ORIGINAL ARTICLE

Surface treatment properties of CdS quantum dot-sensitized solar cells

Abdul Razzaq · Jun Young Lee · Bhaskar Bhattacharya · Jung-Ki Park

Abstract The dye-sensitized solar cells (DSSCs) are attractive due to their low cost and promising efficiency. One of the research perspectives in the respective field is to replace the expensive and photodegradable ruthenium metal-based dyes. Present work describes a simple, modified in situ route designed by mimicking the adsorption principle of dyes in DSSCs for surface modification and linking of CdS-Quantum Dots (QDs) to TiO_2 electrode. An organic compound 2-mercaptoethanol (ME) was used as a surface modifying and linking agent. By following this route it was expected to get a well assembled layer of CdS QDs for better cell performance but performances were not as expected. The main reason for low photocurrent density is the partial coverage of QDs surface by ME and the spatial distance between QDs and TiO_2 electrode. Additional surface treatment of the CdS QDs sensitized TiO_2 electrode resulted in an increase in the photocurrent density and photovoltage. This indicates that ME is not an effective capping agent and thus partially covers the QDs surface. The remaining sites, not covered by ME were passivated by sulfur ions in the ionic solution.

Keywords Quantum dots · Semiconductors · Chemical synthesis · Raman spectroscopy · Transport properties

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Introduction

Challenging issues of limited reserves and pollutant emissions of fossil fuels are now directing the research intentions toward the development of alternative, sustainable and renewable initiatives to meet the demand of clean energy. Amongst the various options of renewable sources, solar energy acts as a prominent and abundant source of clean energy. Over the past years tremendous amount of research has been done in the study of solar cells.

In 1991 Professor Michael Grätzel, reported a new architecture for solar cells based upon nanocrystalline TiO_2 electrode and chemical dyes as a photo sensitizer (Regan and Grätzel 1991), termed as dye sensitized solar cells (DSSC). Presently DSSC acts as a promising alternative to the conventional silicon solar cells due to their special features such as low cost, environmental friendly, and moderately high energy conversion efficiency.

Nowadays, enormous efforts are aimed for the performance improvement and long term stability of DSSC. In this regard a variety of investigations have been made by designing dye molecules (Robertson 2006), using organic dyes (Sanghoon et al. 2006) and robust electrolytes (Wang et al. 2003). Currently DSSC based upon ruthenium complex dyes (N3) has reached the power conversion efficiency of about 11 % (Nazeeruddin et al. 2005).

In addition to the dye sensitizers, inorganic semiconductor quantum dots (QDs) have received a wider attention for photovoltaic application. Semiconductor quantum dots can be considered as a good candidate to be a sensitizer due to their specific advantages over chemical dyes. Firstly due to quantum confinement effect they possess tunable band gaps and thus their photoresponse can be tuned (Yu et al. 2003). Secondly semiconductor QDs open a new way to utilize hot electrons via impact ionization (Nozik 2002). In

addition to these, semiconductor QDs are easy to synthesize, cheaper, more stable alternative and posses a larger extinction coefficient as compared to conventional dyes. To fascinate such unique properties of QDs, the QDs are attached to the electrode material surface for solar cell application and the device configuration is termed as quantum dot sensitized solar cell (QDSC). At present there exist various reports about different semiconductor quantum dots such as CdS (Lin et al. 2007), CdSe (Lee and Chang 2008), PbS (Plass et al. 2002), InAs (Yu et al. 2006) and InP (Zaban et al. 1998) which have been synthesized and employed as a photosensitizer.

For the sensitization of semiconductor QDs to the electrode material, generally two routes are followed:

(i) In situ synthesis and deposition of QDs by chemical bath deposition (CBD) (Niitsoo et al. 2006) or successive ionic layer adsorption and reaction (SILAR) (Nicolau et al. 1990) method. In this route the QDs are directly grown onto the electrode material surface by the chemical reaction of respective ionic species.

(ii) Ex situ route linking the synthesized colloidal QDs to the electrode material surface via a bifunctional linker (Robel et al. 2006).

Both routes have their own advantages and disadvantages. The former route shows better performance due to less spatial distance between the QDs and the substrate surface. While the later route has an advantage of controlling the QDs properties like size and shape. However, despite of the advantages known for using QDs, the efficiency of QDSC is still low as compared to DSSC and requires further development. The possible reasons for low photovoltaic performance of QDSC includes the difficulty of assembling QDs into mesoporous film of TiO_2 and presence of a large number of unpassivated or partially passivated surface states. These surface states can act as trap centers for holes and electrons, leading to lower photovoltage and photocurrent (Hodes 2008).

It's well known fact that the carboxylic group in the dyes plays vital role for the adsorption of the dyes upon TiO_2 surface in DSSCs (Thavasi et al. 2008; Statthatos et al. 2001). Adopting the same principle an attempt was made to modify the surface of the CdS QDs by rendering the surface with hydroxyl group using suitable bifunctional linker material. It was expected that with surface representation of hydroxyl group it will lead to better assembling of CdS QDs into mesoporous TiO_2 layer. The experimental procedure based upon in situ route consists of two main steps:

(i) First step consists of synthesis of CdS QDs using conventional CBD method.

(ii) Second step includes the addition of bifunctional linker in the chemical bath. The purpose of this addition is the surface modification of the CdS QDs and linking the QDs to the electrode material simultaneously in the same bath.

This route is expected to yield better coverage of surface modified CdS QDs which in turn may result in the better assembling upon TiO_2 surface.

In this paper, we report our observations, following this route. 2-Mercaptoethanol (ME) has been used as the bifunctional linker. The role of ME has been verified and been shown to work as a linker with the TiO_2 electrodes. QDSC properties have also been examined upon ME linkage. Furthermore we have also analyzed the effect of additional surface treatment of the TiO_2/CdS QDs electrode using an ionic solution providing sulfur ions and evaluate the cell performance before and after the surface treatment. After the surface treatment an increase in the cell efficiency was observed which was mainly contributed by increase in the photocurrent. This demonstrates that after the sensitization of QDs followed by this route there are still many unpassivated sites in terms of Cd^{2+} dangling bonds, acting as electron trap centers. Sulfur ions in the ionic solution may coordinate with these sites and thus enhances photocurrent by removal of these electron trap centers.

Experimental section

Materials used

The precursors employed in this investigation to synthesize CdS QDs include cadmium acetate dihydrate [$(C_2H_3O_2)_2$ Cd·$2H_2O$, 98 %], thiourea [(CH_4N_2S), 99 %], ammonium chloride[(NH_4Cl), 99.5 %], liquid ammonia[(NH_4OH), 28 %] and 2-mercaptoethanol (98.0 %). Sodium sulfide nonahydrate [$(Na_2S·9H_2O)$, 98 %] ionic solution was used for surface treatment of TiO_2/CdS QDs electrode. All the chemicals except liquid ammonia were purchased from Aldrich and used as such. Liquid ammonia was purchased from Junsei Chemicals and also used as procured.

Preparation of CdS/TiO_2 Photoelectrode

Synthesis of CdS QDs

Firstly the solutions of 0.3 M cadmium acetate dihydrate [$(C_2H_3O_2)_2$ Cd·$2H_2O$, 98 %] and 0.9 M ammonium chloride [(NH_4Cl), 99.5 %], were mixed and vigorously stirred. Then this solution was mixed with 2.1 M thiourea

[(CH_4N_2S), 99 %] solution. The pH of the complete mixture was maintained between 9 and 12 by adding liquid ammonia with the temperature maintained at 45 °C. Liquid ammonia acts as a complexing agent forming intermediate cadmium complex [$Cd(NH_3)_4^{2+}$] releasing cadmium ions and also making the medium alkaline to release sulfide ions from thiourea. The ionic states of both precursors combine to form CdS nanocrystals. The reaction mechanism considered to be the same as proposed by Ortegu-Borges (Borges and Lincot 1993).

Sensitizing of CdS QDs

Just after the addition of the liquid ammonia the growth prevention and linking of the QDs were carried out by adding 0.1 mM ME directly into the mixed solution. In the meanwhile, already prepared TiO_2 electrode was dipped in the chemical bath with CdS QDs solution and the CdS QDs got linked to the TiO_2 electrode via bifunctional linker. The TiO_2 electrode was immersed in the CdS QDs solution for 1 h. After that the TiO_2 electrode was washed with DI water and air dried. The nanoporous TiO_2 electrode was prepared according to procedure described elsewhere (Bhattacharya et al. 2007).

Surface treatment

The surface treatment of above prepared TiO_2/CdS QDs electrode was performed by dipping in 0.3 M ionic solution of sodium sulfide nonahydrate [($Na_2S \cdot 9H_2O$), 98 %]. The TiO_2/CdS QDs electrode was dipped for 15 min in the ionic solution, taken out, washed with DI water and air dried.

Solar cell fabrication

TiO_2/CdS QDs electrode and a drilled platinum coated counter electrode were used to fabricate a solar cell. The procedure followed for platinum coated counter electrode is described elsewhere (Bhattacharya et al. 2007). 60 μm thick Surlyn (Solaronix) film was used as a spacer and sealant. The Surlyn film was sandwiched between front and counter electrode and pressed for 2 min at 100 °C. The liquid electrolyte was injected by using syringe via drilled holes. The holes were sealed by using 0.5×0.5 cm^2 dimensions of Surlyn film. Both pieces of Surlyn film were placed on the holes, covered with cover glass and pressed for 1 min at 100 °C.

Two samples of solar cells were prepared. One having TiO_2/CdS QDs electrode without additional surface treatment while the other with TiO_2/CdS QDs electrode after the surface treatment by sodium sulfide ionic solution.

The liquid electrolyte used here was based upon polysulfide redox couple. The electrolyte was prepared as reported by Lee et al. (2008). Briefly 0.5 M Na_2S (nonahydrate) and 2.0 M sulfur were dissolved in methanol/water system having ratio of 7:3 respectively. The electrolyte used was without any additives.

Measurements

To confirm the change in bandgap, the UV Vis spectra of CdS QDs, bare TiO_2 electrode, TiO_2/CdS QDs electrode and surface treated TiO_2/CdS QDs electrode were recorded using Optizen 3220 UV (Mecasys) spectrometer. The transmission electron microscopy (TEM) of CdS QDs sample was carried out in order to analyze the size of CdS QDs. For TEM, samples of CdS QDs were prepared as described above. One drop of this suspension was loaded onto a copper grid and dried prior to the TEM examination. For TEM observations, Techni 20G2 TEM operationg at 200 kV was used. Raman Spectroscopy of the TiO_2/CdS QDs electrode was carried out to confirm the linking of CdS QDs to the substrate. Photoluminescence measurements were performed using a Perkin-Elmer Model LS-55 with a Xenon flash lamp and gratings to provide required excitation. All the measurements were carried out at room temperature with excitation wavelength of 370 nm.

Photocurrent density–photovoltage (J–V) characteristics of the solar cells were recorded with a computer controlled digital source measure meter (Solartron 1287 potentiostat/galvanostat) by applying external potential bias to the cell and measuring the photocurrent generated under a solar simulator (LS-150, ABET technologies) with a 1.5 AM filter.

Results and discussion

Synthesis and characterization of QDs

The CdS QDs were synthesized, surface modified and linked to TiO_2 electrode as per the scheme mentioned above. The bare TiO_2 electrode and TiO_2/CdS QDs electrode (linked via ME) were subjected to UV–Vis spectroscopy. Figure 1 shows the UV Vis absorption spectra of the bare and TiO_2/CdS QDs electrode. On comparison with bare electrode, a shift in the absorption towards the longer wavelength is observed for the TiO_2/CdS QDs electrode and is indicated by absorption peak around 380 nm. This shift indicates the presence of the CdS QDs upon TiO_2 electrode.

The optical bandgap for bare TiO_2 electrode and TiO_2/CdS QDs electrode were calculated using Tauc's relationship (Bhattacharya et al. 2009).
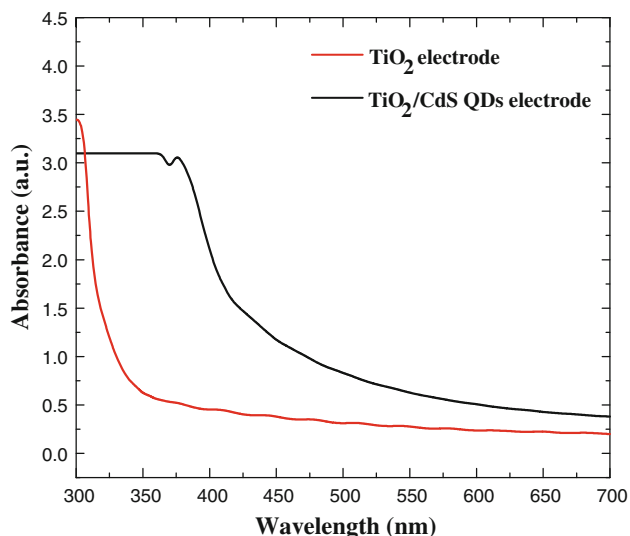
Fig. 1 Absorption spectra of bare TiO₂ electrode and TiO₂/CdS QDs electrode. A clear observation of shift in absorption towards *red* region after CdS QDs deposition
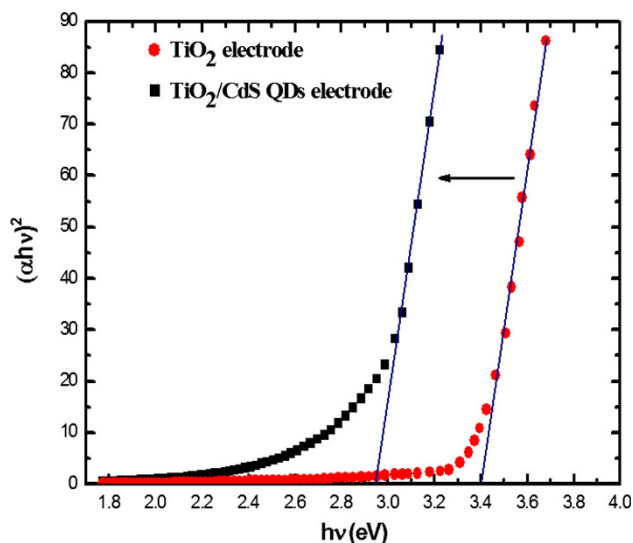

Fig. 2 Optical bandgaps of bare TiO₂ and TiO₂/CdS QDs electrode. Optical bandgaps estimated by plotting $(\alpha h\nu)^2$ (a. u) versus photon energy hν (eV) using the data of Fig. 1

$$\alpha h\nu = (h\nu - Eg)^n$$

(Where $n = 1/2$ for CdS).

The plots between the $(\alpha h\nu)^2$ and hν (incident light) is shown in Fig. 2. It is obvious from figure, the bandgap of bare TiO₂ electrode is shifted from 3.4 to ∼2.9 eV in case of TiO₂/CdS QDs electrode. This shift is attributed to the linking of the CdS QDs to the bare TiO₂ electrode thus reconfirms the shifting of absorption spectra towards the red region.

For the assurance of quantum confinement effect and size estimation, the UV Vis spectra and bandgap calculation were also conducted for colloidal CdS QDs. The

colloidal CdS QDs were prepared by following proposed experimental procedure. Figure 3 shows the UV Visible absorption spectra and bandgap estimation for the as pre-pared CdS QDs colloidal solution. It is evident from the figure the CdS QDS shows a blue shift in the absorption as compared to the bulk CdS. The absorption region for the CdS QDs was observed in the range of 375–450 nm while the absorption region corresponding to bulk CdS appears in the range of 475–550 nm (Thangadurai et al. 2008). This shift is attributed due to the increase in bandgap with decrease in size assuring the quantum confinement effect. The bandgap estimated is shown in Fig. 3(b). The bandgap for CdS QDs was found to 2.8 eV showing the shift in the excitonic peak to higher energy as compared to bulk CdS bandgap which is 2.42 eV. This shift again indicates the quantum size effect due to decrease in particle size. According to the absorption peak position, the size of the QDs was estimated around 4.0 nm. The size was estimated using the data reported by Yu et al. (2003).

For further confirmation of size, the QDs were examined under TEM. The TEM image of the Cds QDs is shown in Fig. 4. The particles could be seen as clusters of random orientation and sizes. The average size of the dots, as estimated from the image is ∼4–5 nm which is in good agreement with that obtained from the absorption data.

As mentioned earlier for CdS QDs sensitization, the TiO₂ electrodes were immersed in the chemical bath just after the addition of ME. ME here is assumed to acts as surface modifying, capping as well as linking agent. ME consists of the thiol (HS⁻) group at one end and the hydroxyl (OH⁻) group at the other end. The thiol group is known as a soft electron donor and coordinates with the cadmium ions present on the surface of CdS nanocrystal. Thus ME can be easily adsorbed to the CdS nanocrystal surface, rendering the CdS surface by hydroxyl group at the other end at the same time acts as capping agent restricting the further growth of the CdS. While the hydroxyl group is a hard electron donor and coordinates with the TiO₂ elec-trode surface and thus plays the linking role of ME.

For confirmation of our presumption that ME works as the linker, dispersive Raman spectra was obtained for the TiO₂/CdS QDs electrode and is shown in Fig. 5. It can be seen with the absorption bands of TiO₂ (645, 520, 395, 145 cm⁻¹) and CdS (300, 600 cm⁻¹), the appearance of absorption band near 2,935 cm⁻¹ corresponding to the CH₂ stretching indicates the presence of the ME as a linker. Inset shows the enlarged view of the region showing CH band.

Surface treatment

The TiO₂/CdS QDs electrode was subjected to surface treatment by ionic solution providing the sulfur ions. The TiO₂/CdS QDs was dipped in 0.3 M sodium sulfide

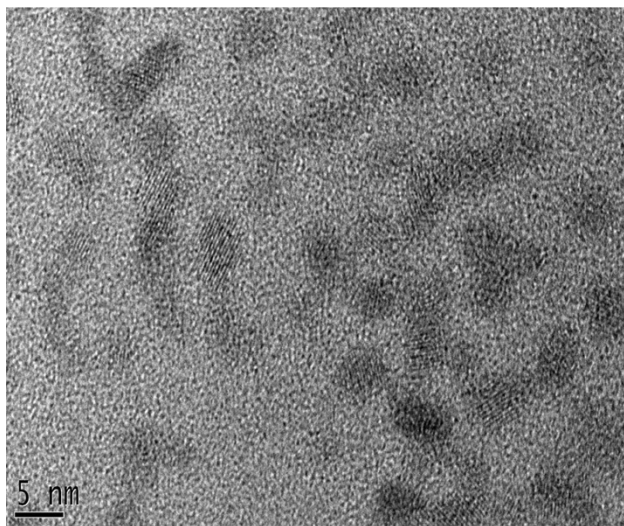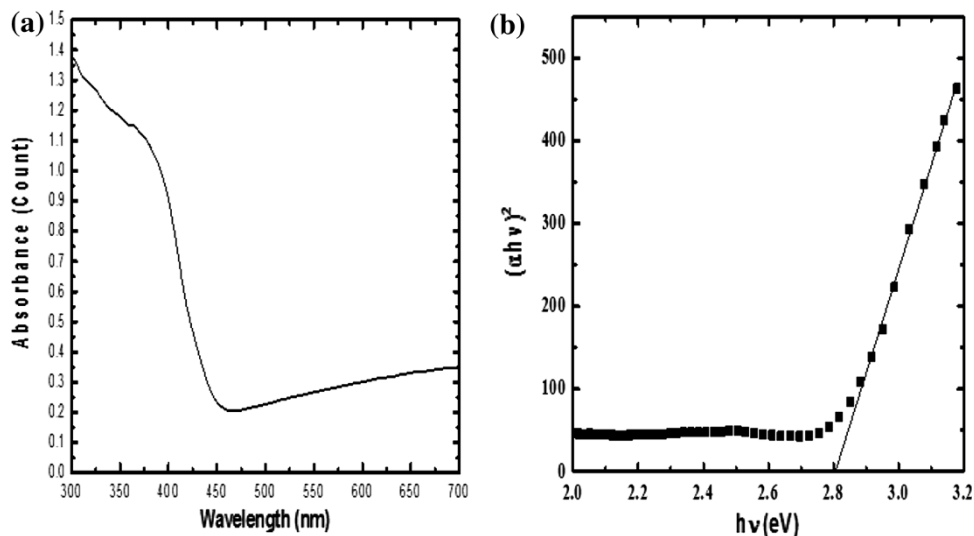**Fig. 3** UV Vis absorption spectra (**a**) and optical bandgap (**b**) for CdS QDs

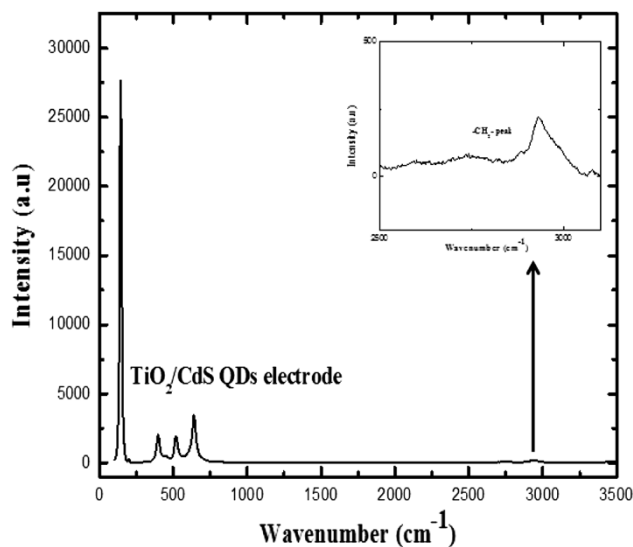**Fig. 4** TEM photograph of the CdS QDs capped by mercaptoethanol

Fig. 5 Raman Spectra of TiO$_2$/CdS QDs electrode. –CH$_2$– absorption band peak observed at 2,935 cm^{-1} along with the peaks of anatase TiO$_2$ (645, 520, 395, 145 cm^{-1}), rutile TiO$_2$ (615, 445 cm^{-1}) and CdS(300 cm^{-1}). The –CH$_2$– peak indicates the presence of ME as a linker

solution for 15 min, taken out, washed and air dried. After the surface treatment the UV–Vis spectra of the samples were recorded and compared with the untreated sample. The UV–Vis spectra of untreated TiO$_2$/CdS QDs and treated TiO$_2$/CdS QDs electrodes are shown in Fig. 6. It is clear from figure that there is no difference in the absorption peak in either of the cases indicating no modification in the electronic states of CdS but shows an increased energy absorbance after treatment. This means that some surface states still exist upon CdS QDs surface in the form Cd^{2+} bonds even after the surface modification. The sulfur ions (S^{2-} or HS^{-}) in ionic solution, therefore, partially coordinates with the available uncoordinated Cd^{2+} sites thus increasing the optically active CdS sites.

Thus it can be described that the ME acts as surface modifier and linker but not an effective capping agent as there still remains uncoordinated Cd^{2+} bonds upon the

QDs surface. The reason for partial passivation or capping can be described as ME is a bulky organic compound and the surface of the CdS QDs might be partially covered by the ME limited by steric hindrance (Lee et al. 2009). The sulfur ions (S^{2-} or HS^{-}) provided by the ionic solution of sodium sulfide might coordinate with the remaining Cd^{2+} dangling bonds, removing the electron trap centers and thus might lead to increase in cell efficiency.

In order to further confirm the surface treatment effect of sulfide ions, photoluminescence spectra (PL) of colloidal solutions containing CdS QDs and surface treated CdS QDs were obtained and are shown in Fig. 7. It is evident from the figure that the PL spectra for both CdS QDs and surface
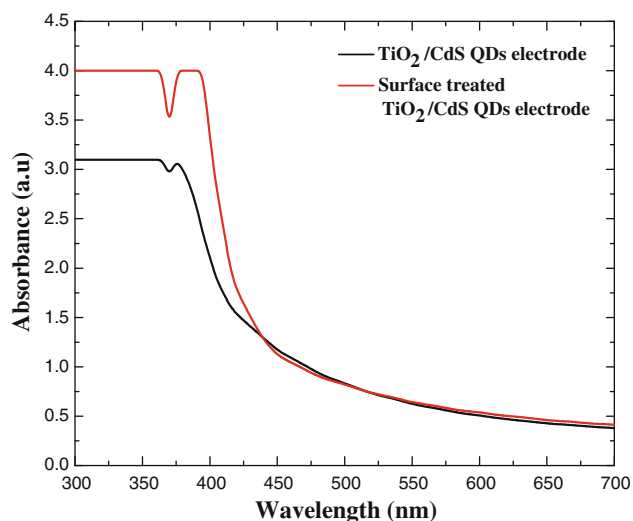
Table 1 The cell parameters, with TiO$_2$/CdS QDs electrode and with surface treated TiO$_2$/CdS QDs electrode

Electrode type	J_{sc} (mAcm^{-2})	V_{oc} (V)	Fill factor	Efficiency (%)	IPCE (%) ($\lambda = 350$ nm)
TiO$_2$/m-CdS electrode	1.14	0.40	0.40	0.18	14
Surface treated TiO$_2$/m-CdS electrode	2.1	0.48	0.24	0.34	19
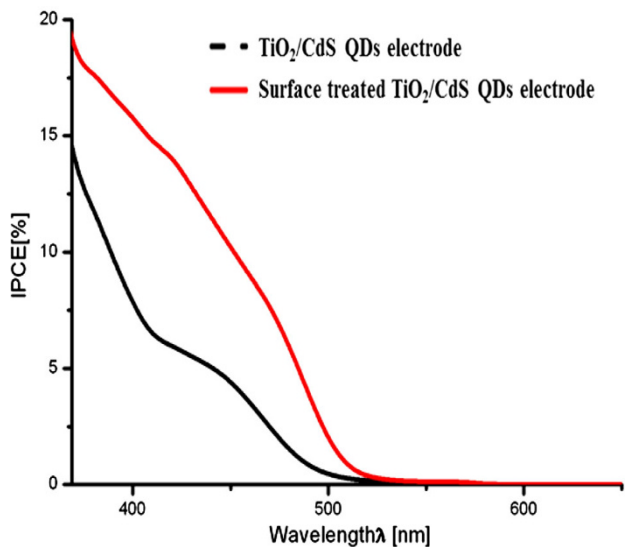
Fig. 9 The IPCE of TiO$_2$/CdS QDs electrode and surface treated TiO$_2$/CdS QDs electrode as a function of wavelength of the illumination light

a possible reason for the low fill factor and cell efficiency (Iván et al. 2008). The distance can be reduced by selection of feasible deposition method and an energy barrier shell approach can be employed to prevent the electron recombination losses.

The IPCE spectra of both cells were also measured as shown in Fig. 9. The IPCE spectra carried out at different wavelengths measured for short circuit currents. The spectra of the cells closely resembles with absorption spectra of both electrodes shown in Fig. 6. It is clear from figure, the IPCE measurement shows the max. value at a wavelength of 350 nm. At this wavelength an increase in IPCE was observed from 14 to 19 % after surface treatment of the TiO$_2$/CdS QDs electrodes. This increase in IPCE leads to the increase in the charge collection efficiency after surface treatment due to increased optically active CdS sites leading to increase of photo injected electrons in TiO$_2$ conduction band. This is in confirmation with our earlier statement on the passivation of surface states of TiO$_2$/CdS QDs electrode before and after surface treatment.

Conclusions

An in situ route mimicking the adsorption principle of dyes in DSSCs, was employed for synthesis, surface modification and linking the CdS QDs to the TiO$_2$ electrode.

Following this route it was expected to obtain a well-covered monolayer of QDs upon TiO$_2$ electrode thus enhancing the cell performance. An organic compound ME was used for QDs surface rendering and linking simultaneously in single bath. The thiol moiety of ME coordinates with the surface Cd^{2+} ions by donating electron and thus can be considered as surface passivating agent. On additional surface treatment of the TiO$_2$/CdS QDs electrode it was observed that the ME partially coordinates with the CdS surface and there still remain surface states. The possible reason for the partial passivation is the less coverage of CdS QDs by bulky organic compound due to steric effects. An increase in the photocurrent density was observed after the surface treatment of TiO$_2$/CdS QDs electrode by sodium sulfide solution. This increase is due to coordination of sulfur ions with the uncoordinated Cd^{2+} dangling bonds upon the QDs surface which were previously not coordinated by ME. However the fill factor and efficiency are still very low and requires enormous efforts for improvement. Low hole scavenging rate of polysulfide redox couple and the spatial distance between the QDs and TiO$_2$ electrode created by linker are considered to be the possible reasons for low fill factor and cell efficiency.

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