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Electrical properties enhancement of Liquid and Polymer Gel based electrolytes used for DSSC applications

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Abstract

Electrolytes have been considered a major component of DSSC and play a vital role in determining the ionic conductivity and efficiency of the cell. Emphasis was laid on the confirming the conductivity upon fabrication of an electrolyte using in situ gelation process from a newflanged combination of Triiodide (KI/ I2/ Glacial acetic acid /distilled water) and gel-based polymer electrolyte (Gelator: PMMA/THF/PC/EC) in the ratio 8:2. The electrolyte portion so framed a photo conversion efficiency of 11.32% and a fill factor of 0.439. The conductivity of the sample characterized by Scanning Electron Microscopy showed that the uniform tracks confirmed extreme ionic conductivity of the blend electrolyte which showed dependance on the layered movement of PMMA-co-THF-KI2/PC/EC based electrolyte system. Energy Dispersive x-ray Analysis (EDX) reports engraigned the percentage weight proportions of conductive elements (C & O with a wt% of 65.48 and 30.18 in one spectrum and K & I with a wt% of 44.7 & 35.25 in another spectrum respectively). FTIR test analysis was performed to identify the functional groups of the PGE which identified the ionic conductivity of the sample, shown by the intensity of peak absorbance in the range 400–4000 cm\(^{-1}\). Further it was observed, the conductivity of the different concentrations of the liquid and gelator solution demonstrated an increase in exhibiting ionic conductivity and the same was depicted by the morphological studies that featured dark pores of the sample which were spread consistently indicating the amorphous nature of the material (at room temperature).

1. Introduction

Polymer electrolytes have demonstrated advantages over liquid electrolytes including high tensile strength, better safety, ease of shape based fabrication, high processing ability, intact interfacing properties between electrodes, high flexibility and no spillage (Bharati et al 2020). Recent research has showed that electrolytes based on polymer components such as PEO (poly (ethylene oxide)), PAN (poly acrylonitrile), PU (poly- urethane), PVDF (poly (vinylidene fluoride), PVC (poly (vinyl chloride)) etc have led to improved stability and enhanced efficiency of the DSSC (Abisharani et al 2020). One such PEO base polymer component, i.e. PMMA has been used here to fabricate the Polymer Gel Electrolyte (PGE) using various ratio proportions with liquid electrolytes, resulting in an enhanced efficiency of 11.32% as compared to the already existing electrolytes based on same PEO polymer compound. This new flanged combination of liquid electrolyte and gelator was prepared for greater efficiencies as it is capable to produce increased ionic conductivities with the help of PEO based gelator composed of PMMA as polymer along with PC/EC/THF combination as plasticizer salt complexes and also consists iodide ions for improved conduction (Gohel et al 2020). Further, the use of protic ionic liquids (PIL) in acidic medium such as glacial acetic acid, which has also been used as a part of composition of liquid electrolyte in this work, have depicted higher ionic conductivity (Nair et al 2020). Polymer electrolytes also have an additive advantage of more appealing features as compared to liquid electrolytes like good fluidity and excellent dielectric
constants which is due to their properties of dissolving high salt concentration and greater ion transfer rate (Janakiraman et al 2019).

Keeping in consideration the above-mentioned effective parameters of polymer electrolytes, many researches have been made for increasing ionic conductivity. Use of fillers and plasticizers have reported to have increased values of conductivity (Banitaba et al 2019). Therefore, effort was made to increase ionic conductivity and hence efficiency of the electrolyte by adding plasticizers like ethylene carbonate (EC) and propylene carbonate (PC) in the amorphous regions of the electrolyte for an effective solution. Classic polymer electrolytes were plasticized gel ionic rubber polymers and ion-conducting electrolytes. Commonly available polymer electrolytes that can be used with these plasticizers are polymethyl methacrylate, polyethylene glycol, polymethyl ether methacrylate, polyacrylonitrile, polyvinylidene fluoride-co-hexafluoropropylene (Tsai et al 2013, Sachdeva et al 2018).

Also, as suggested by Vani et al the ionic conductivity according to the nature of their surface, the functional group and size increases with the addition of fillers in the compound (Vani et al 2019). Moreover, for improvement in carrier ion mobility, a hybrid ionic liquid polymer electrolyte can be used since by band bending, it reduces the barrier height hence leading to an increase in concentration of carrier ions (Lv et al 2019). Hence, a PGE composed of both polymer electrolyte and liquid electrolyte can be said to have enhanced conductivity.

The electrolyte must be capable of continuously renewing the dye used in fabrication of a DSSC and its own properties as well (Ammar et al 2019). The efficiency of DSSC depends on various parameters mainly photo-current density, photo-voltage, and fill factor (FF) (Teo and Arof 2018, Zheng et al 2020). Considerable research work is done towards creating polymer electrolytes by using different polymers and salts (Bella et al 2013, Dong 2020). In this study, analysis of the different parameters that may be implied to enhance the electron emission capabilities of an electrolyte was done and the performance parameters like efficiency and fill factor were observed after the formation of a PGE formed using in situ gelation (Sachdeva et al 2018, Si et al 2020).

2. Experimental details: in situ gelation (solvent evaporation)

The solar cell performance widely gets effected by the many detrimental parameters in its life cycle, unavoidable environmental parameters cannot be deterred, hence mainly electrical parameters such as the voltage at open and short-circuited conditions of the cell, fill factor, efficiency, short circuit current density, etc can help in enhancing the efficiency of the cell. A DSSC is composed of many layers and each layer plays a significant role for effectively contributing to the output of the cell. The middle layer and the heart of the cell is the electrolyte. By selecting an appropriate electrolyte, cells efficiency and hence its performance parameters can be improved and can also lead to further increase in its lifespan. Keeping these constraints in mind, the following objectives have been framed for the research done:

a. Fabrication of the polymer gel electrolyte samples (A, B, C & D) by in situ gelation by using different portion combinations of liquid electrolyte and gelator.

b. Mathematical modeling for calculating the performance parameters (efficiency and fill factor) of each DSSC framed by PGE samples A, B, C & D and performance comparison of all the fabricated cell samples.

c. Characterization testing by SEM, EDAX & FTIR of the best sample selected (A) based on results of performance parameters from mathematical models framed from PGE samples.

For creating the advantages of PGE, the copolymer obtained was studied for long-term stability as suggested by Jørgensen et al (Jørgensen et al 2008). Basically, the aim of the research was finding the stable nature of the cell and it was observed that Polymer Gel Electrolyte came out to be versatile for different solvents that showed high efficiency and long-term durability (Shen et al 2014). Polymer gel electrolyte (PGE), a liquid electrolyte (LE) entombed by a small amount of polymer mesh, was characterized by properties between those of a liquid and solid electrolyte (Sharma et al 2009). Features of these PGEs were observed to be ionic conductivity and different amorphous stages were obtained during its making. Drawbacks of liquid electrolytes like leakage and flammability could be allayed by using PGs (Cho et al 2019). However, it became challenging to integrate them into the spectra of electrode voids (Yue et al 2011). The liquid state polymer electrolyte was prepared and converted to gel state by In Situ Gelation i.e. by simply driving away from a portion of solvents in the electrolyte at elevated temperatures (Gu et al 2017). In this research, the focus was laid on PGEs which was gelated by in situ process. For this method, the liquid polymer electrolyte solution was prepared and then gelated with the polymer electrolyte so as to obtain the PGE. The steps of fabrication of PGE have been enlisted in flowchart 1.
The evaporation of the solvents from Step 3 in flowchart 1 ensured the absorption of electrolytes in the TiO$_2$ pores coated on the cathode hence lead to improvement of contact and surface interface properties (Wang et al 2011).

For establishing the advantages of the PGE, the copolymer that was obtained after the gelation process was studied with different flanged liquid electrolyte ratio combinations. The process as shown in the flowchart 1 above depicted the preparation techniques of LE and gelator and the PGE prepared was analysed for performance parameters. The sample which gave best results was chosen for further characterization and application for the fabrication of DSSC. The experimental details of the fabrication of PGE are shown in figure 1.

The liquid state polymer electrolyte solution was obtained by mixing 2:8, 3:7, 4:6, 1:1 (50% each) ratio of gelator solution(gelator) with liquid electrolytes(triiodide) respectively and four samples were prepared. The efficiency of each sample was calculated and it was observed that a liquid state polymer electrolyte with a 2:8 ratio gave the best efficiency, hence was selected for further testing and performance analysis of DSSC. The high viscous state of the liquid polymer electrolyte was then obtained by heating the sample in a hot air oven at 70 °C for 20 min to drive away salts from the portion of the THF solvent (Sachdeva et al 2018).
Formation of polymer gel electrolyte: Polymer gel electrolyte was formed by varying the ratio of liquid electrolyte and gelator solution (Khamidy et al 2017). Four types of electrolytes were formed by varying ratios as shown in table 1.

### Table 1. PGE (Polymer Gel Electrolyte) samples.

| Gel-based electrolyte | Ratio of LE | The ratio of PGE gelator |
|-----------------------|-------------|--------------------------|
| Electrolyte Sample A  | 8           | 2                        |
| Electrolyte Sample B  | 7           | 3                        |
| Electrolyte Sample C  | 6           | 4                        |
| Electrolyte Sample D  | 1           | 1                        |

2.1. Selection of Electrolyte sample for testing and further use in DSSC
Addition of I$_2$ in the electrolyte led to an increase in its ionic conductivity and enhanced its power conductivity (by approximately 4%). Further, it also boosted the stability of the cell (Soni et al 1940, Kesavan et al 2018). Similar enhancements in the electrical properties were observed in the results of DSSC parameters that used Electrolyte Sample A (8:2 ratio of liquid electrolyte and polymer electrolyte). The electrical parameters were obtained through mathematical modelling of DSSC using sample A as the electrolyte (Singh et al 2017).

2.1.1. Mathematical modeling of DSSC (using electrolyte sample A)
A mathematical model was framed as shown in figure 2(a) by observing the readings of the various parameters of the DSSC from figure 2(b) under open circuit and short circuit conditions and the efficiency was hence calculated (Shanian and Savadogo 2006).

The various parameters of DSSC hence fabricated were observed from the circuit as

Short circuit current density can be calculated as (Bella et al 2013)

\[
J_{sc} = \frac{I_{sc}}{A} \tag{1.1}
\]

Here, \(A = \) area of the cell
Current density at load,
\[ J_m = \frac{I_m}{A} \]  
(1.2)

Fill Factor,
\[ FF = \frac{V_m \times I_m}{Voc \times Isc} \]  
(1.3)

Where, \( V_m \) = Voltage at load and \( I_m \) = Current at load

Therefore, Energy conversion efficiency,
\[ \eta = \frac{Voc \times Isc \times FF}{Pin(mWATT \ cm^{-2})} \times 100 \]  
(1.4)

\( P_{in} \) = Input power (observed through the Lux meter)

Hence, the efficiency obtained by mathematical modelling to the DCCS circuit under open circuit and short circuit conditions, using Electrolyte Sample A as an electrolyte was 11.32%. Apart from the mathematical analysis of the sample A electrolyte cell, characterization of the sample was also done to affirm the conducting properties of the electrolyte.

The results obtained after the mathematical modelling were compared to the results of performance parameters (\( \eta \), fill factor (FF), open circuit photovoltage (Voc) & short circuit current density (Jsc)) of various
Table 2. Comparison of Performance Parameters of various electrolytes with the Sample A, PGE.

| Performance-Parameter | Name of Electrolyte (from literature) | PGE- Fabricated (Experimentally) |
|-----------------------|--------------------------------------|----------------------------------|
|                       | E1 (Lee et al 2010)                  |                                  |
|                       | E2 (de Freitas et al 2010)           |                                  |
|                       | E3 (Fenton 1973)                     |                                  |
|                       | E4 (Ahn et al 2012)                  |                                  |
|                       | E5 (Andualem and Demiss 2018)        |                                  |
|                       | E6 (Wu et al 2015)                   |                                  |
|                       | E7 (Pablo et al 2016)                |                                  |
|                       | E8 (Zhang et al 2008)                |                                  |
|                       | E9 (Bella and Bongiovanni 2013)      |                                  |
|                       | E10 (Ngai et al 2016)                |                                  |
|                       | E11 (Zhang et al 2008)               |                                  |
|                       | E12 (Yang et al 2016)                |                                  |
|                       | E13 (PMMA-co-THF-KI2/PC/EC)          |                                  |

η(%) | 9.61 | 9.2  | 8.31 | 8    | 7.5  | 6.82 | 6.7  | 6.55 | 5.81 | 5.3  | 5.45 | —    | 5.25 | 5.18 | 4.83 | 11.32 |

V_{OC}(V) | 0.7 | 0.89 | 0.75 | 0.75 | 0.77 | 0.72 | 0.74 | 0.76 | 0.73 | 0.66 | 0.72 | 0.75 | 0.70 | 0.75 | 0.67 | 0.386 |

J_{SC} (mA cm\(^{-2}\)) | 19.68 | 12.9 | 17.35 | 15.3 | 15.3 | 14.62 | 13.1 | 13.00 | 12.20 | 11.69 | 11.71 | 10.03 | 11.73 | 10.03 | 11.19 | 24.91 |

Here, in table 2.

E1: N-Phthaloylchitosan + polyethylene oxide + tetra propylammonium iodide + dimethylformamide + tetra propylammonium iodide ethylene carbonate + BMII.
E2: Polyethyleneimine + polyethylene glycol + KI + I\(_2\) + 4,4’-(((oxybis(ethane-2,1-diyl)) bis(oxy)) bis(ethane-2,1diyl)) bis(sulfanediyl)) dipyridine.
E3: Polymer gel electrolyte + propylene carbonate.
E4: Polymethyl Methacrylate + 1-butyl-3-methylimidazoliumiodide/\(_2\).
E5: Polystyrene beads + 1-Butyl-3-methylimidazoliumiodide + \(_2\).
E6: Electrospun polyvinylidene fluoride-co-hexafluoropropylene (ePVDF-co-HF) + \(_2\) + Tetraethylammoniumiodide + PMII + PC or EC + CN bipheryl.
E7: Polynvinylidene fluoride- hexafluoropropyle + PMII + \(_2\) + NMBI in MPN.
E8: Polyacrylonitrile: polyethylene glycol/ carbon ethylene carbonate + 1-N-butyl-3-hexyl imidazolium iodide + \(_2\).
E9: PEGDME + BMII + Polyaniline loaded carbon black.
E10: PVDF-HF + MPII.
E11: Agarose + 1-allyl-3 methylimidazolium iodide propylene carbonate guanidinium thiocynate + N-methylbenzimidazole/\(_2\).
E12: Carboxymethyl cellulose sodium salt(cmcs) + polyethylene oxide.
E13: Agarose + MPII.
E14: Carboxymethyl cellulose/polyethylene oxide + sodium iodide + MPII + TBP + \(_2\).
E15: 1-ethyl-3-methylimidazolium bis trifluoromethylsulphonyl imindle + EMIIm-I + lithium iodide + \(_2\) + Tert-butylpyridine + carbon black composite.
3. Characteristics testing of DSSC based on electrolyte

3.1. Sample A (8:2 LE & PGE)
It was desired that the absorption and homogeneity tests of the samples should be performed to affirm the photoconduction & absorption properties of the electrolyte hence obtained (Singh et al. 2015). FESEM equipped with Energy Dispersive Spectroscopy (EDAX) gave quantification of the mapping elements of the sample (Sardar et al. 2017). These helped in determining the penetration and extent of absorption of the sample prepared (Chiku et al. 2011).

4. FESEM test characteristics (gold coated)

Based on the morphology used, FESEM told about the porous nature and some other typical properties of the specimen as shown (Chiku et al. 2011, Chiappara et al. 2016).

A scanning electron microscope (SEM) gave microstructure morphology of the conducting surface. The SEM images of the electrolyte film are shown in figure 5.

- Figure 3(a) gave a photograph of PMMA- co- THF- KI₂ salt complex with pores which supported electrolytes increased ionic conductivity.
- Figure 3(b) showed the micrograph having spherical grains homogeneously dispersed in the structure. It was observed that the sample membrane depicted a porous surface due to the presence of pores and dark sections, with a size 1–10 μm which gave increased conductivity value to the sample.
- Figure 3(c) depicted the even surface morphology of PMMA - KI₂ blend. It was told about the confirmation of the complete amorphous nature of PMMA polymer and the complete dissolution of an iodide salt.
- The appearance of uniform tracks of a few micrometre sizes in figures 3(d) and (e) corresponded to enhanced conductivity. This showed that the extreme ionic conductivity of the blend electrolyte depended on the layered movement of PMMA- co- THF- KI₂/PC/EC based electrolyte system.
- The pores in figure 3(f) became responsible for capturing huge volumes of the plasticizer (EC) + salt (KI₂). These sizes were of the range 50–100 nm. The smooth surface told that the polymers and salts used in this study had a good compatible nature and light grey regions in the figures indicated the presence of assistive ionic motion component rich in plasticizer medium (Chiappara et al. 2016, Sachdeva et al. 2018).

4.1. EDAX characteristics
Energy-dispersive x-ray spectroscopy, also known as energy dispersive x-ray analysis, an analytical procedure used for the elemental analysis or chemical characterization of a sample was carried out for the prepared sample.
FTIR was performed to identify the functional groups of the PGE Sample A which may be crucial for incorporating plasticizer EC bonding and presence of functional groups in the electrolyte. It helps in monitoring energy of vibration of Fourier Transform Infrared Spectral analysis is a very helpful analysis to identify the nature of ionic bonding and presence of functional groups in the electrolyte. It helps in monitoring energy of vibration of molecules. The FTIR spectra of the blend electrolyte of PGE sample A is shown in figure 5 which has an incorporation of plasticizer EC (ethylene carbonate) with the filler Triiodide.

4.2. FTIR test analysis
Fourier Transform Infrared Spectral (FTIR) analysis is a very helpful analysis to identify the nature of ionic bonding and presence of functional groups in the electrolyte. It helps in monitoring energy of vibration of molecules. The FTIR spectra of the blend electrolyte of PGE sample A is shown in figure 5 which has an incorporation of plasticizer EC (ethylene carbonate) with the filler Triiodide.

- FTIR was performed to identify the functional groups of the PGE Sample A which may be crucial for identifying the ionic conductivity of the sample, shown by the intensity of peak absorbance in the range 400–4000 cm$^{-1}$.
- The existence of C–O band responsible for ionic movement during bonding is confirmed by strong absorbance band at around 1012 cm$^{-1}$.
- The strong band at 1390 cm$^{-1}$ is ascribed to CH bending vibration of PMMA.
- The band at 1254 cm$^{-1}$ is assigned to C–O–C symmetric stretching mode of vibration.
- The peak at 610 cm$^{-1}$ gives linking of CH3–C–O group.
- The amorphous phase of copolymer is confirmed at peak 884 cm$^{-1}$.
- The absorption peak at 3423 cm$^{-1}$ gives the presence of O–H group.
- The strong absorption peak appearing at 1705 cm$^{-1}$ is assigned to symmetrical starching of the C-12 group.
- The vibrational peaks between 455 cm$^{-1}$ to 610 cm$^{-1}$ are assigned to bending and wagging vibrations of –Cl2 of PC-co-THF polymer respectively.

5. Comprehensive result analysis
From figure 6, a comparison of current densities, open-circuit photovoltages and fill factor of various DSSC’s hence fabricated from the above four samples of polymer electrolytes by in situ gelation is shown graphically (Cho et al. 2014, Buraidah et al. 2017). The mathematical model of each electrolyte sample was framed as referred to in section 2.1.1 and relative electrical parameters (Wu et al. 2015, Guo et al. 2018) i.e. open circuit photovoltage, current density, fill factor and efficiency were obtained as shown in table 3.

Table 3 and figure 6 show the analysis done for all four samples, i.e. DSSC (fabricated based on electrolyte sample A) gave best efficiency (11.32%) and short circuit current density (24.91 mA cm$^{-2}$) followed by DSSC (fabricated based on electrolyte sample B) which gave an efficiency of 9.58% and short circuit current density of 19.21 mA cm$^{-2}$. This was further preceded by DSSC (fabricated based on electrolyte sample C) which gave an efficiency of 8.27% and short circuit current density of 14.27 mA cm$^{-2}$ followed by DSSC (fabricated based on electrolyte sample D) which gave an efficiency of 6.34% and short circuit current density of 12.62 mA cm$^{-2}$.

These DSSC’s were tested under both sunlight and low-intensity light and the observations & calculations gave the above results of the detrimental parameters for obtaining the efficiency of the DSSC, as was also studied by Hsu et al. (Hsu et al. 2013).
6. Conclusion

Ionic properties of polymer electrolytes were drastically improved by the process in situ gelation. The results of the samples hence prepared characterized the best ionic conductivity verified by the maximum obtainable efficiency of 11.32% as compared to earlier obtained efficiency of 9.61% by researchers. FESESM & EDAX affirmed the homogeneous mixing of various components present in the complex polymer electrolyte. The final sample selected can be used in multiple applications, primarily for the fabrication of DSSCs due to its enhanced conductivity with LE blend.

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Table 3. Electrical Parameters calculated based on Electrolyte Samples A, B, C & D.

| DSSC’s based on electrolyte samples A, B, C & D | Open circuit photovoltage, Voc (V) | Short circuit current density, Jsc (mA cm$^{-2}$) | Fill factor, FF | Efficiency (%) |
|------------------------------------------------|-----------------------------------|-----------------------------------------------|----------------|----------------|
| DSSC (A)                                        | 0.386 V                           | 24.91                                         | 0.439          | 11.32          |
| DSSC (B)                                        | 0.376 V                           | 19.21                                         | 0.492          | 9.58           |
| DSSC (C)                                        | 0.379 V                           | 14.27                                         | 0.57           | 8.27           |
| DSSC (D)                                        | 0.33 V                            | 12.62                                         | 0.567          | 6.34           |

Figure 6. Relativity of photovoltage, current density & fill factor of various DSSC’s and Relative Percentages showing the best efficiency of sample A.
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