Salen Zn complexes along with ZnO nanowires for dye sensitized solar cells

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Abstract

Zn Schiff based complexes with high conductivity were synthesized with a facile approach at low cost. As dye sensitizers, Zn complexes have been attempted to construct dye sensitized solar cells (DSSCs), along with vertically aligned ZnO nanowires as a scaffold and an electron transporting layer for more dye load and better electron transporting pathways. The power conversion efficiency was examined by the I-V curve. Compared with the solar devices based on conventional TiO2 nanoparticles with an efficiency of 0.815%, the solar cells with ZnO nanowires outperform greatly, with a much better efficiency of 3.68%, which holds a great potential for the widespread applications in dye sensitized solar cells.

Introduction

The ever-increasing energy demand, particularly by the rapid industrialization of developing countries, prompts more and more people to accept solar energy as a promising renewable substitute for the conventional energy sources like fossil fuels, because of the non-exhaustible sunlight resources, yielding no harmful byproducts [1] to our environment. Solar cells are electrical devices which directly convert the solar energy into electricity by the photovoltaic effect. In spite of a great deal of attention on solar cells for more than a few decades, some challenging issues in solar energy fields have not yet been addressed. For instance, silicon-based and inorganic compound type solar cells have been intensively investigated and even commercialized, because of their high efficiency. The pricey manufacturing cost and detrimental effects to the environment or the instability are still problematic [2, 3]. Comparatively, dye-sensitized solar cells (DSSCs) are set apart from the conventional photovoltaic devices because of their remarkable power conversion efficiency, low price-to-performance ratio, low processing cost, ability to work at wide angles, low intensities of incident light, light weight, mechanical robustness and environmental friendliness [4, 5]. For this reason, enormous efforts have been devoted to the investigation into DSSCs in hopes of achieving better competitive efficiency.

Typically, DSSCs consist of four main parts: the porous nano-crystalline wide bandgap semiconductor electrode (photoanode), dye (sensitizer), counter electrode and electrolyte [6]. One striking merit of DSSCs lies in the flexibility of the choice of materials, in which all included parts can be varied and properties may be finely tuned. This is particularly true for nanostructured wide bandgap semiconductor photoanodes, which play a dual role in device functioning, acting as a scaffold for the dye loading and transporting photo-excited electrons from dye to external circuit. Generally, two requirements must be fulfilled. The first one is the large surface area which ensures high dye loading. The second is a fast charge transport to guarantee high electron collection efficiency, which are the defining characteristics of an ideal photo-anode. The best performing DSSCs to date utilize the sintered nanoparticle-based mesoporous TiO2 films as photoanodes [7]. Although the large surface area (∼50 m2 g−1) of spherical TiO2 nanoparticles enables high dye loading capacity, numerous grain boundaries, resulted from randomly disordered network, hampers electron mobility, which consequentially leads to slow transport and recombination of photo-excited electrons. As a result, the overall efficiency of DSSCs is greatly
restricted. Therefore, the inherent drawbacks of the standard photoanode construction necessitate a search for the ideal material for the effective transportation of charges generated in the dye over the semiconducting nanostructures, which is pivotal to optimizing the overall performance of DSSCs. As a stable, nontoxic oxide and n-type semiconductor, zinc oxide (ZnO) (3.37 eV energy gap), with a large exciton binding energy of 60 meV at room temperature, is known for its high electric conduction and carrier mobility \[8\], which has been extensively explored as popular photoanodes \[9\]. Particularly, ZnO nanowires (ZnONw) have attracted tremendous attention because of their exceptional photo-electrical properties and promising potential applications in optics, electronics, optoelectronics, light-emitting diodes \[10\], and solar cells \[11\].

Another critical issue in DSSCs is the ‘right option’ of dye. As semiconductors with high reductive activity and conductivity, Schiff bases and their metal complexes have been proven as promisingly effective light-absorbers (DSSCs photosensitizers) with high photoelectric conversion efficiency \[12\]. Salen ligands are members of Schiff bases. The salens are excellent alternatives to porphyrins because the tetradentate binding site of salen ligands is similar to the porphyrin framework. Additionally, salen derivatives are more easily synthesized with much lower cost compared with porphyrins. More importantly, salen ligands possess many advantageous properties. For example, salen ligands can form complexes with almost all metals, which provides a large freedom to finely tune the optical and electrochemical property by adding electron-withdrawing or electron-donating substituents on the salen framework or changing the metal center of the complexes. The resulting salen-metal complex based dyes are supposed to have better sensitizing properties for photovoltaics as well as higher chemical and thermal stability, intense absorption in the visible region, strong adsorption onto the semiconductor surface and efficient injection of electrons onto the conduction band of the semiconductor than pure Schiff base compounds. However, little research has been reported on the use of salen-metal complexes as dyes in DSSCs.

Therefore, in this project, ZnO nanowires will be adopted to substitute for the conventional electro anode TiO$_2$. The target central metal will be zinc (Zn) because Zn complexes have the highest conductivity in dye sensitizers \[13, 14\]. New salen-Zn complexes with carboxyl anchoring groups will be developed and added onto ZnO nanowires as novel photosensitizers to improve the efficiency of solar cells.

**Experimental details**

ZnO nanowires (NWs) were synthesized through a one-step thermal decomposition approach \[15\]. The precursor zinc acetate dihydrate (Zn(CH$_3$CO$_2$)$_2$·2H$_2$O, 99% pure), 0.8 grams, was put in an alumina crucible with an ITO glass as a lid, then placed in an oven. With the rate of 2 °C min$^{-1}$, the crucible was heated to 300 °C and retained at 300 °C for a time period of 5 h, and then naturally cooled to room temperature. The scanning electron microscope (SEM) image, in figure 1(a), reveals that ZnO NWs forest were grown on the alumina crucible and on the ITO lid. The high quality and high purity nanowires are distributed very uniformly with a few micrometers in length, about 40–60 nanometer in diameter. UV-vis optical absorption spectra were recorded in a transmission mode on Varian Cary Bio 50 UV-Visible Spectrophotometer. X-ray diffraction patterns were collected on a Rigaku Ultima IV diffractometer in a grazing incident mode with the incident angle of 0.5 degree. Cu K-alpha line ($\lambda = 154,059$ pm) was utilized as a light source. The XRD profiles in figure 1(b) substantiates further that the NWs are single phased ZnO, possessing the Wurtzite crystal structure with a space group of P6$_3$mc. The estimate lattice constants are $a = 3.25$ Å, $c = 5.21$ Å. The SEM image in figure 1(a) also
displays that the hexagonal structure of each rod and nanorods have uniform size. The quality of crystallinity ensures an excellent carrier transporting property of ZnO, which allows ZnO to be an outstanding electron transporting layer in solar cells.

Salen-Zn complexes was synthesized with an ease approach. The salen ligand was synthesized by a condensation reaction between a substituted salicylaldehyde and 3, 4-diaminobenzoic acid in the molar ratio 2:1. During the condensation reaction, two H2O molecules was eliminated to form two CH=N bonds in the salen ligand as depicted in figure 2(a). An ethanol solution of a substituted salicylaldehyde (2 mmol, 25 ml) was added to an ethanol solution of 3, 4-diaminobenzoic acid (1 mmol, 25 ml) and refluxed at 70 °C–80 °C for 4–6 h. The excess solvent was distilled to separate the precipitates. Then the obtained precipitates were filtered, washed with ethanol, and dried over anhydrous calcium chloride under a vacuum to produce the pure ligand in a decent yield. Salen ligands possess N2O2 donor atoms. When the ligand reacts with metal salt, the metal ion is bonded by coordinate bonds through the N of imno-CH=N groups and the O of phenolic-OH groups to form a stable chelate. The synthetic scheme in figure 2(b) indicates how to obtain salen-Zn complexes from the pure ligand. A mixture of salen H2L (1 mmol, 50 ml) in ethanol was added to an aqueous solution of 1 mmol of the zinc acetate (0.18 g) salt. The reaction mixture was refluxed at 70 °C–80 °C for 4–6 h, and the precipitated solid was filtered from the ice-cooled reaction mixture and washed with ethanol, followed by drying under a vacuum to afford pure metal complex products.

As indicated in the figure 3, construction of DSSCs is as follows: as produced, ZnO NWs on the top of an ITO (indium tin oxide) glass is referred to as ZnO-ITO. ZnO-ITO was carefully placed on the lab station with the ZnO side facing upward. Then two spacers were placed on the ITO glass. Next the sample was dipped into the dye solution for 10 mins. Meanwhile, the as-purchased carbon paste was developed into a film on the conducting surface of another ITO glass as a counter electrode. The sample in the dye solution was taken out and washed with ethanol. All the layers were sandwiched between two ITO slides with binder clips. The last step was to drop electrolyte solution (iodine/triiodide) into the spacing between the spacers. For comparison’s sake, another batch of cells based on TiO2 nanoparticles (as purchased along with ITO, electrolyte as dye sensitized refill kits from Arbor Scientific) was assembled by following the similar steps, in which ITO with a doctor-blading TiO2 film was utilized instead of ZnO-ITO.
Results and discussions

The recorded XRD patterns are shown in figure 4. The XRD measurement was performed at $2\theta = 10^\circ$–$50^\circ$ to examine the structural properties of the synthesized Zn(II) complex. It is clearly seen in figure 4 that Zn(II) complex exhibit diffraction peaks at $2\theta = 17.5^\circ$, $20.6^\circ$, $21.5^\circ$, $24.5^\circ$, $25.3^\circ$, $30.1^\circ$, and $35.7^\circ$, indicating the crystalline phase of Zn(II) complex. The result is consistent with the similar results reported in previous studies [16, 17]. The crystallite size of Zn(II) complex can be estimated from the full width at half maximum of diffraction peaks by following Debye–Scherrer equation, which can be expressed as the formula:

$$t = \frac{k\lambda}{\beta^* \cos \theta}$$

where $t$ is the calculated grain size of the sample, $\lambda$ is the wavelength of X-ray source (about 0.154 nm in our case), $\beta$ is the broadening measured as the full width at half maximum (FWMH) in radians, $\theta$ is the Bragg’s diffraction angle and $k$ is a correction constant (typically $\sim 0.9$). The crystal sizes of Zn(II) complex varies from 25.17 nm to 66.81 nm. Apparently, the synthesized Zn(II) complexes are in nano-size and the average crystal size of Zn(II) complexes were estimated to be $\sim 40$ nm.

The UV–vis absorption spectrum of the Zn(II) complexes inside DMF (dimethylformamide) solution was scrutinized at room temperature, as depicted in figure 5. Two bands at 380 and 430 nm are supposed to appear on the absorption spectrum of Schiff base in which the band (380 nm) is assigned to $\pi-\pi^*$ transitions of phenyl ring with higher energy, while the band at 430 nm is ascribed to $n-\pi^*$ transition of azomethine (–CH=N) group with lower energy [18]. These two absorption bands occur in the spectrum of Zn(II) complex but undergo a blue shift. The high energy band shifts from 380 nm to 350 nm, while the band at 430 nm moves to 410 nm. A blue shift is caused by the coordination of the ligand to Zn(II) ion. In the Zn(II) complex, its d$^{10}$ configuration does not show any d–d transition, hence possibly having tetrahedral geometry. The combination of d$^{10}$ metals and aromatic organic linkers displays a stable structure and enhanced optical properties [19]. As a matter of fact, the presence of zinc atom increases the ligand conformational rigidity, favoring a more planar and conjugated structure. This further enhances the mobility of the electron transition due to back coupling $\pi$-bond between the metal and ligand [20]. Meanwhile, the planar Zn(II) complexes are capable of being specifically absorbed onto the electrode surface, yielding so-called stacks bound by donor and acceptor interactions or inter-molecular interactions [21]. In comparison, metal complex dyes possess more intense absorption in the visible region and stronger absorption onto the semiconductor surface than pure organic dyes leading to more efficient electron injections onto the conduction band of the semiconductor [22].

The IR spectrum of the synthesized Schiff base Zn(II) complex is shown in figure 6. The insert discloses the molecular structure of Zn(II) complex. The broad bands centered on 3200 cm$^{-1}$ in the complex are attributed to the coordinated water with metal ions. A characteristic band at 1601 cm$^{-1}$ in Zn(II) complex is associated with the –CH=N stretching vibrations of the azomethine $\nu$(CH=N), in which an appreciable red shift can be observed when compared with that of the Schiff base ligand at 1618 cm$^{-1}$ indicating coordination of the azomethine nitrogen to zinc metal atom in complexation [23]. The existence of this band, due to the transitions
of $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ and $d \rightarrow d$ of C=N, along with the charge transfer transitions, is caused by $\pi$ electron interactions between metal and ligand, which could be a metal-to-ligand or a ligand-to-metal electron transfer. The metal complexes with Schiff bases are conductive materials allowing for conjugated electron-ion charge transfer. It is worth noting that the band at 3434 cm$^{-1}$, attributed to the phenolic –OH group of the ligand [24], disappears in the Zn(II) complex, due to the involved oxygen atom of the –OH group in the coordination to Zn(II) ion. The coordination of the O atom in the phenolic –OH group to Zn(II) metal is further substantiated by the shift of $\nu$(C–O) band of ligand at 1105 cm$^{-1}$ to 1205 cm$^{-1}$ [23]. The weak peaks observed in the region of 1600–1300 cm$^{-1}$ are caused by C–O, C–H and aromatic ring vibrations. The band that appears at 750 cm$^{-1}$ can be assigned to the C–H bending vibration of O-substituted benzene [25]. In the complex, the band between 1300 cm$^{-1}$ and 1200 cm$^{-1}$ is ascribed to C–O–C symmetric stretching mode of vibration. C–Br bonds are evidenced by peaks around 565 cm$^{-1}$. Due to the presence of different stretching vibrations caused by bonds with metallic ions forming $\nu$(Zn–O) and $\nu$(Zn–N) in Zn(II) complex, the involvement of phenolic O and azomethine N is particularly corroborated by the development of two non-ligand bands in the spectra of complex at 545 cm$^{-1}$ and 456 cm$^{-1}$, respectively. Therefore, it is believed that the Schiff base ligand behaves as a dibasic tetradentate ligand coordinated to the metal ion via the azomethine N and deprotonated phenolic O. The complex possibly has a tetrahedral geometry because of the d$^{10}$ configuration of Zn(II).
Figures 7(a) and (c) show the photo-current-voltage curves of the TiO\textsubscript{2} and ZnO nanowires based DSSCs, respectively. The dark I–V characteristics of two different configurations were investigated as shown by the black curves in figures 7(a) and (c). Both devices show a nonlinear rectifying characteristics in the dark, which is a typical diode-like behavior. It verifies that electrons, staying in the frontier molecular orbital (HOMO), are easily delocalized from the zinc Salen skeleton to the framework of benzene and the anchoring group of carboxylic acid (LUMO). The energy level of conduction band of TiO\textsubscript{2} or ZnO (similar band gaps) is located between the LUMO and the HOMO of Salen Zn dyes. This allows for the electron injection from the LUMO of dyes to be energetically favorable to the conduction band of TiO\textsubscript{2} or ZnO [26]. Under the 1 sun illumination (∼100 mW cm\textsuperscript{-2}), the nonzero photocurrents in both cases indicate the generation of the free carriers by the absorption of the incident photon energy within the depletion region as revealed by the red curves in figures 7(a) and (c).

In order to calculate the efficiency, the photo-currents were flipped into the first quadrant as revealed in figures 7(b) and (d). The most important parameters for solar cells are short circuit current density ($I_{SC} = \frac{I_{sc}}{A}$ A is the surface area of the cell under illumination), open circuit voltage ($V_{oc}$), fill factor (FF), and efficiency ($\eta$). These parameters under 1 sun AM1.5 illumination were summarized in table 1. The efficiency can be estimated by the following equation:

$$\eta = \frac{P_m}{P_{in}} = \frac{I_{sc}V_{oc}FF}{P_{in}}$$

| Solar cell devices | $V_{oc}$ (Voltage) | $I_{sc}$ (Ma cm\textsuperscript{-2}) | Vmp | Imp | FF(%) | Efficiency (%) |
|-------------------|-------------------|-----------------|------|-----|-------|----------------|
| TiO\textsubscript{2} | 0.72              | 3.15            | 0.39 | 2.1 | 36.1  | 0.815          |
| ZnO nanowires     | 0.52              | 17.5            | 0.32 | 11.5| 40.4  | 3.68           |

Table 1. Photovoltaic performance comparison for the cells based on TiO\textsubscript{2} and ZnO NWs.
In which $P_m$ is the maximum output power density of the cells, $P_n$ is the power density of the incoming light, $J_{sc}$ is the short circuit current density in the cells; $V_{oc}$ is the open circuit voltage of the cells and FF is the fill factor:

$$FF = \frac{P_m}{J_{mp} V_{mp}} = \frac{I_{mp} V_{mp}}{J_{sc} V_{oc}}$$

$J_{mp}$ and $V_{mp}$ are the current density and voltage corresponding to the maximum power point, respectively, as indicated in figures 7(b) and (d). Comparing in terms of open circuit voltage, short circuit photocurrent, fill factor and power conversion efficiencies, it can be found that the choice of semiconductor oxide photo anode has a profound impact on the performances of DSSCs. Due to charge (carrier) recombination losses at grain boundaries between nanoparticles, DSSCs containing TiO$_2$ nanoparticle films, which are composed of interconnected spherical particles, show a lower efficiency (0.815%). The average values of open circuit voltage ($V_{oc}$) and short circuit current ($J_{sc}$) for nanoparticle DSSCs are 720 mV and 3.15 mA cm$^{-2}$, respectively. In contrast, the values are 520 mV and 17.5 mA cm$^{-2}$ for DSSCs based on ZnO nanowires film, a significantly larger value for $J_{sc}$, leading to a much higher conversion efficiency (3.68%) than that of nanoparticle DSSCs (0.815%). ZnO NWs have a much better crystalline quality than TiO$_2$ nanoparticles, which increases electron diffusion from the point of electron ejection into the circuit because electrons do not suffer any grain-boundary scattering. In the meantime, the multi-scattering effect between the vertically aligned ZnO NWs include a direct pathway for accelerating electron transport, simultaneously extending the lifetime of electron, suppressing the recombination of electrons and holes, and improving electron collection efficiency which directly lead to the enhancement in external quantum efficiency of solar cells [29]. Moreover, the photo–electric conversion efficiency of DSSCs based on ZnO nanowires films is considerably higher because of the enlarged surface area for dye adsorption on the cylindrical structure, thus increasing dye loading for improved light harvesting efficiencies and a much larger $J_{sc}$.

Although the photovoltaic performance of DSSCs hinges on multiple parameters, the importance of the electron transport electrode has been amply illustrated in this work. Equally crucial is the option on dyes. The conjugated bonds allow for electron displacing along the chain and the better likelihood for charges transferring, which promises good carrier conductivity, particularly for Zn(II) complex. As discussed above, the introduction of a zinc atom increases the ligand conformational rigidity, which alleviates the aggregation of the dye molecules because of the high sterically crowded structure, which suppress the recombination of injected electrons in DSSCs and reduce loss of conversion efficiency. Additionally, the conjugated Schiff bases are very stable (physically and chemically), resistant to oxidative attacks by their electron withdrawing structures [30]. At the same time, the dye structure and adsorption mode substantially impact the shift of energy levels of the conduction band of the semiconductor [31], consequentially, affecting the photovoltaic performance as well. The absorption of dyes can be achieved by two modes—physisorption or chemsorption. Hydrogen bonding is formed between an oxygen atom on the semiconductor oxide surface and a hydrogen atom of the dye’s carboxylic group to realize physisorption. The hydrogen atom of carboxylic group dissociates and the bond is formed between carboxylic oxygen atoms and the surface atoms on ZnO or TiO$_2$. The higher $V_{oc}$ on TiO$_2$ based cell is most likely caused by a relatively larger shift in energy level when Zn(II) complexes anchor on TiO$_2$. However, due to the much higher $J_{sc}$, the considerable efficiency is realized by ZnO NWs-based solar cells.

**Conclusions**

In this study, the high conductivity Zn complexes were developed and attempted to construct DSSCs as dye sensitizers, along with vertically aligned ZnO nanowires as an electron transporting layer for more dye load and better electron transporting pathways. The power conversion efficiency was examined by the I–V curve. When compared with the efficiency of (0.815%) of the solar devices based on conventional TiO$_2$ nanoparticles, the incorporation of ZnO nanowires in the solar cell significantly elevates the energy conversion efficiency to 3.68%. Zn(II) schiff-based complexes with their easy synthesis at low cost, along with the right option of semiconductor oxides, can potentially find widespread applications in dye sensitized solar cells devices with competitive efficiency.

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