

RESEARCH ARTICLE

N719 and N3 dyes for quasi-solid state dye sensitized solar cells - A comparative study using polyacrylonitrile and CsI based electrolytes

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Received: 30 April 2016; Accepted: 21 July 2016

Abstract: Dye sensitized solar cells offer a low cost alternative technology for solar energy harvesting. However, there are long term stability issues connected with these cells due to the liquid electrolytes normally used. Gel or solid polymer electrolytes which do not contain volatile solvents have been used in this investigation in order to alleviate these problems. Two types of solar cells were assembled using a double layered TiO₂ film sensitized with two types of dye sensitizers, namely N719 (Ruthenizer 535-bisTBA) and N3 (Ruthenizer 535) in order to compare their performance. Quasi-solid-state electrolytes based on PAN (polyacrylonitrile) as the host polymer and CsI as the iodide salt were prepared by incorporating ethylene carbonate (EC) and propylene carbonate (PC) as plasticizers. The conductivity of the electrolyte was further improved by adding tetrapropylammonium iodide (Pr₄NI), 1-methyl 3-propyl imidazolium iodide (MPII) and 4-tert-butylpyridine (4TBP). The incorporation of these additives resulted in 17% enhancement in ionic conductivity. This improved electrolyte was used to fabricate the solar cells with N3 and N719 dyes. The efficiency of the N3 dye based solar cell was 3.85% whereas the efficiency of the N719 dye based solar cell was 4.14% representing a significant efficiency enhancement by 7.53% by the N719 dye. In addition, due to additives the solar cell efficiency has enhanced by 19% compared to the solar cell without any additive.

Keywords: N719 and N3 dyes, Quasi-solid-state, dye-sensitized, solar cells.

INTRODUCTION

The demand for energy resources is ever-increasing and thus the search for eco-friendly

and sustainable energy resources have gained special attention. On the other hand, the solar energy received on Earth in one hour is sufficient to fulfill the human energy consumption in an entire year (Lewis, 2007; Bella and Bongiovanni, 2013). This clearly shows that the solar energy is an enormously large and reliable energy resource. As a result, enormous efforts have been devoted to develop highly efficient and stable solar energy conversion devices.

The high production cost of conventional high efficient inorganic silicon solar cells has hindered the wide spread utilization of solar cells (Ning *et al.*, 2010). Another novel type of solar cells that has attracted a lot of attention recently is the solar cells based on methylammonium lead tri-halide perovskites, which have reached efficiencies of about 20% (Green *et al.*, 2015). However, the light and oxygen induced degradation (Bryant *et al.*, 2016), the use of lead (Pb) as a fundamental constituent and the use of an expensive hole conducting material, (spiro-MeOTAD) (Niu *et al.*, 2015; Green *et al.*, 2014), have also hindered the realization of low cost and stable solar cells.

As a low cost alternative, dye sensitized solar cells (DSSCs) have attracted wide attention after reports by O'Regan and Grätzel in 1991 (O'Regan and Grätzel, 1991). The efficiency reported for DSSCs is 14.3% under 1.0 Sun irradiation (Kakiage *et al.*, 2015). The chemical and physical instability issues, mainly originating from the volatile liquid electrolyte used in DSSCs, are the primary disadvantages for their

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utilization as a long term, stable, outdoor applications (Bella and Bongiovanni, 2013). However, the instability issues can be resolved to a certain extent through the replacement of liquid electrolytes by gel or solid polymer electrolytes, which do not use volatile solvents (Huo et al, 2007; Bandara *et al.*, 2012). Still, the cell efficiency suffers due to resistive losses originated by poor ionic conductivity in such electrolytes. Accordingly, it is important to investigate the compatible cell ingredients for such solid or quasi solid state DSSCs.

Dye sensitized solar cells work on the basis of a sensitizing dye attached to a wide band gap semiconducting materials such as TiO₂ for light harvesting. Transition metal coordination compounds, such as N3, N719 and N749 are reported as most efficient sensitizers with iodide/tri-iodide liquid electrolyte based DSSCs (Kalyanasundaram and Grätzel, 2009; Karmakar and Ruparelia, 2011). However, better efficiencies have been reported using cobalt based electrolyte systems with mixed dyes (Kakiage et al, 2015). In the present study, N3 and N719 dyes were selected to investigate the cell performance with iodine/iodide ion conducting gel polymer electrolytes based on polyacrylonitrile (PAN).

Several gel polymer systems have been tried out in order to prepare efficient and stable quasi solid state DSSCs (Bella and Bongiovanni, 2013; Shen et al, 2008; Bandara *et al.*, 2013). In a study to determine the best single alkaline salt suitable for PAN based gel polymer electrolytes, CsI have shown higher DSSC performance (Bandara *et al.*, 2016). The present study is also focused on further improvement of the reported electrolyte by incorporating various additives. For example, tetrapropylammonium iodide

(Pr₄NI) and imidazolium iodide based ionic liquids, which have shown their potential to improve DSSC performance, were selected to be studied in this work. Subsequently, the improved electrolyte is employed in quasi solid state DSSCs in order to compare the performance with N3 (Ruthenizer 535) and N719 (Ruthenizer 535-bisTBA) dyes.

EXPERIMENTAL

CsI, tetrapropylammonium iodide (Pr₄NI), 1-methyl 3-propyl imidazolium iodide (MPII) ionic liquid, polyacrylonitrile (Mw = 150,000), iodine (I₂), ethylene carbonate (EC) and propylene carbonate (PC), all purchased from Aldrich and with purity greater than 98%, were used as starting materials. Prior to use, PAN was vacuum dried for 24 h at 50 °C in a vacuum oven. The other materials were used as received. Fluorine doped tin oxide (FTO) conducting glass with a sheet resistance of 7 Ω cm² and sensitizing dye Ruthenizer 535-bisTBA (N719) and Ruthenizer 535 (N3) were purchased from Solaronix SA. TiO₂ powder with two different particles sizes, namely P25 and P90, were purchased from Degussa.

Electrolyte sample preparation

Gel polymer electrolyte samples were prepared according to the stoichiometric composition (PAN)₁₀(EC)₂₅(PC)₂₀(CsI)_{1.2}(I₂)_{0.12}. In this formula, PAN represents one monomer of polyacrylonitrile. A series of electrolytes were prepared by adding relevant amounts of Pr₄NI (0.0443 g), MPII (0.0126 g), 4-tert-butylpyridine (4TBP) to the (PAN)₁₀(EC)₂₅(PC)₂₀(CsI)_{1.2}(I₂)_{0.12} electrolyte as given in Table 1. Gel polymer electrolytes were prepared using the method already described in a previous report (Bandara *et al.*, 2012).

Table 1: The weight ratios of the (PAN)₁₀(EC)₂₅(PC)₂₀(CsI)_{1.2}(I₂)_{0.12} electrolyte with and without additives (Pr₄NI, MPII and 4TBP).

Sample	PAN/g	EC/g	PC/g	CsI /mg	I ₂ /mg	Pr ₄ NI/ mg	MPII/ mg	4TBP/ mg
<i>a</i>	0.10	0.4151	0.3851	58.977	5.76	-	-	-
<i>b</i>	0.10	0.4151	0.3851	58.977	5.76	44.3	-	-
<i>c</i>	0.10	0.4151	0.3851	58.977	5.76	44.3	12.6	21.7

Photo-anode preparation

In order to prepare the photo-anode, two layers of TiO₂ were deposited on the FTO substrate. The first layer was spin-coated on the FTO using slurry containing TiO₂ P90 powder. The second layer was coated on the first layer with slurry containing TiO₂ P25 powder using the doctor blade (DB) method.

For the first layer (spin-coated layer) preparation, 0.5 g of P90 TiO₂ powder was ground for ~30 minutes with ~2 ml of HNO₃ (pH = 1) in a mortar. The resulting slurry was coated on the well cleaned FTO glass substrate with a Laurel multi-speed spin coater. The coating was done using two speeds. The first stage was done at 1000 rpm for 2 s and the second stage at 2300 rpm for 60 s. During the spin-coating, a part of the FTO substrate was covered with an adhesive tape to avoid coating on the areas required for making electrical contacts. After drying the film in air for ~30 minutes, it was calcined at 450 °C for ~30 minutes.

For the preparation of the second layer, 0.5 g of TiO₂ P25 powder was ground well for ~30 minutes with ~2 ml of HNO₃ (pH = 1) in a mortar. Subsequently, ~0.1 g of Carbowax and few drops of Triton X 100 (surfactant) were added and mixed well. This colloidal suspension was coated on the previously prepared spin-coated TiO₂ layer using the doctor blade method. The resulted electrode was calcined at 450 °C for 30 min to obtain the 2nd TiO₂ layer on the 1st layer. The total TiO₂ thickness of films was ~10 µm. For the dye adsorption, the TiO₂ coated glass plates were immersed in ethanolic solutions of dyes while both the electrode and the dye solutions were kept at about 60 °C. TiO₂ electrodes were kept in the dye solution for 24 hours at room temperature to complete the dye adsorption. After taking out, dye-adsorbed TiO₂ electrodes were rinsed with acetone prior to cell assembly.

Fabrication of DSSCs

The gel electrolyte was casted on the dye-sensitized TiO₂ electrode and then a platinum (Pt) coated conducting glass plate (counter electrode) was gently pressed on top of the TiO₂ electrode to form a DSSC with the configuration glass/FTO/TiO₂ (Spin-coated)/TiO₂(Doctor-blade) +Dye/electrolyte/Pt/glass.

Measurements

The complex impedance measurements of electrolytes were performed using a HP 4192A RF impedance analyzer in the 10 Hz – 10 MHz frequency range. The sample cell for impedance measurements was prepared by sandwiching a thin film of the electrolyte, ~0.5 mm, by two stainless steel electrodes. The measurements were undertaken by changing the temperature of the sample from 60 to 0 °C (on cooling runs) at 5 °C steps. Assembled solar cells were irradiated by a LOT-Oriel GmbH solar simulator 1000 W m⁻² (1.0 Sun) in order to obtain *I-V* characteristics using an eDAQ Potentiostat and e-coder. The area of the cell exposed to light was 12 mm² and the scan rate was 100 mV s⁻¹.

RESULTS AND DISCUSSION

Characterization of TiO₂ Films

Figures 1 and 2 show scanning electron micrographs (SEM) of the 1st and the 2nd layers of TiO₂ prepared on the FTO substrate. The compact nature of the spin coated film (1st layer) is visible in Figure 1 and meso-pores or cracks are not visible in this layer. The film surface is microstructurally homogenous. According to the manufacturer's specification the particle size of Degussa (Evonik) P90 powder is 14 nm, thus particle aggregates are seen in Figure 1 (Jeng *et al.*, 2013) have also observed higher solar efficiencies when smaller particle sizes of TiO₂ are in the bottom layer (the 1st layer) and larger particle sizes of TiO₂ are in the 2nd layer.

The meso-porous nature of the TiO₂ layer prepared by doctor blade method using Degussa (Evonik) P25 (particle size 21 nm) can be seen from Figure 2. This film is also quite homogeneous without any cracks. It has to be noted that this meso-porous nature of the film is important for the efficient dye adsorption due to increased effective surface area of the TiO₂ photo-anode, which finally contributes to increase the short circuit photocurrent and enhance the efficiency. The larger particle size of TiO₂ layers in the 2nd layer could enhance the back-scattering light effectively and increase the photocurrent (Jeng *et al.*, 2013; Zhang and Jianga, 2015). The presence of the compact-spin coated layer in TiO₂ photo anode is important to scatter the light, to provide efficient electron percolation towards FTO from the porous TiO₂ layer and also to prevent internal short circuiting.

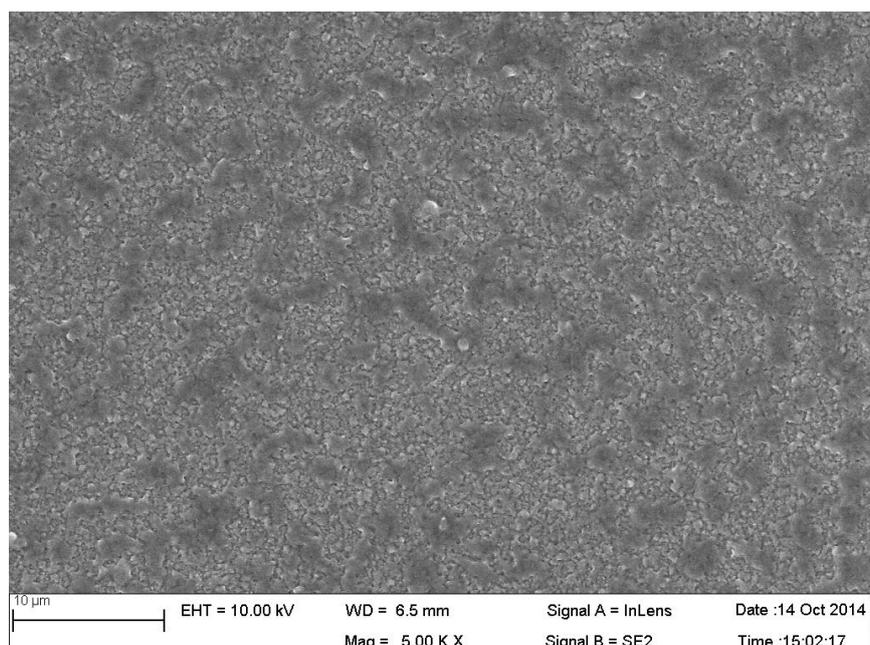


Figure 1: SEM image of the TiO₂ spin-coated layer (the 1st layer) prepared on FTO substrate.

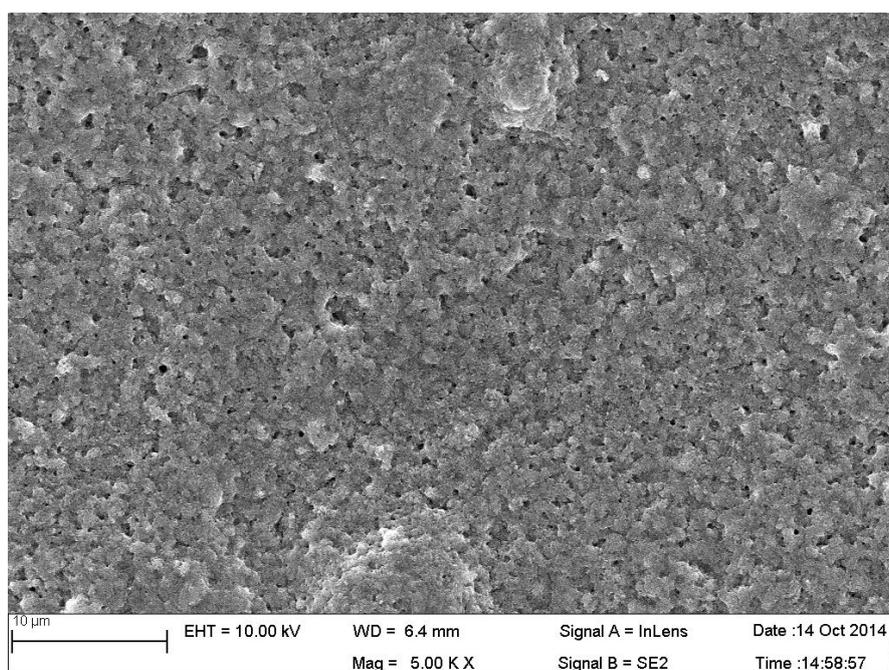


Figure 2: SEM images of the TiO₂ film prepared using Doctor blade method (the 2nd layer) prepared on the spin coated layer.

Characterization of Electrolytes

The ionic conductivities are calculated using complex impedance analysis for all three samples *a*, *b* and *c* given in Table 1. The calculated ionic conductivity isotherms are shown in Figure 3 for the three electrolytes investigated in this work with the molar composition, (PAN)₁₀(EC)₂₅(PC)₂₀(CsI)_{1.2}(I₂)_{0.12} and with different additives (see Table 1).

Samples *a*, *b* and *c* exhibited ionic conductivities of about 2.80, 3.22 and 3.28 mS cm⁻¹ respectively at room temperature. This increasing trend of conductivity with added Pr₄NI and MPII+4TBP is common for all the temperatures studied in the present work. In comparison with single salt (CsI) system 15% and 17% conductivity enhancement is observed in this work with added Pr₄NI and MPII +4TBP respectively.

The conductivity enhancement due to added Pr₄NI salt can be attributed to the increase of density of charge carriers in the electrolyte due to favorable ionic dissociation. In addition, the ternary mixture of cations that provide an increase of the ion concentration and ionic strength do not contribute to the stiffening of the system as an equivalent increase of Cs⁺ concentration would do. The addition of MPlI +4TBP can also contribute to the plasticizing effect, typical of an ionic liquid (Zhang, 2015).

conductivity in an electrolyte depends on the degree of dissociation of the ionic species in the electrolyte. The greater the ionic dissociation, the better is the conductance (Sharma, 1995; Tiyapiboonchaiya *et al.*, 2004). Thus the increasing trend of conductivity with increasing temperature is an expected behavior. However, the conductivity variation with temperature observed in this work can be attributed to VTF (Vogel–Tammann–Fulcher) nature rather than Arrhenius nature (Figure 4). In general, the VTF behavior is common for this kind of gel polymer electrolytes (Bandara *et al.*, 2012).

Figure 4 shows the conductivity as a function of 1000/T for the three electrolyte samples, namely *a*, *b* and *c*. In general, the ionic

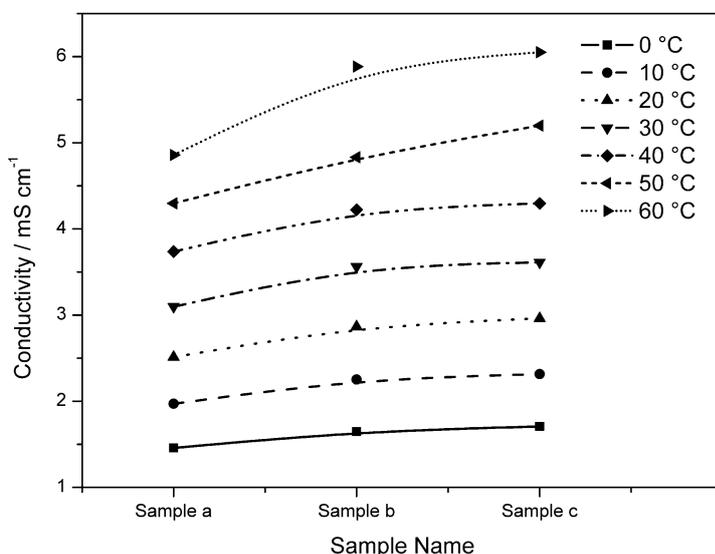


Figure 3: Conductivity isotherms of electrolyte samples *a*, *b* and *c*, prepared according to the compositions given in Table 1.

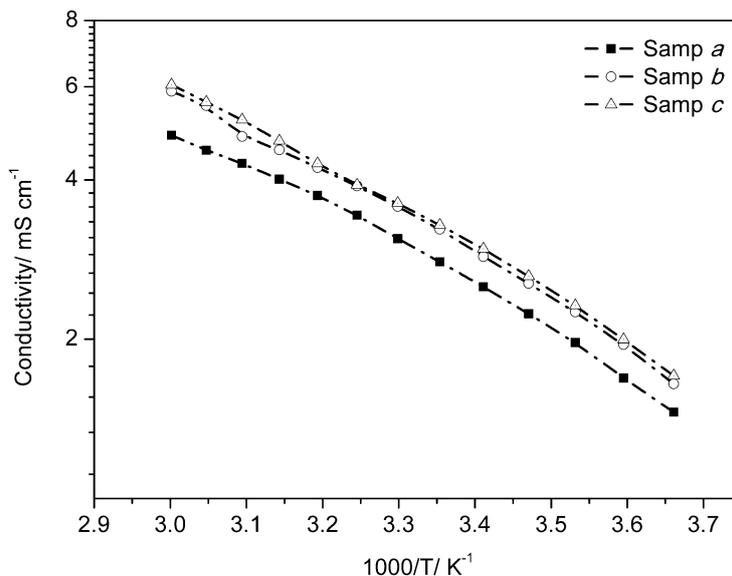


Figure 4: Conductivity versus 1000/T for (PAN)10(EC)25(PC)20(CsI)1.2(I2)0.12 electrolytes prepared according to the compositions given in Table 1.

The photocurrent density-voltage (J - V) curves of the two cells measured under 1000 W m^{-2} (AM 1.5 G) simulated solar irradiation are shown in Figure 5. The short-circuit photocurrent density (J_{SC}) open-circuit voltage (V_{OC}), fill factor (ff) and energy conversion efficiency (η) under the irradiation of 1000 W m^{-2} of cells with N719 and N3 sensitizers are shown in Table 2. The fill factor, ff was calculated using;

$$ff = \frac{J_{opt} V_{opt}}{J_{SC} V_{OC}} \quad (1)$$

where, J_{opt} and V_{opt} are the current density and voltage at maximum power output. The fill factor and the energy conversion efficiency, η , of the cells are also given in Table 2 for all cells. The η value was calculated using;

$$\eta = \frac{J_{SC} V_{OC} ff}{\text{Total incident power density}} \quad (2)$$

As it can be seen from Figure 5, when the N719 dye is attached to the TiO_2 electrode instead of the N3 dye, the DSSC shows a significant increase in photo-current density. The DSSCs with N719 dye show a J_{SC} of 9.77 mA cm^{-2} , whereas the cell with N3 shows 7.77 mA cm^{-2} . The structures of the sensitizing dyes N719 and N3 are shown in Figure 6. As shown in Figure 6, the difference between the sensitizing dyes N719 and N3 is the attachment of two tetra-butylammonium group instead of two carbocyclic groups (COOH) in N3. The larger ligand in N719 dye reduces the charge density on the sensitizer which reduces the electrostatic repulsion and leads to the improvement of dye adsorption (Nazeeruddin, 2011). However, this change causes a significant efficiency enhancement in DSSCs. Though DSSCs with sensitizer N719 showed higher efficiency and J_{SC} , the cell with N3 dye showed higher V_{OC} and ff .

Table 2: The performance of DSSCs prepared using electrolyte sample c for two different dye sensitizers.

Dye	$J_{SC} / \text{mA cm}^{-2}$	V_{OC} / mV	$ff / \%$	$\eta / \%$
N719	9.77	720	58.85	4.14
N3	7.77	746	66.42	3.85

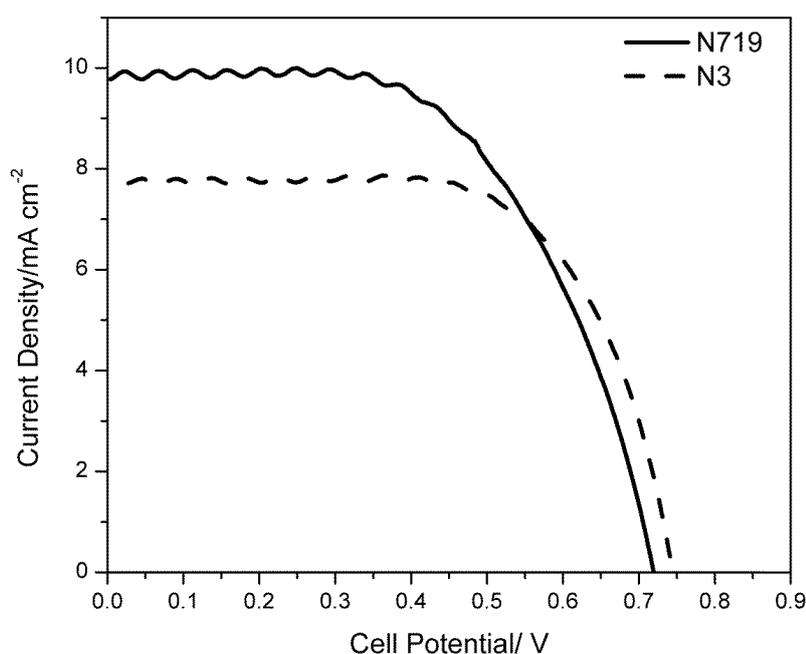


Figure 5: I-V characteristics curves of DSSCs fabricated with electrolyte sample c for N719 and N3 sensitized photo-anodes.

As revealed by Nazeeruddin *et al.*, the photovoltaic performance of a dye complex carrying two protons (N719) is superior to that of compounds contain four (N3) or no protons (Nazeeruddin *et al.*, 2011). The DSSC efficiency enhancement for N719 compared to N3 dye has already been reported for liquid electrolyte based cells (Kalyanasundaram and Grätzel, 2009). However, to the best of our knowledge, this is the first report that compares the performance of quasi-solid state DSSCs with N719 and N3 dye sensitizers. In this study, the maximum energy conversion efficiency of 4.14% was shown by the quasi solid state solar cell with N719 dye sensitizer. In comparison, the N3 based cell exhibited only an efficiency of 3.85%, but produced a higher V_{OC} and ff compared to the N719 based cell. The enhancement of efficiency is about 7.5% due to the change of dye from N3

to N719 in this quasi-solid state configuration. On the other hand, efficiencies of liquid electrolyte based DSSCs with N3 and N719 dyes are 10.0 and 11.18% respectively (Kong, 2007). The enhancement of efficiency between the two dyes is therefore about 10.5%. However, when compared to the maximum efficiency (3.48%), reported for the CsI based quasi solid state single salt system (Bandara *et al.*, 2016) the reported value in this work 4.14% with incorporation of additives represents about 19% efficiency enchantment.

Figure 7 shows the variation of output power density of the two cells against cell potential for DSSCs fabricated with N719 and N3 sensitizers. The maximum power density of 4.14 and 3.85 mW cm^{-2} are shown for the two cells assembled in this study with N719 and N3 respectively.

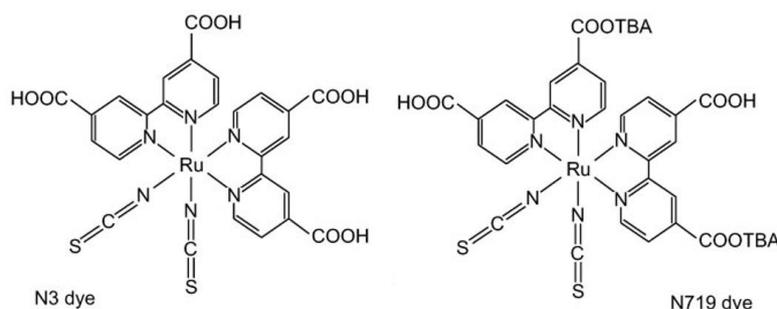


Figure 6: The structure of N3 (cis-Bis(isothiocyanato) bis(2,2'-bipyridyl-4,4'-dicarboxylato ruthenium(II)) and N719 (Di-tetrabutylammonium cis-bis(isothiocyanato)bis(2,2'-bipyridyl-4,4'-dicarboxylato)ruthenium(II)) dye sensitizers. (TBA=tetra-butylammonium)

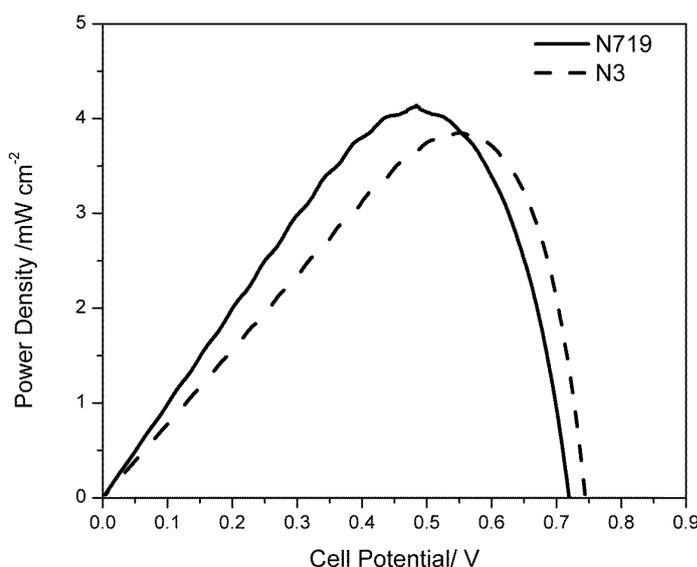


Figure 7: Power density against cell potential for DSSCs fabricated containing electrolyte sample c with N719 and N3 sensitized photo-anodes.

CONCLUSIONS

Gel polymer electrolytes based on PAN host polymer and CsI salt were prepared by incorporating plasticizers EC and PC. The conductivity in the electrolytes were improved by adding Pr₄NI MPII+ 4TBP. The amalgamation of these additives Pr₄NI, and MPII+4TBP enhanced the ionic conductivity in the electrolyte from 2.80 to 3.28 mS cm⁻¹. In comparison with single salt (CsI) system, 15% and 17% room temperature conductivity enhancement is recorded in this work with added Pr₄NI and with MPII +4TBP respectively. This improved electrolyte was used to assemble quasi-solid state solar cells with double layered TiO₂ films sensitized with two dyes N719 and N3.

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ACKNOWLEDGEMENTS

The authors wish to acknowledge the research support provided by Chalmers University of Technology Sweden, Carl Tryggers Foundation for Scientific Research, Swedish Research Council and National Research Foundation of Sri Lanka (Grant 11-196).

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