode and electrolyte interface. Additionally, the bulk resistance of each system is thoroughly analyzed from the EIS curve to measure the ionic conductivity of these ILDPPEs using the formula:

$$\sigma = \frac{1}{R_b} \times \frac{l}{A} \quad (2)$$

where  $R_b$  is bulk resistance,  $l$  is the thickness of the polymer electrolyte film, and  $A$  is the electrode area where  $\sigma$  values are shown in Table 1 and Fig. 4(ii) [14].

It is clear from Fig. 4(i) and Table 1 that conductivity  $\sigma$  increases with IL wt% concentration and then decreases after a specific composition of IL. Single/Double conductivity maxima in these ILDPPEs are well documented in literature where  $\sigma$  enhancement is depicted due to enhancement of ionic charge species ( $n$ ) or decreases in crystallinity. In contrast, a decrease in  $\sigma$  value is correlated with ionic charge formation at the interface. The maximum  $\sigma$  value is at 30 wt% IL concentration with  $\sigma$  value of  $3.85 \times 10^{-4} \text{ Scm}^{-1}$ . This ILDPPE has been sandwiched between two symmetric porous carbon-based electrodes to further developed EDLC.

Fig. 4(iii) shows ionic transference number measurements ( $t_{ion}$ ) to confirm the ionic nature of our ILDPPEs. The ionic transference number ( $t_{ion}$ ) is calculated using Wagner's polarization method with the formula as indicated below:

$$t_{ion} = \frac{\text{Initial current} - \text{Final current}}{\text{Initial Current}} \quad (3)$$



Fig. 2. Highly flexible Ionic liquid doped solid polymer electrolyte (ILDPE) films.

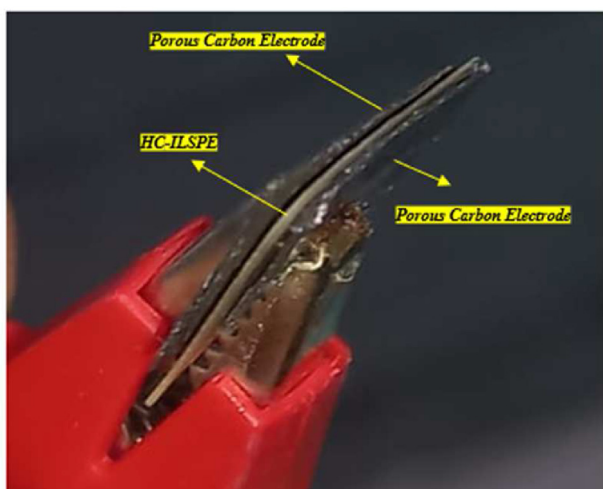


Fig. 3. Fabricated Laboratory scale sandwiched EDLC and DSSC using maximum conducting ILDPes as electrolyte.

From the curve presented in Fig. 4(iii), the  $t_{ion}$  was found to be 0.95, showing that our ILDPes are ionic and suitable for varying energy storage applications.

Moreover, to check the electrochemical stability window of ILDPes, we have carried out Linear Sweep Voltammetry (LSV). A typical curve of maximum conductivity films, where the voltage in the  $-3$  to  $3$  V range was applied. For the measurement, the film was positioned between two stainless steel electrodes. The curve presented in Fig. 4(iv) shows that the stability window is 4V which shows the promising nature of these ILDPes for vast electrochemical applications [15].

### 3.2. Optical characterization

Fig. 5 depicts polarized optical microscopy (POM) pictures of the samples. Pure PVA (Fig. 5 (a)) film morphology shows a dense structure where we can see a large number of spherulites [16]. After adding the 30 wt% of IL, i.e., PVA+30 wt% ILDPE, we can see a reduction in dense structures and an increase in the black region (Fig. 5(b)). These black regions already assign an amorphous nature to film, which always enhances ionic conductivity. These morphological parameters certainly assisted our assumption that doping with IL may reduce the crystallinity and improves  $\sigma$ , which is our prime aim in these ILDPes system.

### 3.3. Structural characterization

FTIR spectroscopy was performed in the  $4000$  to  $400$   $\text{cm}^{-1}$  range to confirm the ionic interaction with pure PVA using ionic liquid (Fig. 6) [17]. From the spectra of pure PVA, the OH stretching vibration of the hydroxyl group of PVA, also C–H symmetric and C–H asymmetric stretching vibrations, are observed at  $2844$   $\text{cm}^{-1}$  and  $2920$   $\text{cm}^{-1}$ , respectively. The acetyl group in the backbone of PVA corresponding to C=O stretching is shown at  $1087$   $\text{cm}^{-1}$ . The bending and wagging of  $\text{CH}_2$  vibration are confirmed at  $1087$   $\text{cm}^{-1}$ . Also, the spectra of ILDPE show the perfect complexation of ionic liquid with a pure polymer exhibiting a shift of bands.

A close comparison of pure PVA and ILDPE clearly shows that no new peaks appeared in ILDPE, proving its composite nature. Additionally, shifting in peak shows interaction/complexation between polymer and IL.

## 4. Device performance (specific capacitance measurement)

### 4.1. Low-frequency impedance

The performances of fabricated EDLC (specific capacitance  $C_{sp}$ ) were characterized using different electrochemical techniques such as alternating current (ac), low-frequency impedance spectroscopy, cyclic voltammetry, and galvanostatic charge–discharge methods. The low-frequency impedance measurements were carried out using CH electrochemical workstation in a frequency range of  $0.01$  Hz– $10^6$  Hz. The overall capacitance value of the fabricated EDLC was calculated using the equation:

$$C_{sp} = \frac{-1}{\omega Z''} \quad (4)$$

where  $\omega$  represents the angular frequency and  $Z''$  represents the imaginary component of the total complex impedance  $Z'$ . The specific capacitance values of a single electrode were calculated by multiplying the overall capacitance by a factor of two and dividing by the mass of the material on a single electrode.

Fig. 7 shows a semicircle in a high-frequency region representing the parallel combination of bulk resistance, i.e.,  $R_b$  and geometrical capacitance, i.e.,  $C_g$ . A steeply rising hike toward a low-frequency region is seen, leading to the formation of the double-layer capacitor at the two identical interfaces caused by interfacial polarization [18,19]. The specific capacitance was calculated as  $32.4$   $\text{Fg}^{-1}$  in a frequency range of  $0.01$  Hz– $10^6$  Hz.

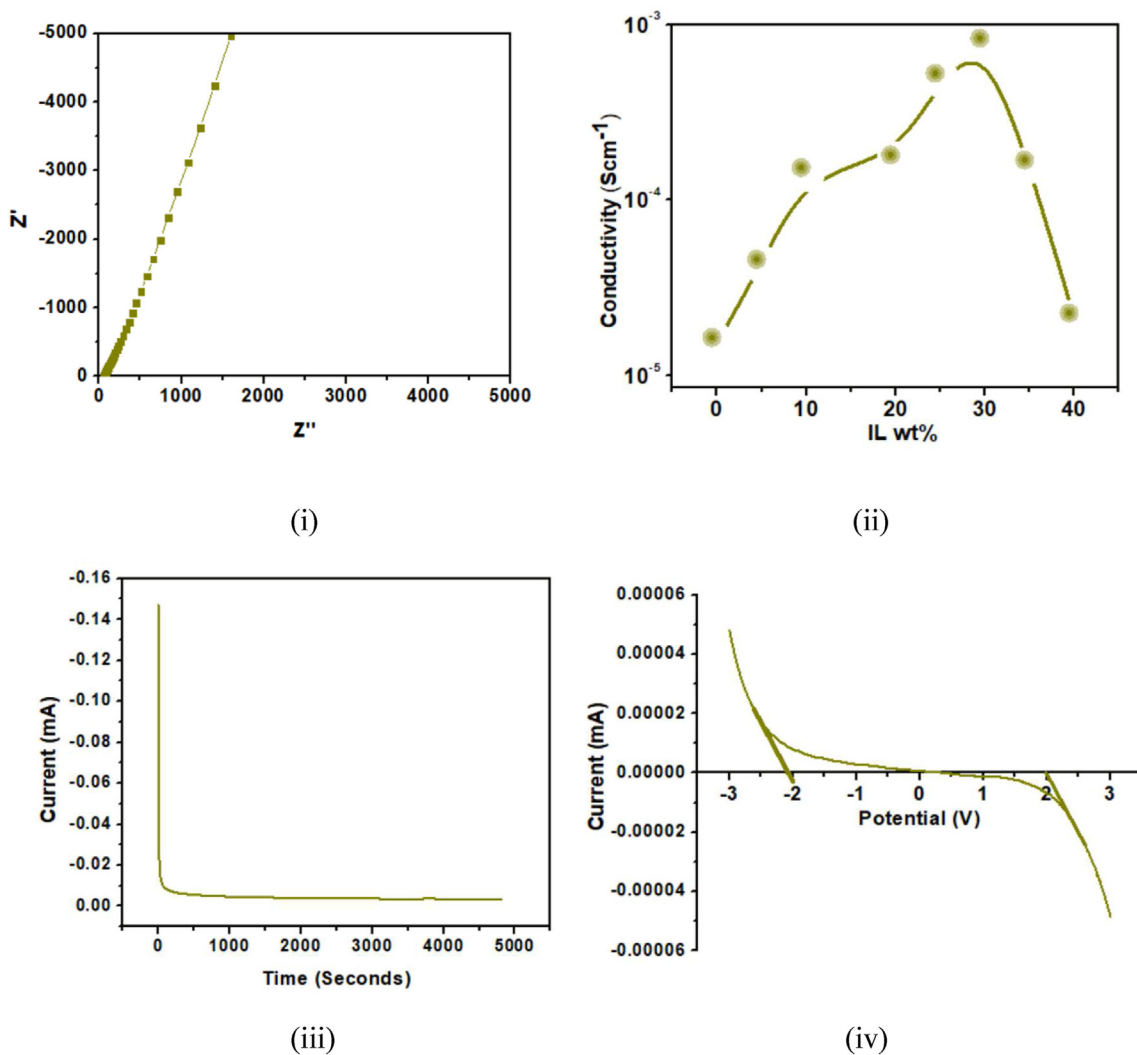


Fig. 4. Electrical characterizations of HC-SPE (i) Impedance Spectroscopy (ii) Conductivity Measurements (iii)  $t_{ion}$  (iv) ESW measurements.

**Table 1**  
Conductivity data in relation to the amount of ionic liquid concentration.

Ionic Liquid Concentration (wt%)	Conductivity ( $\text{Scm}^{-1}$ )
0	$5.42 \times 10^{-8}$
5	$1.04 \times 10^{-5}$
10	$8.20 \times 10^{-5}$
20	$8.89 \times 10^{-5}$
25	$8.89 \times 10^{-5}$
30	$3.85 \times 10^{-4}$
35	$2.37 \times 10^{-4}$
40	$7.64 \times 10^{-5}$

#### 4.2. Cyclic voltammetry (CV)

The CV measurement was carried out to determine  $C_{sp}$  according to an equation:

$$C_{sp} = \frac{i}{s} \tag{5}$$

where “i” represents the current and “s” represents the scan rate ( $\Delta V/\Delta t$ ).

An ideal capacitor with very low or negligible internal resistance shows the perfect rectangular shape that is scan rate dependent. Fig. 8 presents the deviation from the rectangular shape, which corresponds to the real capacitor performance, and is a series combination of internal resistance and ideal capacitance [20].

The value of specific capacitance was calculated from the characteristic presented in Fig. 8, and it was found to be  $58 \text{ Fg}^{-1}$  for the voltage range  $-1$  to  $1 \text{ V}$  and a scan rate of  $50 \text{ mV/s}$ .

#### 4.3. Dye-Sensitized Solar Cell performance analysis

Recorded I–V curve of laboratory scale DSSC is shown in Fig. 9. Evaluated parameters are given in Table 2. Fabricated DSSC showed 1.3% efficiency at 1 sun condition.

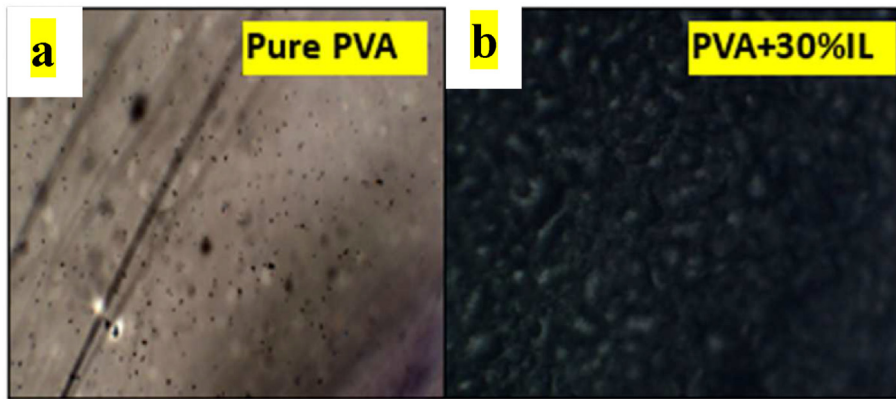


Fig. 5. Polarized Optical Microscopy.

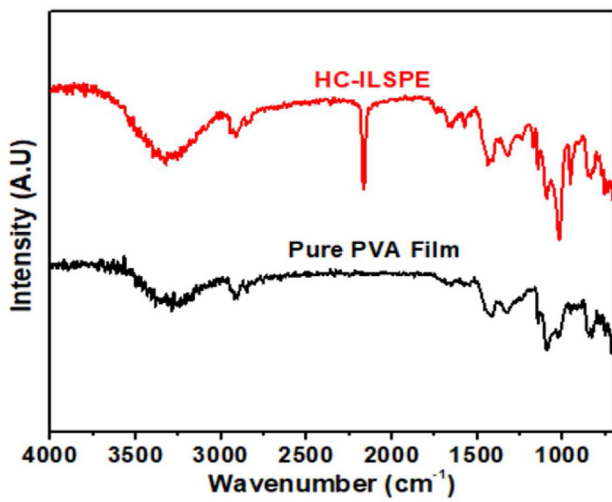


Fig. 6. FTIR Spectra of Pure PVA and ILDPE.

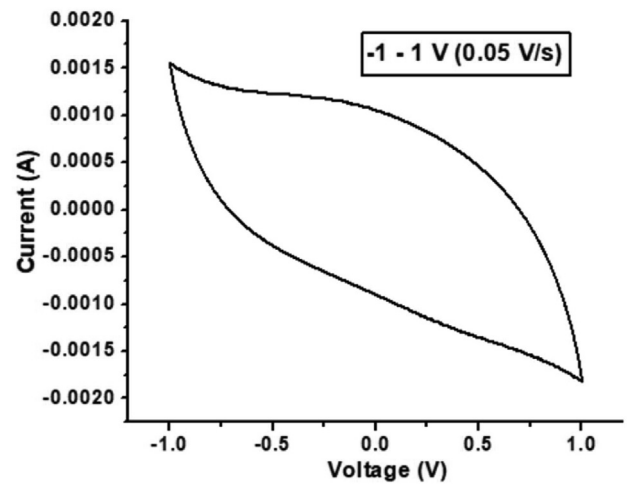


Fig. 8. Cyclic Voltammetry of fabricated EDLC.

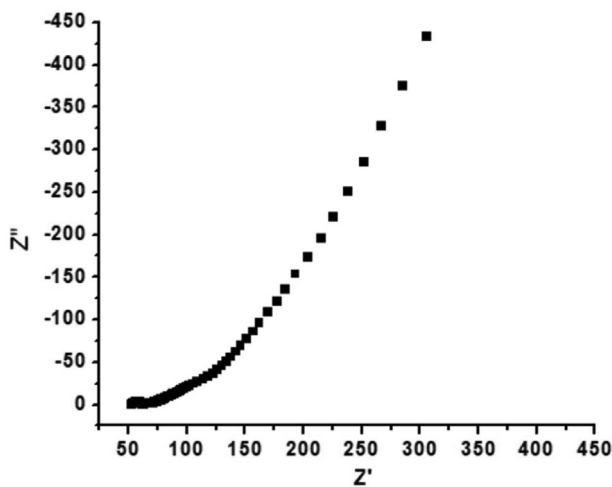


Fig. 7. Low-Frequency Impedance Spectroscopy of EDLC measured in our laboratory.

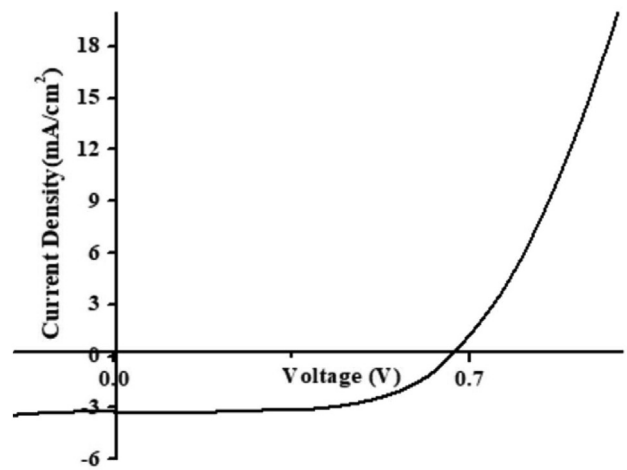


Fig. 9. J-V characteristics of the DSSC device.

**Table 2**  
DSSC performance at 1 sun condition.

Isc (mA)	Voc (V)	Jmax (mA)	Vmax (V)	Efficiency (%)	Fill Factor (%)	Jsc (mA/cm <sup>2</sup> )
0.64	0.66	2.71	0.48	1.3	61	3.23

## 5. Conclusion

Using the solution cast technique, ionic liquid doped polymer electrolytes (ILDPE's) were prepared and analyzed using impedance spectroscopy, ionic transference number, LSV, POM, and FTIR. Doping ionic liquid enhances ionic conductivity with a conductivity value of  $3.85 \times 10^{-4}$  S/cm at 30 wt% ionic liquid concentration. Ionic transference number measurement ( $t_{ion}$ ) shows that ILDPES are predominantly ionic in nature, while LSV confirms good stability (up to 4 V). Doping ionic liquid confirms the reduction of PVA's crystallinity, which is confirmed by POM. FTIR study shows composite nature and complexation behavior. Using maximum obtained conductivity ( $3.85 \times 10^{-4}$  S/cm) of the film sandwiched EDLC at room environment conditions using porous carbon symmetric electrodes shows  $C_{sp}$  up to 50 F/g and DSSC performance of 1.3% at 1 sun condition.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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