Application of LiBOB-based liquid electrolyte in co-sensitized solar cell

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A B S T R A C T
Co-sensitized solar cells have been fabricated using metal complex N3 dye and Ag2S/CdS quantum dots coupled with LiBOB-based liquid electrolyte. Quantum dots (QDs) were synthesized via the successive ionic layer adsorption and reaction (SILAR) route. The absorbance and band gap energy of Ag2S and CdS QDs were determined. Their refractive indices were observed to be in the range of 1.5175–1.5200. It has been shown that LiBOB-based liquid electrolyte is able to function in the QD/N3 dye co-sensitized solar cells but some stability issues of the QD were observed in the electrolyte system containing iodide whereby the QD-sensitized TiO2 was easily etched. Overall efficiencies and fill factors of the co-sensitized solar cells varied from 0.98% to 1.66% and 40% to 46% respectively. CdS QD was shown to be effective when coupled with polysulfide electrolyte while Ag2S QD was favorable towards the LiBOB-based liquid electrolyte.

1. Introduction

Since the introduction of dye-sensitized solar cells (DSSCs), numerous researches have been focusing on materials for use in DSSCs to achieve higher energy conversion efficiency, η [1]. This profound interest is due to the low-cost of the DSSCs and the promising electricity conversion efficiency route for solar energy conversion system [2,3]. DSSC architecture is made up of an electrolyte sandwiched between a dyed nanocrystalline porous semiconductor electrode and a counter electrode. The dye acts as an agent for effective absorption to sunlight. Typically, there are two major types of dye sensitizers used: metal-complex dyes and metal-free organic dyes [4]. A new alternative for dye is to use semiconductor quantum dot (QD) as sensitizers. QD sensitizers have excellent opto-electronic properties [5,6]. Unfortunately, quantum dot-sensitized solar cells (QDSSCs) have lower performance compared to DSSCs.

In order for the DSSCs to be effectively functional, a good electrolyte is required. An efficient electrolyte should take precedence in reducing the oxidized dye rather than recombination with neighbouring dye molecules [7]. This reduction must be able to occur rapidly.

The use of ionic liquid as an electrolyte component is imperative for stable DSSCs [8–10]. Ionic liquids are essentially molten salts which consist of cations and anions. The application of ionic liquids in DSSCs is mainly due to their characteristics of non-volatility and high ionic conductivity. Addition of salt helps in enhancing the efficiency of interfacial charge injection which in turn affects the photocurrent uptake and DSSC’s efficiency [11–13].

In recent years, there has been more research activities in alternative salts such as lithium bis(oxalato)borate (LiBOB). LiBOB is used as an additive for liquid and solid electrolyte particularly in the application of lithium batteries. Thus far, based on the authors’ knowledge, there is no reported work of DSSCs or QDSSCs using LiBOB-based electrolyte albeit its vast benefits such as longer operational lifetime at elevated temperatures and halogen-free environmentally benign material [14].

In this paper, we report the performance of Ag2S and CdS QD solar cells with N3 dye and LiBOB-based liquid electrolyte. The liquid electrolyte also contains 1-butyl-3-methylimidazolium iodide (BMII). The photoelectrochemical properties of the solar cells have been investigated and results are presented and discussed.

2. Experimental

2.1. Materials

The liquid electrolyte was prepared by dissolving lithium bis(oxalato)borate (LiBOB) and 1-butyl-3-methylimidazolium iodide (BMII) in dimethylsulfoxide (DMSO). A fixed amount of lithium iodide (LiI) was also added to the double salt liquid electrolyte system. A desired amount of iodine crystals was added to all electrolytes as well. Both DMSO and BMII were purchased from Fluka while LiBOB powder was obtained from Chemetall, Germany. Iodine crystals and LiI were purchased from Sigma–Aldrich.
Di-isopropoxytitanium bis(acetylacetonate) solution was procured from Aldrich and was diluted with ethanol to obtain a 0.38 M solution. Titanium dioxide (TiO$_2$) paste was purchased from Japan. Platinum catalyst solution (Plastisol), fluorine doped tin oxide (FTO) conducting glass (8 Ω/sq) and Ruthenizer 535 (N3) dye were purchased from Solaronix, Switzerland. Silver nitrate was obtained from Fluka while cadmium nitrate and sodium sulfide were purchased from Sigma-Aldrich.

2.2. Preparation of liquid electrolytes

DMSO, LiBOB and BMII were used as received. Two sets of LiBOB-based liquid electrolyte (LE) were prepared under argon gas condition inside a glove box. In the first set of electrolyte, 0.015 mol of LiBOB salt was dissolved into 15 mL DMSO to form a 1.0 M LiBOB in DMSO electrolyte. The mixture was stirred until homogeneous solutions were obtained. BMII was added to the 1.0 M LiBOB/DMSO electrolyte. BMII was varied from 10 to 50 wt%. The impedance of these electrolytes was determined by electrochemical impedance spectroscopy (EIS) technique using the HIOKI 3520 LCR Hi-Tester interfaced to a computer. The measurement frequency was from 50 Hz to 100 kHz at room temperature. Measurement was done with a liquid sample holder. The complex impedance plot was then established and conductivity was calculated using the formula:

\[
\sigma = l/(R_b A),
\]

where \( R_b \) is the bulk resistance measured at the intersection of the complex impedance or Nyquist plot with the real impedance axis, \( l \) is the sample holder length and \( A \) is the surface area of the electrode/electrolyte contact. The mixture with the highest conductivity at room temperature was selected for use in the DSSC assembly.

A second set of LE was prepared by repeating the same procedure as described above but with 0.0075 mol LiBOB and 0.0075 mol LiI dissolved in 15 mL DMSO. BMII was also added and varied from 10 to 50 wt%. The conductivity of the electrolyte was determined with the same apparatus as described above. For comparison, a polysulfide electrolyte was prepared from 0.5 M sodium sulfide, 2 M sulfur and 0.2 M potassium chloride in methanol/water solution (7:3 in volume) [15].

2.3. TiO$_2$/dye film electrodes and counter electrodes preparation

Fluorine doped tin oxide (FTO) was used as a substrate for both working electrode (WE) and counter electrode (CE). Di-isopropoxytitanium bis(acetylacetonate) was spin coated at 3000 rpm for 10 s on the WE. The di-isopropoxytitanium bis(acetylacetonate) layer and WE were then sintered at 450 °C for 30 min. After sintering, TiO$_2$ paste was doctor bladed on the di-isopropoxytitanium bis(acetylacetonate) layer and the di-isopropoxytitanium bis(acetylacetonate) layer will improve the adhesion of TiO$_2$ to the substrate and at the same time provide a larger TiO$_2$/FTO contact area ratio and prevent electron recombination in the solar cell by minimizing the contact between the redox electrolyte and the conductive FTO surface [16], thus serving as a compact layer. The compact and TiO$_2$ layers were again sintered at 450 °C for 30 min in order to remove any organic residues, moisture as well as to obtain a nanoporous TiO$_2$ electrode.

Ag$_2$S QDs were prepared via the successive ionic layer adsorption and reaction (SILAR) deposition method [17]. The TiO$_2$-coated FTO glass with compact layer was dipped into a 0.1 M AgNO$_3$-ethanol solution for 1 min. The electrode was then washed with ethanol and dipped into a 0.1 M Na$_2$S-methanol solution for 3 min. The cycle was completed with another round of methanol rinsing. The same procedure was applied for the preparation of CdS QDs. In CdS QDs, the electrode was first dipped into a 0.5 M Cd(NO$_3$)$_2$-ethanol solution for 5 min [18]. This was immediately followed by ethanol rinsing before dipping into a 0.5 M Na$_2$S-methanol solution for another 5 min. The cycle was completed with methanol rinsing. For the two QDs, four cycles of SILAR were performed on the electrode, herein referred to as Ag$_2$S (4) and CdS (4). The Ag$_2$S-coated electrode would become darker as the number of dipping cycle increased.

The dye sensitizer was prepared from commercially available N3 dye. 0.003 M N3 dye was prepared by dissolving 10 mg Ruthenizer 535 (N3) powder into 5 mL ethanol. The solution was kept for 24 h in the dark before the electrode was soaked in it for dye anchoring process. Photoanode electrodes were immersed in the dye solution for at least 6 h. The electrodes were then rinsed with ethanol and dried before the assembly of the solar cell. Optical characteristics of the electrodes were characterized with a Shimadzu PC3101 UV–Vis NIR spectrophotometer.

Counter electrodes were prepared by spin coating a thin layer of Plastisol solution on the FTO conducting surface. The electrodes were then sintered at 450 °C for 30 min before used.

2.4. Assembly of QDSSCs

A sandwich-type cell was fabricated by clamping the working electrode with the counter electrode. A 130 μm thick paraffilm was used as a spacer. A droplet of the liquid electrolyte was dropped onto the surface of TiO$_2$/QD/dye film prior to the assembly. The procedure was repeated until the TiO$_2$/QD/dye film was covered with the electrolyte. The effective working area was 0.25 cm$^2$. In both electrolytes, I$_2$ crystal was added to form the redox couple. It was assumed that I$^-$ (which was derived from both Li and BMII) and I$_2$ immediately formed I$_3$ in the ionic liquids [13].

2.5. Photoelectrochemical measurement

Photocurrent–voltage (\( J-V \)) characteristics of the QDSSCs were measured under illumination from a xenon lamp at the intensity of 100 mW cm$^{-2}$ using a Keithley 2400 electrometer. Efficiency was calculated from the equation:

\[
\eta = (J_{SC} \times V_{OC} \times FF) / P_{in},
\]

where \( J_{SC} \) is the photocurrent density measured at short-circuit, \( V_{OC} \) is open-circuit voltage, \( FF \) is fill factor and \( P_{in} \) is the intensity of the incident light. Measurement for each cell was repeated three times to ensure the consistency of the data.

3. Results and discussion

QDs were fabricated via SILAR method which proved to be facile and fast. The dipping cycle for the QD deposition was based on literature [16,17]. The highest efficiency was obtained with four SILAR cycles as reported.

UV–Visible absorption spectrum was performed on the TiO$_2$ film anchored with QD and N3 dye in order to measure their photosponse. Shown in Fig. 1 are UV–Visible absorption spectra of a TiO$_2$ film with Ag$_2$S, CdS, N3 and their combinations deposited on it.

The band gap energy for Ag$_2$S and CdS deposited on TiO$_2$ can be determined by plotting \( (xh\nu)^2 \) against incident photon energy \( h\nu \). Here \( x \) is absorption coefficient, \( h \) is Planck’s constant and \( \nu \) is frequency of the incident photon. The plot is shown in Fig. 2. Band gap energy of the film is obtained by extrapolating a straight line to the horizontal axis, as shown in the figure. The band gap for TiO$_2$/CdS and TiO$_2$/Ag$_2$S films is 2.70 and 2.50 eV, respectively. The band gap energy obtained for TiO$_2$/CdS film is in good agreement with the value of 2.42 eV for CdS [19]. Meanwhile, the band gap energy...
for TiO$_2$/Ag$_2$S film obtained in this study is higher compared to that of pure Ag$_2$S, which is 1.0 eV as reported by Dlala et al. [20]. However, the value obtained in this work is close to the value of Ag$_2$S thin film 2.09 eV reported by Jadhav et al. [21] and between 2.0 and 2.3 eV as reported by Lekshmi et al. [22]. In another study, Chen et al. [23] reported the direct band gap energy for Ag$_2$S thin film as 2.85 eV. The different band gap energy values reported by different group could be related to different synthesis and deposition method of the semiconductor thin film.

It is observed from Fig. 1 that the pure TiO$_2$ film showed low absorption in the wavelength range from 400 to 800 nm. The absorption edge for pure TiO$_2$ film is estimated to be around 387 nm (not shown here) which is similar to the result obtained in reference [24]. The excitonic peak of both Ag$_2$S and CdS QD are observed as broad band from 400 to 550 nm. This is inline with the observed high absorption rate of the QD into the TiO$_2$ film. CdS QD exhibits absorption edge at approximately 520 nm. With the addition of N3 dye attachment, the absorption edge shifted to approximately 550 nm which corresponds to red-shift. The attachment of N3 dye with CdS QD to the TiO$_2$ enhanced the absorption. The same red-shift effect is also observed in Ag$_2$S QD with N3 attachment where the adsorption edge shifted to 580 nm from approximately 530 nm. Ag$_2$S QD having wider band gap as compared with CdS QD as discussed above. The difference in absorption can also be explained using the Urbach model [19,25]:

\[ \alpha(h\nu) = A(h\nu - E_g)^a, \]  
where $E_g$ is the band gap energy of the QD and $a$ is 0.5 and 2.0 for direct transition semiconductor and indirect transition semiconductor, respectively. Since CdS and Ag$_2$S are direct transition semiconductors, $a = 0.5$ [19]. As the $E_g$ is decreased, the light absorption range will be widened, as observed from Ag$_2$S QD. By anchoring N3 dye with Ag$_2$S QD, the absorption intensity is further enhanced.

In order to estimate the refractive index of the QDs and dye, a transmission spectrum of each sample was obtained using Shimadzu PC3101 UV–Vis NIR spectrophotometer. The transmission spectra are shown in Fig. 3. The transmission spectrum is in correlation with the absorption spectrum where high absorption value will yield low transmission rate. High transmission value is observed in TiO$_2$/CdS. However, with the addition of N3 dye as in the case of TiO$_2$/CdS/N3 film, the transmission rate is suppressed at lower wavelength region (less than 500 nm). The same pattern is also observed in TiO$_2$/Ag$_2$S and TiO$_2$/Ag$_2$S/N3 films.

Generally, transmission spectrum is divided into three regions: transparent, weak and medium, and strong absorption regions [26,27]. For weak and medium absorption region, the refractive index of a film can be calculated by the expression:

\[ n = \left[ N_1 + (N_2^2 - n_s^2)^{1/2} \right]^{1/2}, \]  

where $N_1 = 2n_s(T_m - T_m)/(T_mT_m) + (n_s^2 + 1)/2$, $s$ is the refractive index of the substrate and $T_m$ and $T_m$ are the transmission maximum and
In our case, $n_s = 1.517$ for glass substrate.

The refractive index of the films (QD and/or N3 sensitized TiO$_2$ film) were calculated using the envelope curve for $T_m$ and $T_m$ in the transmission spectra and fitting into Eq. (4). Fig. 4 shows the calculated refractive index of the film samples in the specified wavelength range. TiO$_2$/CdS film has a stable refractive index around 1.5175–1.5178. High refractive index is observed in TiO$_2$/CdS/N3, TiO$_2$/Ag$_2$S and TiO$_2$/Ag$_2$S/N3 films at lower wavelength (less than 600 nm). The high value of refractive index at this region is due to the strong absorbance of the films, which is evidenced from the transmission rate. All the three films have refractive index between 1.5175 and 1.5200. Since the difference is not high, we can conclude that the refractive indices are quite stable throughout the visible light spectrum. It was noted that the calculated refractive index for bare TiO$_2$ film is low compared to theoretical value since we used commercial TiO$_2$ paste which is fully transparent. The refractive index has significance on the electronic band structure and is important to be considered for optical related applications.

Two types of liquid electrolyte were prepared and referred to as LiBOB + BMII and LiBOB + BMII + LiI. The conductivity for both electrolytes was measured with varied BMII content. Fig. 5a shows the conductivity of LiBOB + BMII liquid electrolyte against the BMII content at room temperature. Conductivity of the first set of electrolyte increased as the BMII wt% content increased. It reaches optimum level at 30 wt% BMII with conductivity value of $1.27 \times 10^{-2}$ S cm$^{-1}$. The initial increase of conductivity is due to the addition of charge carriers as the BMII content was increased. However, at high BMII content (above 30 wt%), the conductivity of the liquid electrolyte decreased. This is due to the shorter distance between the dissociated ions which promotes recombination into neutral ion-pairs. The formation of ion-pairs does not contribute to conductivity [28,29].

With the addition of LiI in the liquid electrolyte, the conductivity behavior does not differ as compared with the first set of liquid electrolyte. However, optimum conductivity is achieved at lower BMII content i.e. 20 wt%. The conductivity of LiBOB + BMII + LiI liquid electrolyte is plotted as in Fig. 5b. The calculated conductivity at the point is $1.53 \times 10^{-2}$ S cm$^{-1}$. It is noted that at lower BMII content, liquid electrolyte with LiI has better conductivity performance. At 10 wt% BMII, the conductivity of the LiI containing LE is about one and a half times higher than the first set of LE. At higher BMII content, conductivity of liquid electrolyte having additional of LiI dropped significantly as more ion-pairs are formed. It can be observed that at 50 wt% BMII, the conductivity of the first set of LE is higher than the LE containing LiI.
Fig. 5. (a) Conductivity of \((100 - x)\) wt% LiBOB + \(x\) wt% BMII with different BMII content. (b) Conductivity of \((100 - x)\) (60 wt% LiBOB – 40 wt% LiI) + \(x\) wt% BMII with different BMII content.

Fig. 6. \(J\)-\(V\) curves for QDSSCs in 70 wt% LiBOB + 30 wt% BMII electrolyte.
Based on the conductivity results, LiBOB + BMII and LiBOB + BMII + LiI liquid electrolytes with BMII content of 30 wt% and 20% respectively were used in the assembly of the DSSCs. Higher conductivity of the electrolyte will promote improvement of short current density, $J_{SC}$ and efficiency, $\eta$ of the solar cell [11].

The current density, $J$ versus voltage $V$ curves for QD and QD and N3 dye co-sensitized solar cells with LiBOB + BMII electrolyte system are shown in Fig. 6. The value $J_{SC}$, open circuit voltage, $V_{OC}$, fill factor, FF and $\eta$ values are summarized in Table 1. In this system, all the QDSSCs achieved power conversion efficiency above 1.0%. Power conversion efficiency for pure Ag$_2$S and CdS QDSSC are 1.02% and 1.27% respectively. With co-sensitization (QD and N3 dye), a 30% improvement is observed in efficiency. Both co-sensitized Ag$_2$S QD and CdS QD with N3 dye have power conversion efficiency of 1.40% and 1.31% respectively. FF for the two solar cells under this system remains constant i.e. around 37%. The low fill factor value is an indication of back electron recombination coupled with the limitation of the liquid electrolyte to reduce the oxidized QD and/or N3 dye. On the other hand, the reference cell which used N3 dye alone exhibits a better performance with power conversion efficiency of 2.36% and fill factor of 40%.

For LiBOB + BMII + LiI electrolyte system, the $J$–$V$ curves for QD and co-sensitized QD and N3 dye QDSSCs are shown in Fig. 7 while their $J_{SC}$, $V_{OC}$, fill factor and $\eta$ values are summarized in Table 2. For pure Ag$_2$S QDSSC, power conversion efficiency is 0.67% with fill factor 34%. This result is quite close with the result reported by Tubtimtae et al. where they achieved efficiency of 0.98% at 1 sun using polysulfide electrolyte [17]. Ag$_2$S QD was selected for QDSSC due to its lower toxicity compared to common cadmium or lead chalco-
genide QDs. By co-sensitizing TiO$_2$ film with Ag$_2$S QD and N3 dye, a significant improvement in overall efficiency was observed. Power conversion efficiency is increased to 1.66%. The performance is far better than single usage of QD in the cell. This increment is attributed to the increased $J_{SC}$. From the results, both Ag$_2$S QD and N3 dye have contributed together in generating photocurrents with LiBOB and LiI based liquid electrolyte. However, it was observed that the cell lasted for only a few minutes as the Ag$_2$S QD can be corroded by the electrolyte containing iodide where a

<table>
<thead>
<tr>
<th>Electrodes</th>
<th>$J_{SC}$ (mA/cm$^2$)</th>
<th>$V_{OC}$ (V)</th>
<th>Fill factor (%)</th>
<th>Efficiency, $\eta$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ag$_2$S (4)</td>
<td>5.14</td>
<td>0.55</td>
<td>37</td>
<td>1.02</td>
</tr>
<tr>
<td>Ag$_2$S (4) + N3 dye</td>
<td>6.91</td>
<td>0.56</td>
<td>37</td>
<td>1.40</td>
</tr>
<tr>
<td>CdS (4)</td>
<td>5.13</td>
<td>0.67</td>
<td>37</td>
<td>1.27</td>
</tr>
<tr>
<td>CdS (4) + N3 dye</td>
<td>6.00</td>
<td>0.61</td>
<td>36</td>
<td>1.31</td>
</tr>
<tr>
<td>N3 dye</td>
<td>10.34</td>
<td>0.58</td>
<td>40</td>
<td>2.36</td>
</tr>
</tbody>
</table>

**Fig. 7.** $J$–$V$ curves for QDSSCs in 80 wt% (60 wt% LiBOB – 40 wt% LiI) + 20 wt% BMII electrolyte.
diminishing efficiency was observed as time lapsed [30, 31]. The same effect was also observed in LE without LiI.

For CdS QDSSC, its efficiency is not as good as Ag2S QDSSC although the cell achieved better FF. Like Ag2S QD, cell efficiency is improved by at least 50% when CdS QD was co-sensitized with N3 dye. Unfortunately, the hybrid sensitization could not match with that of the cell with only N3 dye. This phenomenon is attributed to the corrosive nature of electrolyte containing iodide towards QD as explained above. As observed, both Ag2S and CdS sensitized QDSSC turned to pale color upon prolonged exposure to the electrolyte containing iodide. As such, CdS QD is fast corroded by the electrolyte containing iodide although the stability is longer as compared with Ag2S QDSSC. Overall, we are seeing power conversion efficiency of QDSSCs in liquid electrolyte system with LiI addition is not comparable with the liquid electrolyte system without LiI addition. Addition of Li+ salt is only beneficial for DSSC where Li+ is adsorbed onto the TiO2 surface and subsequently assists in shifting the TiO2 conduction band edge to a lower level. This in turn lowers the photovoltage and increase the efficiency of charge injection. Ultimately, photocurrent is increased as well as the power conversion efficiency of the DSSC [32].

The performance of the cell is made possible by the characteristics of the QD, dye and the liquid electrolyte. This study has shown that liquid electrolyte of LiBOB + BMII with or without LiI can function for QD, N3 dye or co-sensitized solar cell. On the arrangement of two sensitizers on TiO2 film, QDs were first deposited onto TiO2 electrode. This arrangement works well considering both QD and dye make a cascade structure relative to the electron of TiO2 and hole acceptor (redox couple) [30, 33]. As the deposition cycle is limited to four cycles, there should be some bare TiO2 surfaces left over for the dye attachment. Dye is assumed either to anchor directly onto the bare TiO2 surfaces or the pre-deposited QDs. The probable energy diagram of the co-sensitization of TiO2 with QD and N3 dye is shown in Fig. 8. As soon as electron in dye receives enough energy from the incident light, it will be excited to fill up the lowest unoccupied molecular orbital (LUMO) state. Electron in QD will also be excited and released to the conduction band. The excited electrons will then percolate through the porous TiO2 and eventually reach the FTO. Upon the injection of electrons, both QD and dye are oxidized. The redox couple will then assist in reducing the electrons.

Since electrolyte containing iodide is not suitable for QDSSC, we evaluated QDSSC using polysulfide electrolyte for comparison. The cell performance under polysulfide electrolyte is summarized in Table 3. Fig. 9 shows the J–V curves of the cell under polysulfide electrolyte system. CdS QDSSC performed better with polysulfide electrolyte. N3 dye, however, did not work with polysulfide electrolyte system. Even with co-sensitization of QD and N3 dye, the cell efficiency did not improve. This could be due to the high pH value (>13) of the electrolyte which hinders the functionality of the dye [30].

In general, TiO2/Ag2S/N3 film which has high absorption rate produces better power conversion efficiency in the application of solar cell with LiBOB-based liquid electrolyte. The performance is attributed to the low band gap energy and compatibility with the liquid electrolyte. TiO2/CdS/N3 film also works well with LiBOB-based liquid electrolyte.

4. Conclusion

Liquid electrolyte based on LiBOB has been developed and used in QD/N3 co-sensitized solar cell. Both Ag2S and CdS have showed good optical properties which are important for solar cell application. The electrolyte works well with Ag2S QDSSC though much work is needed to improve the stability of the cell. CdS QDSSC performed best under polysulfide electrolyte with efficiency achieved at 1.37%. Best efficiency was obtained with co-sensitized Ag2S QD and N3 dye solar cell at 1.66% with liquid electrolyte based on LiBOB.

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References


