

PEO Based Gel Polymer Electrolyte with Acetamide, KI/I₂ Added Composite for Dye Sensitized Solar Cell Applications

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Abstract - Gel polymer electrolytes (GPEs) are synthesized by PEO with added acetamide, KI/I₂ for dye-sensitized solar cells application (DSSC). The ability of the optimized gel polymer electrolyte was investigated by DSSC. GPEs prepared by addition of acetamide with various wt% in PEO (poly (ethylene oxide)) and PEGDME (poly (ethylene glycol) dimethyl ether) with KI/I₂. The best performance is attributed for PEO/PEGDME with 5 wt% acetamide provides highest efficiency ($\eta = 6.14\%$) under 100 mW/cm² illumination and exhibits highest ionic conductivity as $\sigma = 2.764 \times 10^{-3}$ S/cm at 300c among all electrolytes, compared to PEO without acetamide ($\eta = 5.11\%$). The short-circuit current density (J_{sc}) was increased for GPEs due to the increase in the recombination effect and electron lifetime by the adding of acetamide on the PEO. The power conversion efficiency is increased due to the growth in the ionic conductivity and amorphous nature of the GPE by the adding of acetamide on the PEO.

Keywords: Dye sensitized solar cells (DSSC), Gel polymer electrolytes (GPE), poly (ethylene oxide) (PEO), poly (ethylene glycol) dimethyl ether (PEGDME), Broadband dielectric spectroscopy (BDS).

1. INTRODUCTION:

Among different types of photovoltaic cell, the Dye sensitized solar cells (DSSCs) are promising and emerging device due to their simple assembly and low price. Grätzel fabricated Dye sensitized solar cell (DSSC) in 1991 and classified as 3rd generation solar photovoltaics [1]. Now, liquid electrolyte based redox couple used in DSSC and exhibit the photovoltaic conversion efficiency (η) approximately 13% [2-4]. They maintain superior interfacial interaction between the TiO₂ photoelectrode at the nanopores and electrolyte. However, the employ of liquid based electrolytes in DSSC makes some problems like long time constancy at the normal conditions. DSSCs also require airtight protection which makes scale up trouble for fabrication. This type of problems proposes substitute of the liquid electrolytes. The quasi-state solid electrolytes, gel polymer electrolytes are the well replacement which provides good ionic conductivity and ion mobility values. Hence, it is essential to develop dynamic, efficient and cost-effective DSSCs; it requires employing a redox-couple GPE with best iodine ion mobility and ionic conductivity [5-7]. GPEs are the well replacement for DSSC for the reason that the constancy of the electrolytes caused due to the interaction of redox couple on the polymer chain. De Paoli

et al assembled first DSSC by using of co-polymer of poly (epichlorohydrin-co-ethylene oxide) and poly (o-methoxyaniline) with NaI/I₂ based electrolyte [8]. A quasi solid state DSSC, with PEO/P(VDF-HFP) complex polymer using acetamide with SiO₂ nanoparticles, KI/I₂ was made-up by Cui et al revealed photovoltaic conversion efficiency (PCE) of 6.04 % [9]. So far witnessed efficiencies in the literature, reports are reasonably small for DSSCs made-up of solid polymer electrolytes (SPE) because of its unfavorable ionic conductivity in the polymer system, poor crystallinity could result in the drop of PCE. To reduce such limitations, the GPEs are used in DSSC, demonstrated higher ionic conductivity with excellent stability when compared to both the solid and liquid electrolytes. Hence a significant report focused to improve the efficiency of DSSC with GPEs [10, 11]. Usually, GPEs are synthesized by adding a plasticizer in to polymer medium [12]. A recent report by Pavithra et al revealed the PCE of 9.01 % by GPEs consisting with Poly (ethylene oxide)-acetamide composite [13]. Chen et al. achieved 9.5% efficiency using of Poly (acrylonitrile-co-vinylacetate) (PAN-VA) and TiO₂ nano filler [14].

In the present work, GPE is synthesized to support ion mobility in the well viscous matrix. PEO is preferred as a polymer due to its polar nature with photochemical,

thermal and chemical stability. A liquid oligomer (i.e., Poly (ethylene glycol) dimethyl ether (PEGDME)) was mixed with PEO to improve the mobility of ions and distribution of electrolyte through the mesoporous of a TiO_2 based photo electrode. Acetamide is included as a plasticizer in the polymer matrix to synchronize with KI/I_2 which would avoid the sublimation of I_2 .

2. EXPERIMENTAL:

2.1 Materials:

Poly (ethylene oxide) (PEO, $M_w = 10^6 \text{ g/mol}$), Poly (ethylene glycol) dimethyl ether (PEGDME, $M_w = 250 \text{ g/mol}$), Acetamide, LiI , Iodide (I_2), N719 and FTO coated glass from Sigma-Aldrich, acetonitrile (Merck), Titanium dioxide (TiO_2 , Dyesol), platinum solution (Solaronix) were purchased.

2.2 Preparation of Polymer Gel Electrolytes:

GPEs were synthesized by adding 0.5 g of PEO in a blend of acetonitrile and Poly (ethylene glycol) dimethyl ether (PEGDME) under nonstop stirring for two hours. To this mixture about 0.2 g of KI , 0.08 g of I_2 and different wt% of acetamide (0%, 5%, 10%, 20%) were mixed and stirring was continued for an extra 2 hours. The prepared electrolyte solution was kept stirring during the night. Subsequently, such homogeneous solution was heated at 80°C to evaporate acetonitrile and to yield gel polymer electrolyte. For all GPEs, the concentration ratio was maintained at 0.4 for the iodine salt and iodide [KI/I_2]. A series of polymer electrolytes (A: $\text{PEO} + \text{KI} + \text{I}_2$, B: $\text{PEO} + \text{PEGDME} + \text{KI} + \text{I}_2$, C: $\text{PEO} + \text{PEGDME} + 5 \text{ wt\% Acetamide} + \text{KI} + \text{I}_2$, D: $\text{PEO} + \text{PEGDME} + 10 \text{ wt\% Acetamide} + \text{KI} + \text{I}_2$, E: $\text{PEO} + \text{PEGDME} + 20 \text{ wt\% Acetamide} + \text{KI} + \text{I}_2$) were prepared.

2.3 Dye sensitized solar cells fabrication:

DSSCs were fabricated following the procedures identified in the literature [15]. Briefly, as mentioned below:

2.3.1 Preparation of photo electrodes:

Fluorine doped SnO_2 (FTO) coated glass substrates are washed by a soft detergent solution followed by 2-propanol, acetone and deionized water in an ultrasonic bath. A compact TiO_2 blocking layer was deposited onto the surface of cleaned FTO by treat with 40 mM TiCl_4 solution at 70°C for 30 min, cleaned with deionized water. Nanocrystalline titanium dioxide (TiO_2 , Dyesol) transparent layer was covered onto TiCl_4 treated FTO glass substrate, repeated the process to attain 12 μm thick TiO_2 film.

Further, 4 μm thick film of scatter TiO_2 (Dyesol) is coated, sintered at 500°C for 30 min. TiCl_4 treatment was performed at 70°C for 30 min to enhance the surface area of TiO_2 . These electrodes are dipped in the N719 dye solution (0.3 mM) for 18 hr under dark condition. The obtained photo electrodes were rinsed twice by absolute ethanol to eliminate any unanchored dye molecules, finally dehydrated under nitrogen purge.

2.3.2 Preparation of counter electrodes:

The counter electrodes were prepared by a spin coat method. Briefly, cleaned FTO substrates were located on the substrate holder of spin coater and a 40 μl of platinum solution (Solaronix) was dispensed and rotated of 5000 RPM for 30 sec, followed by sinter at 500°C for 15 min.

2.3.3 DSSC Assembling:

Test cells were fabricated by use of 60 μm thickness polymer film, which was placed between the counter electrodes and photo electrodes, and the developed polymer gel electrolytes were filled in between. The fabricated DSSCs have an active area of 0.16 cm^2 .

2.4 Characterizations:

The structural properties of GPE were done by Philips: PW1830 X-ray diffractometer. FTIR spectra of the GPEs were measured by BRUKER FT-IR Spectrometer. Surface morphology was obtained by Hitachi Model S-3400N SEM instrument. DSC thermograms were studied at the heating rate of $10^\circ\text{C min}^{-1}$ with help of a Mettler-Toledo DSC1 instrument. The ionic conductivity of the GPEs was studied by Broadband dielectric spectroscopy (BDS). Voltage - Current density (V-J) characteristics were done with the PEC-L01 solar simulator (Pacell Inc.) connected to source meter (2401, Keithley). The simulator was calibrated with standard Si cell to get 100 mW/cm^2 output powers (1 Sun condition). Broadband dielectric spectroscopy (BDS) studies were conducted by use of NOVOCONTROL in the frequency range of 1 Hz to 1 MHz.

3. RESULTS AND DISCUSSION:

In order to identify modifications in polymer chain, GPEs were analyzed by FTIR, DSC, Xrd and SEM results and in addition how ionic conductivity change by addition of acetamide was explained. Finally, how these factors were affected on ionic conductivity of GPE was verified and the obtained results were compared.

3.1 Fourier transforms infrared spectroscopy:

FTIR is helpful to offer information on the structure of polymer chain and the existence of interactions among the mechanism in PEO type gel polymer electrolyte. FTIR spectra of the GPEs are shown in Figure 1. The stretching vibrations of $-\text{CH}_2$ in PEO occurs at 2871 cm^{-1} due to the communication of K^+ ion and acetamide with a polymer in GPE and intensity of this peak decreases. These changes of $-\text{CH}_2$ confirms the coordination between PEO and acetamide. In addition, a peak obtained due to C-O-C stretching vibrations at 1106 cm^{-1} in PEO chain [16]. For GPEs with acetamide, this peak gets broad and the intensity also reduced by adding of acetamide content up to 5 wt%. At the similar time, the peak obtained at 1340 cm^{-1} due to the characteristic of the crystallized PEO gets sharpened and the intensity also reduced by adding of acetamide content up to 5 wt%. Acetamide has two important groups: one carbonyl group present in the nitrogen atom and another one in the oxygen atom. The C=O group in carbonyl can synchronize simply by the polymer matrix in the GPE through potassium ion while the polar $-\text{NH}_2$ group have a single couple of electrons will cooperate through the I_3^- ions in the redox couple. The absorption peaks of acetamide were observed due to the presence of $-\text{NH}_2$ and $-\text{C}=\text{O}$ groups at 1659 cm^{-1} and 3460 cm^{-1} respectively.

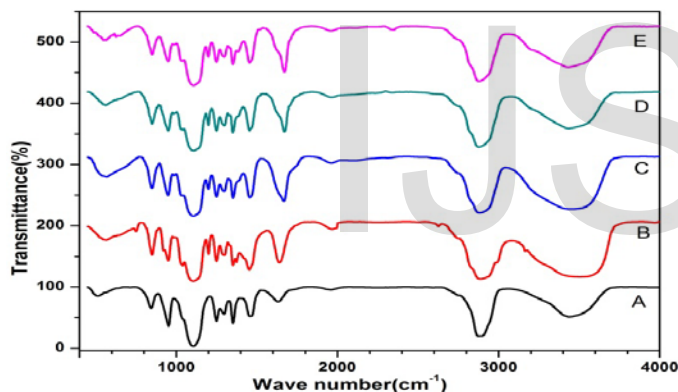


Figure 1: FTIR spectra of GPEs (A, B, C, D and E) shows different wt% of acetamide in PEO/PEGDME)

3.2 Differential Scanning Calorimetry:

Differential scanning calorimetry (DSC) is broadly applied to examine the phase modulation of the gel polymer electrolytes. The DSC thermograms are shown in Figure 2 containing a different wt % of acetamide in GPE. The flexibility of the polymer can be explained by one of the important parameter know as glass transition temperature (T_g). The T_g values of different wt % of acetamide in GPE are given in the Table-1. By addition of acetamide, T_g decreases up to PEO/PEGDME with 5 wt% of acetamide. This shows that the free volume and amorphous nature of the GPE increases by adding of acetamide, as result ion mobility is improved which indicates that the ionic

conductivity of GPEs also increases [17, 18]. Further, adding of acetamide more than 5 wt% provides reduces in T_g which shows that large amount of acetamide can reduce the free volume and flexibility of the electrolyte. Therefore, such changes in T_g may influence the activation energy and ionic conductivity [13].

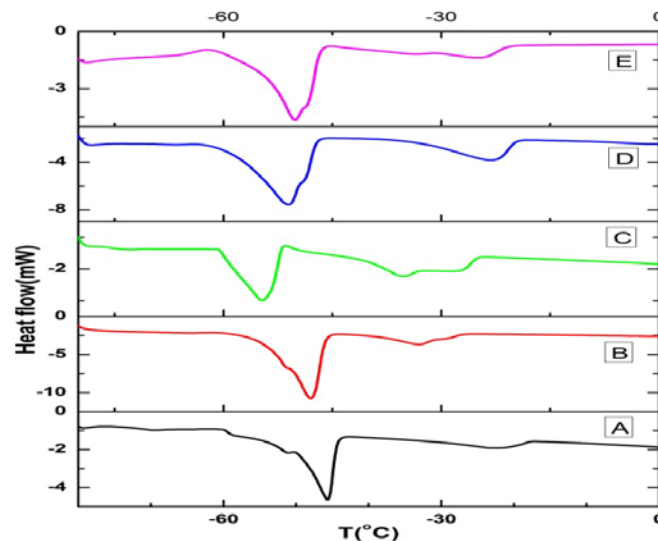


Figure 2: DSC thermograms of GPEs (A, B, C, D and E) shows different wt% of acetamide in PEO/PEGDME) measured at $-80\text{ }^{\circ}\text{C}$ to $0\text{ }^{\circ}\text{C}$

Table-1: Gel polymer electrolyte parameters.

Electrolyte	Glass transition temperature T_g $^{\circ}\text{C}$	Conductivity σ mS/cm	Activation Energy(ev)
A	-45.62	1.335	0.1292
B	-47.93	2.751	0.0688
C	-54.57	2.764	0.0659
D	-52.16	2.302	0.0690
E	-50.11	1.973	0.0724

3.3 Structural properties:

The X-ray diffraction studies provide probable influence on PEO of semi crystalline phase by adding acetamide content. X-ray diffraction pattern of GPEs with various wt% of acetamide are shown in Figure 3. Pure PEO is a semi crystalline polymer and exhibits two strong crystalline peaks at 19.1° and 23.2° [19]. The XRD pattern shows only one broad peak appeared at 22.39° which indicates the variation in the crystalline phase of gel polymer electrolytes. The diffraction peaks become broad up to 5 wt% acetamide content is inserted in the polymer complexes. Hence, it can suggest that the PEO undergoes significant structural reorganization while blending and thus complexation occurs in the amorphous phase. It has been reported that ion mobility is better in case of

amorphous phase because ion mobility is helped by polymer segmental motion [20]. Among all GPEs, acetamide with 5 wt% shows wide and less intensity curve, which exhibits high amorphous nature compare to others.

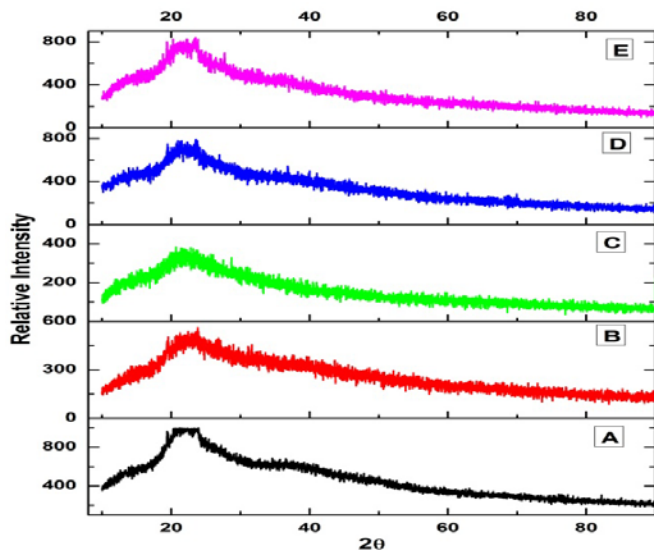


Figure 3: X-ray diffraction pattern of GPEs (A, B, C, D and E shows different wt% of acetamide in PEO/PEGDME)

3.4 Scanning electron microscopy:

SEM image of GPEs with different wt% of acetamide are presented in the Figure 4. SEM images of GPE (Fig. 4a and d) show a cracked fibrous structure but GPE containing 5 wt% of acetamide (Fig. 4c and e) show a fibrous unbreakable structure due to the interrelation between acetamide and the ether oxygen in PEO and also images revealed that the redox couple bound inside the walls of the GPE establishes a homogeneous allotment. SEM images (c and e) show small spherical particles in GPE by adding of acetamide. Therefore, incorporation of acetamide provides considerable modifications of the surface morphology as well as increase in the free volume of the GPE, which makes growth in the ionic conductivity.

3.5. Ionic conductivity of Gel polymer electrolytes:

In DSSC, the enhancement of ionic conductivity as well as motion of the redox couple of the GPE provides higher power conversion efficiency [21]. Figure 5 shows complex impedance plots (Z' vs Z'') of GPEs with different wt% of acetamide. Bulk resistance (R_b) was measured from complex impedance plots by extrapolated intercepts on the Z' of the common part of two arcs. Therefore, the ionic conductivity of the prepared GPEs is measured by bulk resistance (R_b) values obtained from Broadband dielectric spectroscopy (BDS) by applying the formula:

$$\sigma = \frac{l}{AR_b} \quad (1)$$

Where ' l ' is the space between two electrodes, ' R_b ' is the bulk resistance and ' A ' is the area measured by BDS. From Table-1 it is resolved that the ionic conductivity of GPE maximum for PEO/PEGDME with 5 wt% acetamide. It is broadly speaking that ionic conductivity is large in the amorphous phase of GPE [22]. Ionic conductivity is improved by the addition of acetamide; this is due to the decrease of crystallinity of the GPE and due to the interaction of acetamide with PEO. Further addition of acetamide more than 5 wt% makes a decrement in ionic conductivity which indicates a large amount of acetamide can decrease the free volume and flexibility of the electrolyte. The above information is better agreement with the resolutions provided in FTIR, DSC and SEM measurements.

The temperature variation of ionic conductivity of the GPEs with different wt% of acetamide in the range of 303–373 K. is shown in Figure 6. The free volume and flexibility in the GPE increase with an increase of temperature and hence improves the mobility of ion in the polymer medium [23]. The ionic conductivity significantly improves at the melting point of the PEO (60 °C) which shows that the GPE exhibits high amorphous nature. The ionic conductivity (σ) modifications of all GPES exhibit Arrhenius-type thermal activated process which is fitted into the Arrhenius equation:

$$\sigma = \sigma_0 \exp\left[-\frac{E_a}{KT}\right] \quad (2)$$

Where σ_0 is a pre-exponential factor, E_a is activation energy for ionic mobility, K is Boltzmann constant and T is the temperature in Kelvin scale. The activation energy (E_a) values were evaluated from the Arrhenius equation and tabulated in Table-1 for all GPEs. The results clearly illustrate that the inclusion of acetamide to the GPE may reduce the activation energy of ions. In general, lower E_a is accountable for large ionic conductivity when compared with pure PEO type GPE. This is mostly due to the increase of amorphous nature in case of polymer system, which is helpful for mobility of ions [24].

3.6 Photovoltaic performance of the DSSCs:

DSSCs are fabricated to determine the performance of prepared gel polymer electrolyte potentiality in a practical application. The photovoltaic performance of the DSSC by means of gel polymer electrolytes with various %wt of acetamide are shown in Figure 7. and the results are provided in the Table 2. The performance parameters of all DSSC such as open circuit voltage V_{oc} (V), short-circuit current density J_{sc} (mA/ cm²), fill factor (FF) and power

conversion efficiency $\eta\%$ were obtained with different gel polymer electrolytes.

The fill factor (FF) was calculated using:

$$FF = \frac{V_{max} \times J_{max}}{V_{oc} \times J_{sc}} \quad (3)$$

Where V_{max} and J_{max} are the voltage and current density at the maximum power output point.

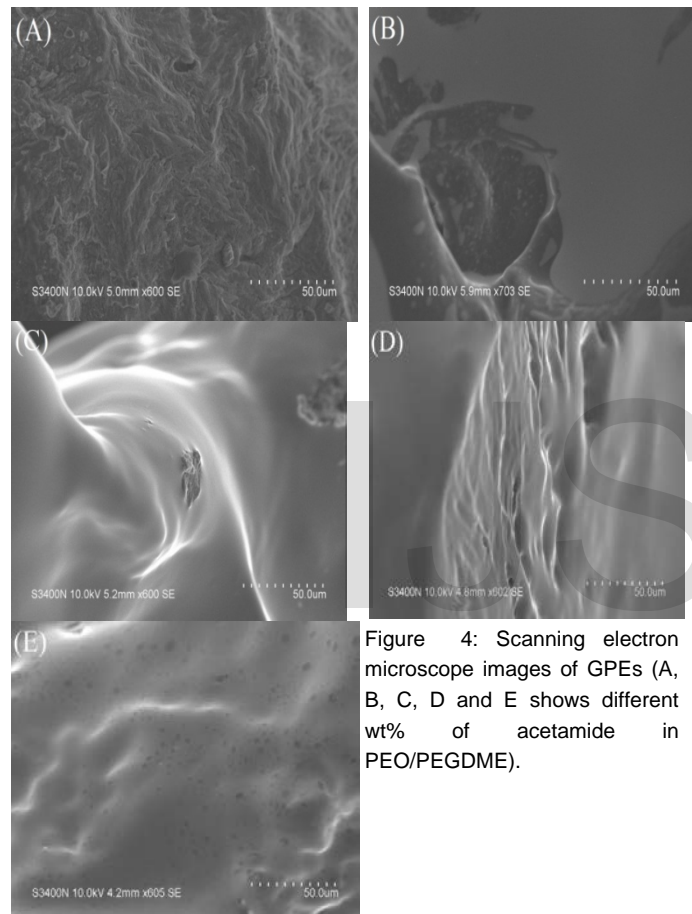


Figure 4: Scanning electron microscope images of GPEs (A, B, C, D and E shows different wt% of acetamide in PEO/PEGDME).

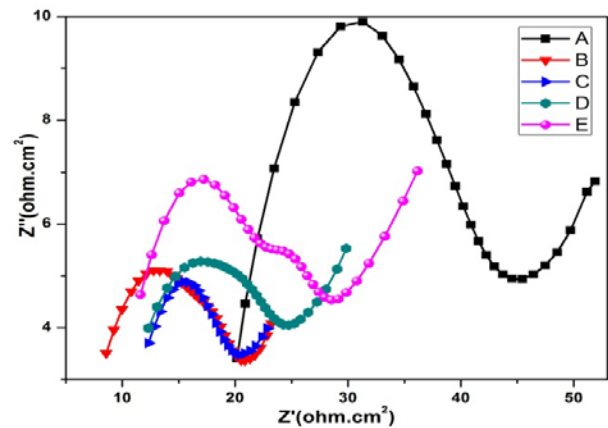


Figure 5: impedance spectra of GPEs (A, B, C, D and E shows different wt% of acetamide in PEO/PEGDME)

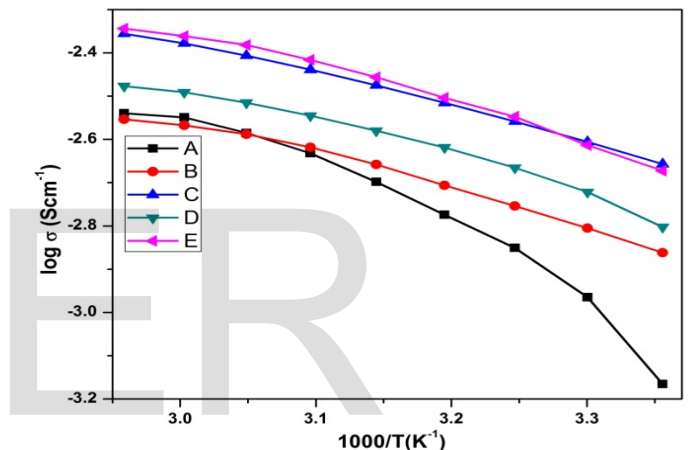


Figure 6. Temperature variation of ionic conductivity of GPEs (A, B, C, D and E shows different wt% of acetamide in PEO/PEGDME)

The power conversion efficiency η was calculated :

$$\eta\% = \frac{V_{oc} \times J_{sc} \times FF}{\text{Total incident light power density}} \times 100 \quad (4)$$

Figure 7 Reveals that the Voltage - Current density characteristics (V-J) of DSSCs with platinum electrodes under simulated solar irradiation of 100 mW/cm² (AM 1.5). The photo-current conversion efficiency of Cell-C exhibits superior in performance of $\eta = 6.14\%$, $V_{oc} = 0.73$ V, $J_{sc} = 13.88$ mA/cm² and $FF = 0.60$, which shows the GPE with 5% acetamide having good potentiality than the other electrolytes, reflects in the enhanced DSSC performance. However, for higher wt% of acetamide, the photo-current conversion efficiency values decrease due to the interaction between acetamide and I_3^- which will decrease the ionic conductivity (σ). Consequently, photo-current conversion efficiency reduces for 10% and 20%wt of acetamide. Considering the favorable high conductivity of GPE with

5% acetamide, the platinum CE can exhibit the improved efficiency. A systematic study is conducted to assessment the acetamide and PEGDME loads in gel electrolytes and shown in Fig. 7. The conversion efficiencies of DSSCs with different acetamide concentrations are 5.11 %, 6.04 %, 6.14 %, 5.91 % and 5.54 % for Cell-A, Cell-B, Cell-C, Cell-D and Cell-E respectively and the other photovoltaic parameters are evaluated from the V-J characteristic, are provided in Table-2.

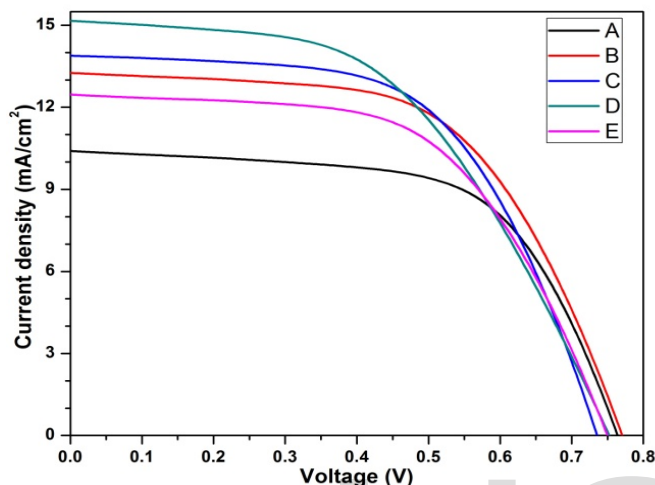


Figure7: Voltage - Current density characteristics of DSSCs under 1 sun illumination.

Table-2. Photovoltaic cell parameters of fabricated test cells.

Parameter	A	B	C	D	E
V_{oc} (V)	0.77	0.77	0.73	0.75	0.76
J_{sc} (mA/cm ²)	10.40	13.25	13.88	15.16	12.46
Fill Factor	0.63	0.58	0.60	0.51	0.58
Efficiency η (%)	5.11	6.04	6.14	5.91	5.54

4. CONCLUSIONS:

In this study, the effect of acetamide in PEO gel polymer electrolyte on the performance of DSSCs was verified. The decrement of crystallinity on the GPE by the addition of acetamide was studied by FTIR spectroscopy and the deviation of glass transition temperature (T_g) on the GPE by the incorporation of acetamide was investigated by DSSC. Ionic Conductivity and temprature dependent of conductivity were investigated by Broadband dielectric spectroscopy. The ionic conductivity high for PEO/PEGDME with 5 wt% of acetamide 2.764×10^{-3} S/cm and less for the PEO without acetamide 1.335×10^{-3} S/cm. Finally, photovoltaic performance of DSSC was analyzed by implementation of GPEs in the dye-sensitized solar cells.

The GPE with PEO/PEGDME and 5 wt% acetamide exhibits a high photovoltaic conversion efficiency of 6.14% when compared with 5.11% for PEO with 0 %wt acetamide under 100 mW/cm² illuminations. However, more amount of acetamide above 5 wt% decreases photovoltaic performance of the DSSCs due to the bad interaction between acetamide and I_3^- which will decrease the ionic conductivity (σ) and mobility of ions. As a result, photo-current conversion efficiency reduces for 10 wt% and 20wt% of acetamide when compare to 5wt% of acetamide.

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