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Ionic liquid (1-Ethyl-3-methylimidazolium tricyanomethanide) incorporated corn starch polymer electrolyte for solar cell and supercapacitor application

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A R T I C L E I N F O

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A B S T R A C T

Taking into account energy demand a new highly conducting ionic liquid (IL) c (EmImTCM) mixed corn starch (CS) biopolymer electrolyte is synthesized for dual electrochemical application electric double layer capacitor (EDLC) and the dye-sensitized solar cell (DSSC) application. Electrical, structural, thermal, and optical studies are carried out in detail and presented in this communication. Maximum conducting IL-incorporated biopolymer electrolyte film has been sandwiched between electrodes to develop EDLC and DSSC. The sandwich-structured EDLC delivers a high specific capacitance of 250 F/gram while DSSC shows 1.44 % efficiency at one sun condition.

1. Introduction

The development of electrochemical devices has a rich history that dates back to the early 16th century, culminating in notable inventions such as the first battery by Alessandro Volta in 1800 and the pioneering dielectric capacitor by Pieter van Musschenbroek around 1746. Over the centuries, these technologies have evolved significantly, showcasing remarkable advancements that have continually pushed the boundaries of innovation and efficiency. However, facing numerous challenges related to the stability and lifespan of devices. Moreover, the widespread adoption of these devices has raised concerns regarding environmental pollution, a problem that has grown exponentially over time. In response, researchers have increasingly focused on developing more sustainable technologies using biodegradable and recyclable materials. Recent studies by Singh et al. (2022), Konwar et al. (2023), Mei et al. (2018), and Muchakavala et al. (2018) highlight significant advancements in this area[1–4].

Recent advancements in electrochemical research have led to innovative substitutions in the materials used in energy devices, with a significant shift from traditional liquid forms to polymers and more sustainable alternatives. In recent years, the traditional liquid electrolytes in electrochemical devices are being replaced with gel. The use of gel or solid polymer electrolytes in electrochemical devices offers superior performance by enhancing stability, reducing leakage risks, and improving safety profiles compared to their traditional liquid counterparts, which are more prone to volatility and degradation under operational stress [5–8]. Moreover, the use of biopolymers has gained immense attention for their environmental friendliness and

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biodegradable nature. The use of biopolymers varies widely depending on regional availability and production methods[5–8]. Among the most common sources of biopolymers are agricultural products like wheat and rice, which are major sources of carbohydrates, starch, and fiber globally. Other sources such as corn, sago, and barley also contribute significant amounts of these materials. Specifically, corn starch—rich in amylose and amylopectin—has not only been utilized as a fuel in the form of ethanol but is also being explored for its potential in electrochemical applications including batteries, supercapacitors, fuel cells, and sensitizer-based solar cells[9–12].

This study explores use of corn starch as a foundational biopolymer for the synthesis of ionic liquid biopolymer electrolyte (ILBPE), which plays a crucial role in the fabrication of advanced energy storage and conversion devices such as electric double-layer capacitors (EDLC) and dye-sensitized solar cells (DSSC). Ionic liquids, such as the stable compound 1-ethyl-3-methylimidazolium tricyanomethanide, are utilized alongside biopolymers to enhance the mobility of ions at temperatures below 100 °C [13–15], a key factor in improving the performance of electrochemical devices. This enhanced mobility is crucial for the performance of devices like electric double-layer capacitors (EDLCs), which are known for their ability to offer significantly higher capacitance compared to traditional dielectric capacitors[16–19]. Moreover, EmImTCM significantly enhances the performance of dye-sensitized solar cells (DSSCs) by improving ionic conductivity and maintaining stability within the electrolyte. Its integration with biopolymer-based electrolytes facilitates faster ion transport and better charge transfer, leading to reduced internal resistance and higher efficiency. EmImTCM's stability under varied thermal conditions and compatibility with biopolymers help create durable and efficient solar cells, aligning with sustainable energy technology goals[20–22].

In this comprehensive study, we have successfully synthesized a high-conducting biopolymer electrolyte using corn starch, potassium iodide (SSKI oral solution), and the organic ionic liquid 1-Ethyl-3-methylimidazolium Tricyanomethanide (EmImTCM) via the solution cast technique. Unique to our approach is the incorporation of this ionic liquid into the biopolymer matrix, which not only enhances ionic conductivity but also significantly improves the electrolyte's electrochemical stability and thermal properties. Our findings demonstrate a marked increase in the amorphous nature of the electrolyte, confirmed by X-ray diffraction (XRD) and polarized optical microscopy (POM), leading to higher ionic mobility and better performance in energy storage applications. The resultant electrolyte showcased exceptional performance in both electric double-layer capacitors (EDLCs) and dye-sensitized solar cells (DSSCs), achieving notable specific capacitance and efficiency improvements over conventional systems. This research highlights the potential of biopolymer-based electrolytes doped with ionic liquids as a sustainable and efficient alternative for the next generation of electrochemical devices.

2. Materials and methods

2.1. Materials used

Biopolymer corn starch (CN), salt potassium iodide (SSKI oral solution), binder Poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP), and solvent acetone are purchased from Hi-Media A-406, Bhaveshwar Plaza, LBS Marg, Mumbai-400 086, India; ionic liquid (IL) 1-Ethyl-3-methylimidazolium Tricyanomethanide (EmImTCM), is purchased from TCI Chemical India Private Limited, No.B-28, Phase-II, 5th Cross Street, MEPZ-SEZ, Kadaperi, Tambaram, Chennai, Tamil Nadu 600,045, India; current collector graphite sheet from Nikunj enterprises, Mumbai Maharashtra, India; and current collector fluorine-doped tin oxide (FTO), active material titania paste, N_7 dye and blocking layer titanium di-isopropoxide bis(acetylacetonate) are purchased from Sigma Aldrich, USA; Solvent double distilled (DD)- water and porous activated carbon produced in our laboratory.

2.2. Characterization tools

Electrochemical impedance spectroscopy (EIS) is used to evaluate ionic conductivity as well as the specific capacitance of the EDLC fabricated using the ionic liquid doped biopolymer Electrolyte (ILBPE). The highest conducting ILBPE are characterized using Wagner's DC polarization and linear sweep voltammetry (LSV) for the calculation of ionic transference number (t_{ion}) and electrochemical stability window (ESW) follow by x-ray diffraction (XRD), polarized optical microscopy (POM) for structural studies. Fourier transforms infrared spectroscopy (FTIR) for complexation and thermogravimetric analysis (TGA) for thermal studies. Cyclic voltammetry (CV), constant current charging and discharging (CCD) for EDLC, and LSV for DSSC performance analysis.

2.3. Synthesis of ionic liquid biopolymer electrolyte

For the synthesis of ILBPE, the host polymer is fixed at 200 mg dissolving in DD water to which an antibacterial agent of 20 mg of glutaraldehyde is added a avoid decay of the electrolyte film or fungal growth. The highest conducting KI incorporated biopolymer electrolyte is achieved for the composition of 2:1 CN: KI. Further ionic liquid EmImTCM of different weight% concentrations (2, 4, 6, 8, 10, 12, 14, 16, 18, 20) is added to this 2:1 composition of CN: KI to synthesize the highest conducting ILBPE. Solution is thoroughly stirred to achieve a homogenous mixture and is poured into polypropylene petri dishes and dried overnight at 50 °C. Finally, the films are used for analysis and electrochemical application[23,24].

2.4. Device fabrication

Fabrication of electric double-layer capacitor

To fabricate EDLC graphite sheet is cut in the size of 1 cm^2 are used as current collector. High-surface porous activated carbon synthesized from waste plastic is used as an active material of the electric double-layer capacitor (EDLC) electrode. A slurry containing 100 mg of active and 10 mg of (PVDF-HFP) is prepared using acetone. The graphite electrodes are coated using the paint brush and dried in oven. The highest conducting ILBPE is then sandwiched between two electrodes to fabricate the final EDLC[25,26].

Fabrication of dye-sensitized solar cell

To fabricate DSSC fluorine doped tin oxide (FTO) conducting glass is cut in the size of 3 cm^2 and washed several times with acetone in an ultrasonic bath and is used as current collector electrodes for both the working as well as the counter electrode of the DSSC. Initially, a thin layer of diluted (Titanium di-isopropoxide bis(acetylacetonate)) is coated over the FTO glass and annealed at 500 °C for 30 min which acts as a blocking layer. A thick layer of nanoporous titania paste is coated using doctor's blade method and further annealed at 500 °C for 30 min. The annealed electrode is finally dipped in a solution of N_7 dye solution for 6–8 h for proper absorption. Another clean and dried FTO is coated with a diluted solution of chloroplatinic acid and annealed at 500 °C for 30 min. The highest conducting ILBPE is modified by adding 10 mg of I_2 and a sandwich in between the working and the counter electrode to fabricate the final DSSC[27,28].

3. Results and discussion

3.1. Ionic conductivity

The bulk electrolyte resistance of all the synthesized ILBPE is measured using EIS technique within a frequency range of 10^2 Hz to 10^6Hz. In this technique, a low amplitude ac voltage is applied with a different selected frequency called decades. The value of current with respect to the applied voltage is measured. Since the system is a capacitive medium hence, a phase difference is developed which is used for the calculation of the real part of the impedance (Z') as well as the imaginary part of impedance (Z''). The plot between Z' and Z'' is called the Nyquist plot and the value of bulk resistance is determined to evaluate the ionic conductivity of different ILBPE films.

Using bulk resistance value in equation (1) ionic conductivity is determined using formula

$$\sigma = \frac{L}{R_b A} \quad \text{(i)}$$

where R_b is bulk resistance, L is thickness of film, A is area of film in contact of sample holder.

The evaluated ionic conductivity values of all the ILBPEs is tabulated in Table 1 plotted in Fig. 1.

From Fig. 1, it has been observed that conductivity increases attain maxima, and then decreases. The same trend is observed in the case of the synthesized ILBPE. Single maxima as well as double maxima in polymer electrolyte systems are widely available and cited in literature where conductivity enhancement is due to the increment in charge carrier and reduction in conductivity on further addition is due to the formation of charge pair ions [29,30]. The maximum ion conducting value achieved was 5.10×10^{-3} S/cm is attained for the composition ratio 2:1:0.01 of CN:KI: IL. This maximum conducting ILBPE is sandwiched between the electrodes to fabricate EDLC and DSSC. In ILBPE we restricted our studies after 16 wt% IL concentration due to non-availability of free-standing biopolymer electrolyte film.

3.2. Wagner's DC polarization

Wagner's DC polarization method is applied to measure the ionic transference number (t_{ion}) of the various ILBPE films. In this technique, a weak DC signal is applied across the ILBPE film for 4500 s. Initially the current shoot up and then rapidly decreased with time and finally stabilized. The stabilized current is termed the residue current due to the presence of an electron. The stabilization of current is due to the reduction of free ions within the electrolyte causing accumulation of ions leading to electrode polarization on the application of DC potential. A typical maximum conducting ILBPE graph is shown in Fig. 2. t_{ion} value has been evaluated using equation (ii)

$$t_{ion} = \frac{\textit{initial current} - \textit{final current}}{\textit{Initial current}} \quad \text{(ii)}$$

The value of an ionic transference number of 0.96 proves that the synthesized ILBPE film is predominantly ionic in nature.

3.3. Linear sweep voltammetry

An electrolyte consists of a fixed number of dissociated free ions at a certain condition of temperature and pressure. The number of free ions can be increased by supplying additional heat to the electrolyte by increasing temperature or by applying an electric field. The linear sweep voltammetry (LSV) technique is applied within a symmetrical voltage range −3.5 V to 3.5 V. From the LSV plot shown in Fig. 3, it is observed that the value of current is almost stable between 1.3 V to −2.5 V for the ILBPE giving an electrochemical stability window (ESW) of 3.8 V. The potential applied above 1.3 V to −2.5 V contributes to the increment in the number of free ions causing growth in current as well as heat which may lead to the failure of the electrolyte. The number of free ions will again get reduced on the removal of the electric field which will contribute to the loss in columbic efficiency and also a failure of the electrochemical device. Hence ESW is an important parameter to maintain the efficiency of the electrochemical devices.

Table 1
Ionic conductivity of ILBPE films.

ILBPE	
Composition (IL)	Conductivity S/cm
2	3.53×10^{-4}
4	6.67×10^{-4}
6	9.78×10^{-4}
8	2.10×10^{-3}
10	5.10×10^{-3}
12	9.97×10^{-4}
14	7.95×10^{-4}
16	7.57×10^{-4}

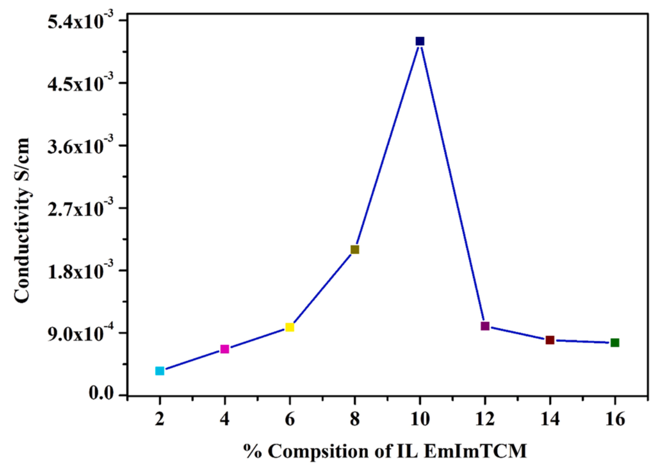

Fig. 1. Ionic conductivity vs IL composition (%) plot for ILBPE films.

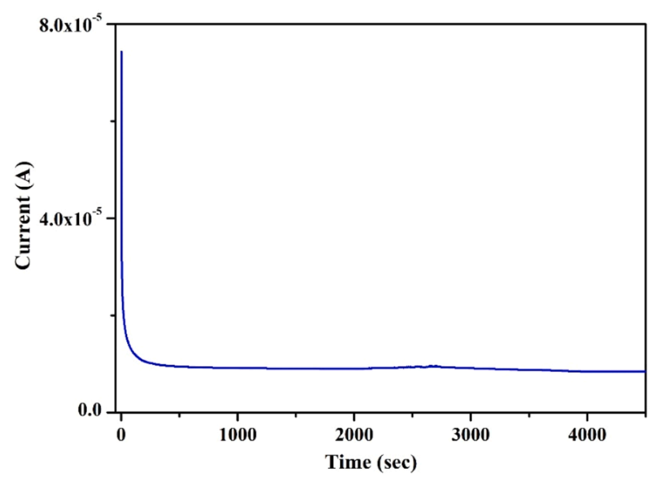

Fig. 2. Wagner's polarization curve to evaluate tion value in ILBPE maximum conducting film.

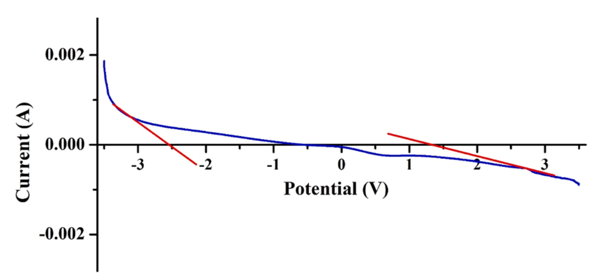

Fig. 3. Linear sweep voltammetry (LSV) plot for the maximum conducting ILBPE film.

3.4. X-ray diffraction

X-ray diffraction (XRD) technique is used to study the impact of the addition of salt KI and Ionic liquid EmImTCM into the matrix of the biopolymer corn starch. Fig. 4 shows the X-ray diffraction pattern of the corn starch, salt KI and the highest conducting ILBPE. The XRD pattern is recorded over a range of $2\theta = 10^\circ$ to 80°. The XRD pattern of the salt KI shows crystalline nature with different high-intensity peaks at 22°, 25°, and, 52°. While the XRD pattern of CN shows a broad peak between 10° to 45° with a maximum intensity of 20.5°. All the peaks of the salt KI vanished in the ILBPE showing the complete dissolution of salt.

Comparing all the three XDR patterns it is clear that the XRD pattern of ILBPE is free from all the peaks of the salt KI showing the proper dissociation of salt. Additionally, reduction in peak height along with broadness in peak confirm increase in amorphous nature. Amorphous region is well defined conductivity rich portion and hence addition of IL increases conductivity.

3.5. Fourier transform infrared spectroscopy

Fourier transform infrared (FT-IR) spectroscopy of CN, KI, IL, and ILBPE are recorded within wave numbers 4000 cm^{-1} to 400 cm^{-1} and plotted in Fig. 5. It is observed a broad peak for all the host material and ILBPE at 3200–3500 cm^{-1} depicted to -OH bond, The CN and IL both being organic compounds similar peaks are observed between 700 and 1700 cm^{-1} for various alkanes, alkene, C—H, O—N, etc. functional groups. An additional peak of IL at 2164 cm^{-1} for the amide group is also recorded. All the relevant peaks of the host materials are present in the ILBPE spectra with diminished intensity and shifting showing the formation of complexation due to the proper dissociation of salt and ionic liquid. The main peaks at 2164 to 2170 cm^{-1} could be easily identified. Close observation and comparative analysis between the host material and ILBPE confirm the composite nature of the ILBPE film.

3.6. Polarized optical microscopy

Polarized optical microscopy (POM) is an important imaging technique to identify the crystalline and the amorphous region present within the polymer and electrolyte. Fig. 6 shows the POM images of CN and ILBPE films. It is clear that CN contains smaller dark and light patch where the dark patch represents the amorphous region and the light patch represent the semi-crystalline region (Fig. 6(a). In Fig. 6(b) it is clearly visible that dark patches going to increase in ILBPE which confirms the enhancement of the amorphous region with the addition of salt/IL. The same trend is also supported by the conductivity and XRD data.

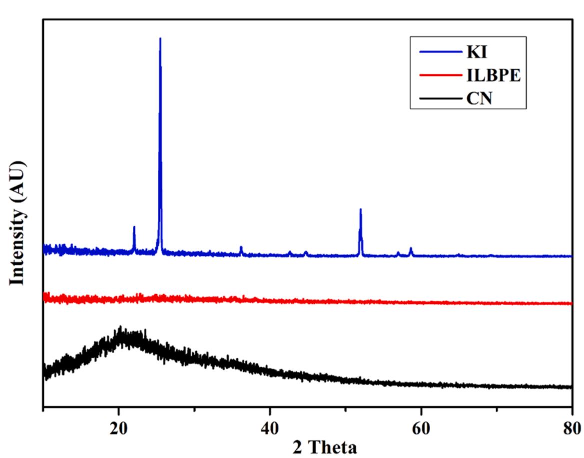

Fig. 4. X-ray diffraction (XRD) pattern of CN, KI, and ILBPE.

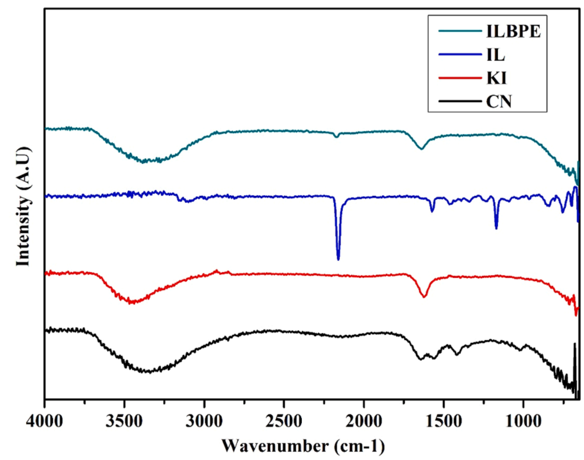

Fig. 5. FTIR spectra of CN, KI, IL, and ILBPE.

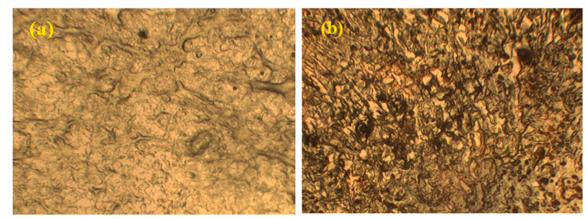

Fig. 6. POM images of (a)Pure CN, (b)ILBPE films.

3.7. Thermogravimetric analysis

Electrochemical devices are driven by endothermic redox and electrostatic reactions. These devices decapitate a lot of heat while charging and discharging which heat the electrolyte to a temperature higher than the surrounding temperature. High-temperature thermal stability hence becomes an important measurement for rapid charging and discharging. Fig. 7 shows the thermogravimetric analysis (TGA) of ILBPE films. It is seen that the ILBPE film suffers initial weight loss till 100 °C, which is due to the presence of a trace of DD water present in the films. The ILBPE film is stable to a temperature of 250 °C. The weight loss at higher temperatures is due to decay, pre-carbonization, and high-temperature reactions. The higher temperature stability of ILBPE makes it suitable for the application of devices at high charging and discharging rate and extreme temperature conditions.

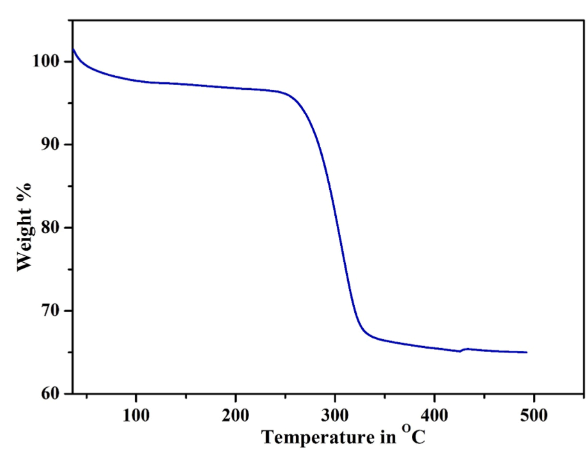

Fig. 7. Thermogravimetric analysis of the synthesized films.

3.8. Performance of EDLC

Low-frequency electrochemical impedance spectroscopy

Low-frequency electrochemical impedance spectroscopy (LFEIS) analysis of the EDLC cell is carried out within the frequency range of 10^6 Hz to 10^{-2} Hz. The Nyquist plot of the EDLC cell is plotted in Fig. 8 and is divided into three different regions. A semi-circular region, a linear region making an angle of $40^\circ - 45^\circ$, and finally a linear region making an angle greater than 45° at three different frequency ranges in between high and low frequency. The occurrence of a semi-circular region is the dielectric nature of the electrolyte and the two different regions occur due to the electrode-electrolyte interaction and ion absorption at the porous electrode.

The specific capacitance (C_{sp}) of the EDLC cell was calculated to be as high as 250 F/gram at a low frequency of 10¯2 Hz. The high specific capacitance is due to the contribution of a high conducting electrolyte along with the high surface area of porous carbon.

Cyclic voltammetry

Cyclic voltammetry (CV) is carried out to study the capacitive nature and the specific capacitance of the EDLC cell. The CV plot plotted in Fig. 9 resembles a rectangular shape which is observed in the case of EDLC because of the mobility of the ion towards the porous carbon electrode forming the Helmholtz layer in either electrode of the EDLC causing the non-faradic process of electrode polarization. CV is carried out at a potential window of 1 volt at a scan rate of 0.01 V/*sec*. Quantitatively, the specific capacitance of the supercapacitor cell has been evaluated by using the formula:

$$Csp = \frac{1}{m \times s \times \Delta v} \int_{v1}^{v2} I(v) dv \qquad \text{(iii)}$$

where I is current, s is scan rate, Δv is the potential difference between the initial (v_1) and final (v_2) voltage range, and m is the mass of active material at a single electrode. The fabricated EDLC cell has delivered a specific capacitance of 250 F/gram.

Constant current charging and discharging

The constant current charging and discharging (CCD) are carried out to evaluate the overall performance of the EDLC cell. In this technique, 4 mA of constant current is applied to charge and discharge the EDLC cell to a potential difference of 1.06 Vs. The discharge-specific capacitance (C_{dsp}), coulombic efficiency (C_{ef}), specific energy density (E_d), and specific power density (P_d) are calculated from the CCD plot plotted in Fig. 10. From this figure it is clear that fabricated EDLC shows the triangular shape resembles that of an EDLC well reported in the literature (first 6 CCD cycle). It is also observed that voltage increases exponentially from a potential difference of 0.32 V to 1.06 and drops suddenly to 0.72 Vs and decreases exponentially, the same trend is observed in all six cycles because of the presence of the internal resistance of the EDLC cell. The EDLC cell can deliver a maximum discharge specific capacitance of 250 F/gram with coulombic efficiency of 79 % followed by an energy density of 37 Wh/Kg and power density of 4080 W/kg.

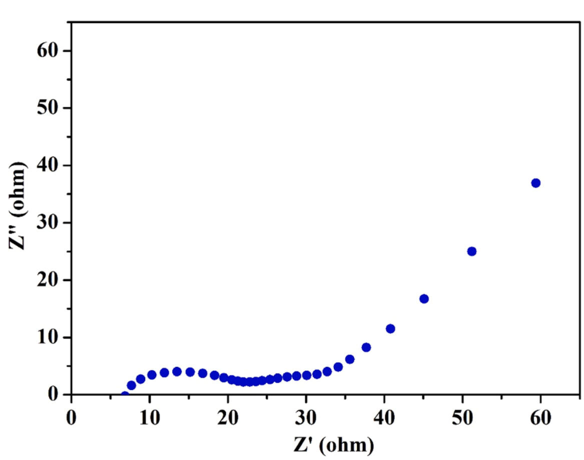

Fig. 8. Nyquist plot of the fabricated EDLC cell in the frequency range of 10^6 Hz to 10^{-2} Hz.

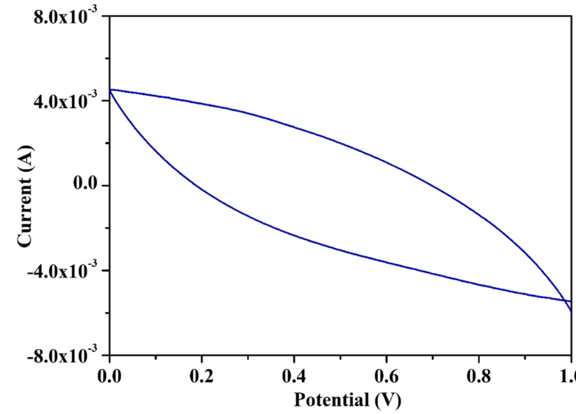

Fig. 9. Cyclic voltammetry profile of fabricated EDLC cell in the voltage range of 0 V to 1 V at 10 mV/s.

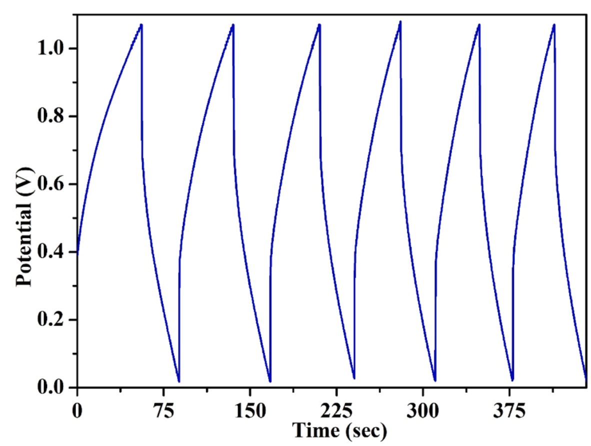

Fig. 10. CCD profile of the fabricated EDLC cell.

3.9. Performance of DSSC

The linear sweep voltammetry technique is a widely used method to test the performance of DSSC. In this technique, a controlled varying voltage between −0.2 V to 1 V is applied across the DSSCs irradiated at a simulated one-sun condition (100 W/m^2) using a solar simulator (Enlitech, Taiwan) at a scan rate of 0.1 V/*sec* by a source meter by Keysight. The 3rd quadrant of the current density (J) vs voltage (V) graph plotted in Fig. 11 is evaluated to measure the overall photon conversion efficiency (η) and the fill factor (FF) of the DSSC. The DSSC cell has produced a short circuit current of 2.52 mA/cm^2 at an open circuit voltage of 0.68 V which decreases at a peak load to the current of 2.39 mA/cm^2 and voltage of 0.63 Volt, FF of 73 with overall DSSC efficiency of 1.44 %.

4. Conclusion

A high conducting new biopolymer electrolyte consisting of inorganic salt KI and organic ionic liquid (EmImTCM) has been successfully synthesized using solution cast technique. EIS measurement confirm enhancement in ionic conductivity by salt/IL doping. This conductivity

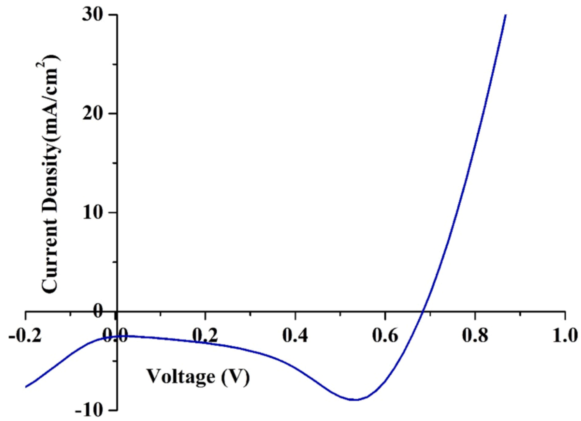

Fig. 11. J-V characteristics of fabricated DSSC using maximum conducting ILBPE film.

enhancement is attributed due to additional charge species provided by IL and also act as plasticizer. Maximum conductivity value as high as order 10^{-3} S/cm has been observed which seems its suitability towards development of dual energy applications EDLC and DSSC. The change in crystalline nature of the biopolymer corn starch in addition to KI or ionic liquid (EmImTCM) is explained by XRD and POM where reduction in crystallinity (increase in amorphous nature) has been observed. The formation of complexation along with composite nature is confirm by FTIR analysis followed by the high-temperature thermal stability of 200 °C using TGA measurement. High ESW value (3.8 volt) clearly affirm ILBPE suitability towards developing efficient electrochemical devices. The fabric electrochemical device EDLC has delivered an average specific capacitance of as high as 200F/g while DSSC delivering an efficiency of 1.44% at 1 sun condition.

CRediT authorship contribution statement

Subhrajit Konwar: Writing – original draft, Investigation. **Sushant Kumar:** Writing – review & editing, Writing – original draft. **Ahmad Azmin Mohamad:** Writing – review & editing, Supervision. **Amrita Jain:** Formal analysis. **Monika Michalska:** Formal analysis. **Vinay Deep Punetha:** Writing – review & editing, Software. **M.Z.A. Yahya:** Formal analysis. **Karol Strzałkowski:** Writing – review & editing, Formal analysis. **Diksha Singh:** Writing – review & editing, Formal analysis. **Markus Diantoro:** Formal analysis. **Faisal Islam Chowdhury:** Validation, Formal analysis. **Pramod K. Singh:** Writing – review & editing.

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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Data availability

Data will be made available on request.

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