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Mixed cation effect and iodide ion conductivity in electrolytes for dye sensitized solar cells

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Abstract Dye-sensitized solar cells (DSSCs) offer an alternative to conventional silicon solar cell because of low cost and easy fabrication. However, one major drawback in DSSCs is their low efficiency. This paper reports the effect of using a binary iodide salt mixture with different size cations on the efficiency enhancement in dye sensitized solar cells based on polymer gel electrolytes. Several different polymers, such as polyacrylonitrile (PAN), polymethylmethacrylate (PMMA), poly (vinylidenefluoride (PVdF)), and polyethylene oxide (PEO) have been used as host polymers. A binary iodide mixture consisting of an alkaline iodide salt (small cation) and a quaternary ammonium iodide salt such as tetrapropyl ammonium iodide (Pr₄NI) (large cation) has been used as the iodide ion source. In some of these systems, efficiency enhancement of more than 25% has been reached due to the "mixed cation effect". From these studies, it was established that the variation of the power conversion efficiency with the concentration ratio of the two iodide salts follows the same trend as the short circuit current density (J_{sc}) and goes through a maximum at a particular salt concentration ratio. The maximum efficiency was found to be higher than the efficiencies of the DSSCs

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with only a single iodide salt in the electrolyte. The J_{sc} in these DSSCs appears to be governed by the iodide ion conductivity of the gel electrolyte. The observed efficiency enhancement has been explained on the basis of the electrode effects as well as electrolyte effects where the cations play a dominant role.

Keywords Dye sensitized solar cells · Mixed cation effect · Iodide ion conductivity

Introduction

Dye sensitized solar cells (DSSCs) based on nanostructured titanium dioxide (TiO₂), first reported by Gratzel and coworkers in 1991, offer a low-cost alternative to conventional silicon and thin film solar cells [1]. DSSCs generally employ a solvent electrolyte based on a volatile solvent such as acetonitrile and a Γ/I_3^{-} redox couple and have impressive energy conversion efficiencies reaching around 11%. However, a major drawback of these solution-based solar cells is the lack of long-term stability due to liquid leakage, presence of volatile liquids such as acetonitrile, electrode degradation, and photodecomposition of the dye in the solvent medium. In order to overcome these problems, more stable, gel polymer electrolytes are being developed [2, 3]. In recent years, polyacrylonitrile (PAN), polymethylmethacrylate (PMMA), poly (vinylidenefluoride (PVdF)), and polyethylene oxide (PEO) based gel polymer electrolytes have been used successfully in quasi solid-state DSSCs [4-8]. Either alkali iodides such as LiI or KI and quaternary ammonium salts such as Pr₄NI and Hex_4N^+ are widely used as iodide salts in these electrolytes. Although iodide ion conductivity is an important parameter in determining the short circuit photocurrent and hence the efficiency of DSSCs, to our knowledge not many reports are available on this important subject.

In DSSCs with iodide/tri-iodide (I^{-}/I_{3}^{-}) redox couple, cations in the electrolyte play a dual role in determining overall efficiency of the solar cell. While influencing the short circuit current density through iodide ion conductivity they also influence the charge injection mechanism at the semiconductor/ electrolyte interface. Therefore, it is clear that iodide salts with bulky cations such as Pr₄N⁺ and Hex₄N⁺ through better ionic dissociation are expected to minimize the cationic conductivity and enhance the iodide ion conductivity in the electrolyte, cations with high charge density, such as K⁺, Li⁺ or Na⁺ are expected to contribute towards better photogeneration of electrons at the dye and faster diffusion dynamics at the dyesemiconductor interface [5]. Therefore, it would be interesting to explore the combined effect of using a binary mixture of two iodide salts consisting of a bulky cation and a small cation in the electrolyte in order to benefit from both above mentioned mechanisms [5, 8-10]. In this paper, several representative examples of efficiency enhancement observed in DSSCs fabricated with gel electrolytes containing mixed cation iodide salts consisting of a bulky cation and a smaller cation are discussed.

Materials and methods

Host polymers polyacrylonitrile (PAN), polymethylmethacrylate (PMMA) or poly (vinylidenefluoride (PVdF)), polyethylene oxide (PEO) and iodide salts tetrapropylammonium iodide (Pr₄NI), potassium iodide (KI), lithium iodide (LiI), sodium iodide (NaI), cesium iodide (CsI) etc. all with purity greater than 98% purchased from Aldrich and ethylene carbonate (EC), propylene carbonate (PC) and iodine chips (I₂) purchased from Fluka were used as starting materials. Ruthenium N 719 dye (cis-diisothiocyanatobis(2,2'-bipyridyl-4,4'-dicarboxylic acid) ruthenium(II)) also known as Ruthenizer 535-bisTBA purchased from Solaronix SA was used as the sensitizer in TiO₂ photoanodes in all the DSSCs. All the chemicals except I₂ and PC were vacuum dried at 60 °C for 24 h prior to use.

When preparing the gel polymer electrolytes for all the DSSC systems (other than system given in section 3.1) reported here, the weight of the host polymer (10 wt%) and the weight of the EC (40 wt%) and PC (40 wt%) co-solvent were kept unchanged. The total weight % of the binary iodide salt mixture and I₂ chips (10 wt%) were also kept unchanged while the relative weights of the two iodide salts were varied. The weight of iodine was taken to be one tenth of the total mole amount of the iodide salts. For the system discussed under section 3.1, instead of the EC:PC cosolvent, only PC was used and its weight was taken to be 80 wt%.

Appropriately weighed quantities were mixed in a glass bottle by magnetic stirring. Initially, all the gel electrolyte components, except iodine were mixed by continuous stirring at room temperature for about 2 h until the mixture has been completely dissolved. Subsequently the mixture was stirred further keeping it at 80 °C for about 30 min until the polymer was fully dissolved. Finally the mixture was allowed to cool down to room temperature and I₂ chips were added and again continuously magnetically stirred in a closed bottle overnight (12 h) to obtain a homogeneous gel electrolyte. This procedure was repeated for all the systems reported in this paper.

Ionic conductivity of the gel polymer electrolyte samples was extracted from AC complex impedance measurements done with Solatron SI–1260 impedance analyzer or Autolab (PGSTAT 128 N) impedance analyzer in the frequency range 20–10 MHz. The ionic nature of the gel electrolyte samples was determined by the dc polarization measurements carried out at room temperature by sandwiching the electrolyte sample between two stainless steel (SS) blocking electrolyte sample of about 1 V and measuring the variation of the resulting current with time. This test was repeated for all the samples sandwiching them between two iodine non-blocking electrode pellets in the SS/I₂/electrolyte/I₂/SS configuration in order to measure the iodide ion transference number and estimate the iodide ion contribution to ionic conductivity.

For the fabrication of dye sensitized solar cells, titanium dioxide (TiO₂) paste was first prepared by grinding TiO₂ powder (Degussa P-25) with triton X-100 and ethanol. This paste was spread on a pre-cleaned, fluorine-doped conducting tin oxide (FTO) glass (Nippon sheet glass 10–12 Ω /sq). The paste was spread using the doctor blade technique and sintered at 450 °C for 45 min. The resulting TiO₂ nano-porous film was allowed to cool down to room temperature. Dye absorbance was carried out by immersing the above film in ethanolic dye solution containing Ruthenium N719 dye for 24 h. The gel polymer electrolyte based dye-sensitized solar cells (DSSCs) of configuration Glass/FTO/TiO₂/Dye/electrolyte/Pt/FTO/ glass were fabricated by sandwiching the polymer electrolyte between the dye adsorbed TiO₂ electrode and a platinum (Pt)coated FTO glass. The photocurrent-voltage (I-V) characteristics of the cells were measured under the illumination of 100 mW cm^{-2} (AM 1.5) simulated sunlight.

Results and discussion

PEO: PC: $Pr_4N^+I^- + x$ KI: I_2 gel polymer electrolyte-based DSSCs

Out of the six polyethelene oxide (PEO)-based gel electrolyte samples prepared with different relative weight ratios of the two iodide salts, the highest conductivity of 6.01×10^{-3} S cm⁻¹ at 25 °C was obtained for the electrolyte sample with 100% wt KI salt. However, the highest solar cell efficiency was obtained for the electrolyte sample with

composition PEO:PC: KI: Pr_4NI : $I_2 = 0.1$ g: 0.8 g: 0.045 g: 0.015 g: 0.008 g.

The total ionic conductivity of the electrolyte samples is primarily made up of individual conductivity contributions from K⁺, Pr_4N^+ , Γ and I_3^- ions [9]. DC polarization curves taken for the gel electrolyte samples PEO: PC: $Pr_4N^+\Gamma^- + x$ wt.% KI: I_2 are shown in Fig. 1. The top six curves were obtained using I_2 non-blocking electrodes and the top most curve was taken for sample where 2.5% TiO₂ was added. These DC polarization test results clearly show the variation of the anionic transference number for different electrolyte compositions.

DC polarization data taken using stainless steel (SS) blocking electrodes show that all the gel electrolytes samples studied in this work are predominantly ionic conductors and the electronic conductivity is negligibly small. The steady state DC polarization curves (Fig. 1), taken with iodine pellet non-blocking electrodes in the SS/I2/electrolyte/I2/SS configuration for the gel electrolyte samples, PEO:PC: $Pr_4NI + x$ wt% KI, I_2 for different x values clearly show the different iodide ion (Γ) contributions to the overall ionic conductivity in each sample. From these measurements, it can be concluded that the iodide ion transference numbers for the six electrolyte samples are in the 0.2 to 0.5 range and follows the same trend as the variation of J_{sc} with salt concentration ratio. This suggests that the anionic conductivity by Γ ions in the electrolyte, essentially determines the short circuit photocurrent density of the solar cell.

Out of all the gel electrolyte samples studied, the sample with 75 wt% KI exhibits the highest iodide ion transference

number of 0.49. Addition of 2.5 wt% of nano TiO₂ filler to this sample (top most curve) clearly shows an enhancement in the iodide ion transference number reaching a value of 0.60. It is interesting to note that the variation of iodide ion transference number with salt weight ratio follows the same trend as the variation of the short circuit photo current density (J_{sc}) of the corresponding DSSCs (not shown). Therefore, the increase in short circuit current density can be attributed to the increase in iodide ion (Γ/I_3^-) conductivity in the electrolyte samples. The maxima of both, J_{sc} of the DSSCs and the iodide ion transference number in the electrolyte, occur at 75 wt% KI electrolyte composition for this system.

Figure 2 shows the variation of short circuit current density and the efficiency of the solar cells with respect to the amount of KI% in the electrolyte. The variation of the solar cell efficiency essentially follows the variation of the J_{sc} . The increase of the J_{sc} could be explained in terms of the enhancement of the conductivity of the mixed salt electrolyte and the intercalation of cations into the TiO₂ electrode. Small cations like K⁺ and Li⁺ can get adsorbed by TiO₂ causing a positive shift in the conduction band leading to an increase in the J_{sc} by increasing the charge injection rate while decreasing the open circuit voltage of the cell . The influence of cation charge density on the photovoltaic performance of DSSCs has been studied for Li⁺, Na⁺, K⁺, and dimethyeimidazolium (DMI⁺) ions by Yushuai Shi et al. [10]. The results show that, from DMI⁺ to Li⁺ the short circuit current density (J_{sc}) increases due to the increase in electron injection rates with increase in cation charge density. This positive shift caused by the intercalation of cations varies according to the type and the size of the cation

Fig. 1 DC polarization curves taken for the gel electrolyte samples PEO: PC: $Pr_4N^+\Gamma + x$ wt.% KI: I₂. The top six curves were obtained using I₂ nonblocking electrodes and the bottom most curve was taken using stainless steel blocking electrodes for sample E. The top most curve was taken for sample H where 2.5% TiO₂ was added





Fig. 2 Variation of efficiency and short circuit current density, J_{sc} with respect to the KI wt.% in the electrolyte PEO: PC: Pr₄N⁺ Γ + x wt.% KI: I₂

used. It is greater for small cations and vice versa. On the other hand, though the positive conduction band shift is smaller for bulky cations, the anion dissociation is greater and the cation can be considered to be essentially immobile compared to the anion. Therefore, the existence of the two salts with dissimilar cations gives rise to the maxium in iodide ion conductivity of the mixed salt electrolyte (with 75 wt% KI), and it also increases the charge injection rate and hence the enhancement in the short circuit current density and the efficiency of the solar cell.

PAN:EC:PC:Pr₄NI + KI gel electrolyte-based DSSCs

In this DSSC system, fabricated with the gel electrolyte consisting of polyacrylonitrile (PAN) host polymer, ethylene carbonate (EC) and propylene carbonate (PC) plasticisers,

Fig. 3 The variation of (a) the efficiency (η) and (b) the short circuit current density (J_{sc}) of all six solar cells as a function of the weight percentage of KI salt in the PAN:EC:PC gel electrolyte with KI and Pr₄NI

tetrapropyl ammonium iodide (Pr₄NI) and potassium iodide (KI) mixed iodide salts, the efficiency (η) and the short circuit current density (J_{sc}) of all six solar cells as a function of the net iodide ion concentration [I⁻] from both salts are shown in Fig. 3. It is interesting to see that the efficiency variation follows essentially the same variation as the short circuit current density (J_{sc}) . What is most important to note is that the solar cell fabricated with the binary iodide mixture with 16.6% (w/ w) KI concentration which corresponds to the relative molar iodide ion concentration [I] of 2.08 has shown the highest energy conversion efficiency of 5.36% whereas the solar cells fabricated with the electrolyte with only KI (at $[I^-] = 1.81$) and with only Pr_4NI (at $[I^-] = 3.36$) have shown efficiencies of 4.98 and 4.47% respectively. The use of the binary iodide mixture has clearly enhanced the solar cell efficiency by a factor of about 8% [5].

PMMA:EC:PC:Pr₄N⁺I⁻ + x wt% KI, I₂ gel electrolyte-based DSSCs

For the DSSCs fabricated with the gel polymer electrolyte consisting of polymethyl methacrylate (PMMA) as the host polymer, ethylene carbonate (EC) and propylene carbonate (PC) as the plasticer co-solvent, and $Pr_4N^+\Gamma$ and KI as the mixed iodide salt, PMMA:EC:PC:Pr_4N^+\Gamma + *x* wt% KI, I₂ the variation of efficiency with salt composition appears to correspond to the change in short circuit photocurrent density (J_{sc}) and the iodide ion conductivity [11]. The two end member DSSCs with 100 wt.% KI and 100 wt.% (Pr_4NI) the efficiencies under AM 1.5 illumination are 3.39 and 3.21% respectively while for the solar cell with mixed cation electrolyte with 22.2:77.8 wt.% ratio of KI:Pr_4NI the highest



Fig. 4 The variation of (a) the efficiency (η) and (b) the short circuit current density (J_{sc}) of all seven solar cells as a function of the wt% KI in the PMMA:EC:PC gel electrolyte containing KI and Pr₄NI



efficiency of 3.99% has been observed (Fig. 4). This is an impressive efficiency enhancement of 18%.

The results obtained for the two end members in the present binary system where the J_{sc} values are 9.84 mA cm⁻² for the 100 wt% Pr₄NI system and 13.05 mA cm⁻² for the 100 wt% KI system is consistent with the findings of Yushuai Shi et al. [10] where the influence of cation charge density on the photovoltaic performance of DSSCs has been reported for Li⁺, Na⁺, K⁺, and dimethyeimidazolium (DMI⁺) ions. The drop in V_{oc} is higher for the Li⁺ ions due to a larger downward shift of the conduction band edge induced by the adsorption of Li⁺ cations on the surface of nano titania grains. It is interesting to note that, with the two end members in the PMMA-based DSSC system, also we see this behavior, where the V_{oc} values are 724.3 mV for the 100 wt% Pr₄NI system and 639.4 mV for the 100 wt% KI system.

With identical polymer : EC/PC-salt compositions, the solar cell with KI alone gave an efficiency of 2.37% with J_{sc} of 6.15 mA cm⁻² and the cell with Pr₄NI alone gave an efficiency of 2.90% with J_{sc} of 4.16 mA cm⁻². The cell with the mixed iodide system, KI: Pr₄ NI = 16.6:83.4 (% weight ratio) however, showed an enhanced efficiency of 3.92% with J_{sc} of 9.16 mA cm⁻², open circuit voltage (V_{oc}) of 674.4 mV and a fill factor of 63.4%. We believe that the efficiency

Fig. 5 Variation of (a) Efficiency and (b) Short circuit current density of dye sensitized solar cells with % wt ratio of KI/Pr4NI. The maximum solar cell efficiency and the maximum Jsc occur at the optimum salt ratio 16.6 wt% KI : 83.4 wt% Pr₄NI



Fig. 6 Variation of efficiency (η) and short circuit current density (J_{sc}) of all seven solar cells as a function of the weight of LiI (bottom *x* axis) and weight of CsI (top *x* axis)



enhancement by about 35% and the occurrence of the maxima in the solar cell efficiency and in the short circuit photocurrent at the same iodide salt concentration ratio is an important finding that supports similar results from our previous work [8].

PVdF:EC:PC: KI and $Pr_4N^+\Gamma$, I_2 gel electrolyte-based DSSCs

In the DSSC system fabricated with gel electrolytes consisting of polyvinylidene fluoride (PVdF) as the host polymer and EC and PC as the plasitizer co-solvent, and $Pr_4N^+\Gamma$ and KI as the mixed cation iodide salt, PVdF:EC:PC:(1-*x*) $Pr_4N^+\Gamma + x$ wt% KI, I₂ with x = 0, 8.3, 16.6, 33.3, 50, and 100%, the variation



Fig. 7 The variation of the efficiency in the cells with the alkaline iodide s alt (MI = LiI, NaI, KI, RbI and CsI) in (PAN)₁₀(EC)₂₅(PC)₂₀(MI)_{1.2}(I₂)_{0.12} electrolyte series with and without Pr_4NI

of efficiency with KI salt concentration follows the same trend as the variation of short circuit photocurrent density (J_{sc}) as shown in Fig. 5.

PAN:EC:PC: LiI:CsI, I₂ gel electrolyte-based DSSCs

DSSCs based on this gel polymer electrolyte with polyacrylonitrile (PAN) as the host polymer, and the binary mixture of (large and small cation) alkaline salts, namely cesium iodide (CsI) and lithium iodide (LiI) have also shown an impressive efficiency enhancement due to the mixed cation effect. The electrolyte with the binary mixture of CsI:LiI = 1:1 (by weight) shows the highest ionic conductivity 2.93×10^{-3} S cm⁻¹ at 25 °C. DC polarization measurements were carried out by sandwiching a sample between two stainless electrodes and applying a DC potential of 1.0 V across the sample and monitoring the current. The results (curves not shown) showed that the electronic contribution to the total conductivity is less than 2% and ionic nature is predominant in the electrolyte samples.

DSSCs of configuration glass/FTO/TiO₂/dye/gel electrolyte/ Pt/FTO/glass were fabricated using gel electrolytes of different salt ratios and nanoporous TiO₂ electrodes sensitized with Ruthenium dye N719. Out of all the cells fabricated with these electrolytes, the cell with only CsI gave an energy conversion efficiency of 3.9% while it was 3.6% for the cells with only LiI. However, the cells with electrolyte containing LiI : CsI with mass ratio 1:1 showed the best solar cell performance with an energy conversion efficiency of 4.8% under the irradiation of one sun highlighting the influence of the mixed cation on the performance of the cell. The use of the binary mixture of alkaline iodides showed 23% efficiency and 24% short-circuitcurrent density enhancements [12] as shown in Fig. 6.

PAN:EC:PC:Pr₄NI:MI, I₂ with MI = LiI, NaI, KI, RbI, and CsI gel electrolyte-based DSSCs

For the series of DSSCs based on gel electrolytes with host polymer polyacrylonitrile (PAN) and EC:PC co-solvent and binary iodide salts with mixed cation pairs $Pr_4NI + MI$, I_2 where MI = LiI, NaI, KI, RbI, CsI, and the trend of efficiency variation of the single cation based as well as the binary cation-based DSSCs is shown in Fig. 7. The energy conversion efficiencies in the solar cells based on single cation electrolyte increased with the increasing size of the cation. Therefore, the maximum efficiency in this series, 3.48%, was shown by the CsI containing DSSC. The LiI containing cell showed the lowest efficiency, 0.75%, out of the series of solar cells. The binary cation salt system has shown higher efficiencies compared to the single cation system, highlighting the importance of employing a binary cationic (a large and small) electrolytes in DSSCs. Although, the larger cation containing cells exhibited higher efficiencies for single cation based solar cells, the best efficiency of the binary cation system was shown by the KI containing cell. The binary salt system with KI and Pr₄NI showed the efficiency of 4.93%. The RbI containing DSSC has also shown the penultimate efficiency of 4.34%.

The observed results in several DSSC systems described above can be explained as follows. It is well established that, in DSSCs the presence of smaller cations such as K⁺ ions in the electrolyte increases the short circuit photo current density of the cells, however, at the expense of a lower open-circuit voltage. On the other hand, the presence of larger cations such as Pr₄N⁺ in the electrolyte increases the open circuit voltage at the expense of the decreased short-circuit current density. An optimized balance will be achieved when both additives were used in conjunction as demonstrated in this paper for several different gel electrolyte systems. The reasons are thought to be that the cation adsorption by TiO₂ photoanode shifts the position of the TiO₂ conduction band edge with respect to the LUMO/HUMO (lowest unoccupied molecular orbital/ highest unoccupied molecular orbital) levels of the dye. The adsorption of small size cations such as K⁺ ions positively shifts the conduction band edge of the TiO₂ allowing for faster injection of electrons from the dye. However, this places the conduction band edge closer to the dye HUMO and electrolyte redox potential resulting a reduced open-circuit voltage V_{oc} . The V_{oc} is the potential difference between the conduction band edge of the TiO₂ and the redox potential. On the other hand, the adsorption of large size cations such as Pr_4N^+ ions negatively shifts the conduction band edge resulting in a decreased short current density and an increased open circuit voltage. The presence of both, small size cations and large size cations in the electrolyte, therefore, leads to the highest J_{sc} and highest efficiency at some optimum iodide salt ratio, due to the interplay between the above two effects.

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