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Photoelectrochromic cell with a CdS quantum dots/graphitic-nanoparticles sensitized anode and a molybdenum oxide cathode

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ABSTRACT

Low cost photoelectrochromic (PEC) cells with a cadmium sulfide (CdS) quantum dots/graphitic-nanoparticles (g-NPs) sensitized titanium oxide (TiO₂) film as the photoanode, an electrodeposited molybdenum oxide (MoO₃) film as the electrochromic counter electrode and an aqueous electrolyte were assembled for the first time. The oversimplified but surprisingly less used approach of incorporating g-NPs (10–50 nm in dimensions) in the photovoltaic film increases the light harvesting ability of the TiO₂/CdS film, by the virtue of their high electrical conductivity and a suitably poised work function at 4 eV, which maximized electron transfer and transport to the current collector. The proportion of the g-NPs in the film was optimized on the basis of solar cell performances, and the PEC cell with the following configuration: TiO₂/g-NPs (90 s)/CdS-S²⁻-MoO₃ delivered a power conversion efficiency (PCE) of 4.59% and a short circuit current density of ~17 mA cm⁻², which were 1.35 times higher than the values obtained for a similar cell with a TiO₂/CdS photoanode. Under 1 sun illumination and short-circuit conditions, the electrons from the photoanode reach MoO₃ via external circuit and it colors deep blue. The transmittance of the device underwent a change from ~58% to ~27% at 630 nm and bleached under dark. The transmission modulation varied between 24% and 31% over a wide wavelength range of 500–840 nm. The optical change was reversible, demonstrating that energy conversion and saving can be combined efficiently using an inexpensive cell, and thus it has the potential to function as a highly affordable self-powered window.

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1. Introduction

Photoelectrochromic (PEC) devices combine the functions of energy conversion and saving, and are capable of serving as self-powered windows that can color spontaneously upon exposure to sunlight, and bleach in its absence. In a PEC cell, a photo-absorber film and an electrochromic film function as the two electrodes, with an electrolyte sandwiched between the two layers [1]. Bechinger et al. [1], developed a PEC device based on a tungsten oxide (WO₃) electrochromic film in combination with a dye-sensitized semiconductor electrode that produced a photovoltage sufficient to color the electrochromic film. In another study [2], an organic photoelectrochromic film was developed. In the cell, the

same molecule, methylene blue, changed its' color from blue to transparent on absorption of light and reverted to the blue color upon oxidation. Most of the reports on PEC devices use a dye-sensitized TiO₂ layer to generate electrons, thereby creating the voltage required to drive the cations from the electrolyte into the electrochromic film (cathode) and color it [3–6]. When sunlight strikes a PEC device, the dye undergoes electron–hole separation, and the electrons are injected into the TiO₂ layer. The electrons are then transmitted through an external circuit to the electrochromic film at the counter electrode. Cations from the electrolyte then migrate to the electrochromic film for charge balance. The injection of cations and electrons into the electrochromic film causes it to color. When sunlight is blocked, the charge stored in the electrochromic layer steers the process in reverse, ejecting the cations from the electrochromic film and causing it to bleach. As a consequence, under no external voltage or current, the device will color upon exposure to sunlight and bleach, when it is dark [1,7].

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In PEC devices, there are reports on the use of nematic liquid crystals [8], a viologen salt [9], organic compounds like methylene blue [10], conducting polymers [4,5,11], and WO_3 [12–14], as electrochromes for the cells. To the best of our knowledge, there are no reports on PEC devices wherein a quantum dots (QDs) sensitized oxide film has been employed as the photovoltaic layer. Similarly, while electrochromism of molybdenum oxide (MoO_3) films is well-reported [15–18], until now, MoO_3 has not been used in a PEC device. Compared to the PEC cells developed in the past, wherein an extremely costly ruthenium dye is in the photoanode, and expensive-salts, ionic liquids, organic solvents and additives are used in the electrolyte, here the CdS QDs and the graphitic-nanoparticles (g-NPs), used in the photoanode, are extremely cheap (for they are prepared from Cd^{2+} and S^{2-} salts, and starch), and so is the aqueous electrolyte, which has only two components: water and a Na_2S salt. This is therefore one among the first examples of a working low cost PEC cell, wherein the photoanode was optimized on the basis of its' performance, prior to cell assembly. Currently, optimization of parameters plays a key role in improving the functionality of a device or an algorithm for a given application [19–22]. In photovoltaic and electrochromic literature, there are no reports on the fabrication and characterization of MoO_3 based PEC cells. MoO_3 , in a manner similar to WO_3 , upon electrochemical reduction, shows a strong blue coloration induced by the intervalence charge transfer between Mo^{5+} and Mo^{6+} states. Further, MoO_3 shows a stronger and more uniform absorption of light in its' colored state and also shows an excellent open-circuit memory [23]. It has also been reported that MoO_3 delivers a greater apparent coloration efficiency since the wavelength span of the absorption band of molybdenum bronze is close to the wavelengths to which human eye is most sensitive [23]. In a PEC cell with a QDs sensitized TiO_2 layer as the photoanode, and an electrochromic MoO_3 layer as the cathode or counter electrode, the mechanism of MoO_3 coloration is expected to be similar to the coloration of an electrochromic layer by a dye sensitized photoanode. Here, under zero external control, and upon solar irradiance, the excitons produced in the QDs, induce MoO_3 coloration, just like the dye does in the above-described PEC cells. A QDs sensitized TiO_2 layer has some advantages over a dye sensitized TiO_2 layer. CdS QDs are very easy to process in the form of thin films at room temperature, under ambient conditions via solution phase methods, and without invoking the use of any expensive equipment or inert conditions. QDs offer the benefit of multiple exciton generation, i.e., the ability to produce more than one electron per impinging photon; a phenomenon which dyes cannot undergo [24]. For TiO_2/CdS based quantum dot solar cells (QDSCs), the highest power conversion efficiencies (PCEs) obtained in photovoltaic literature are of the order of 1.5–2.8% [25–27].

To improve the PCEs of QD sensitized photoanodes, several approaches have been used by researchers. These include: integrating plasmonic metal (e.g. Au) nanostructures to induce near field enhancement [28], or incorporating carbon nanostructures such as multiwalled carbon nanotubes (MWCNTs) [29], single walled carbon nanotubes (SWCNTs) [30], reduced graphene oxide (RGO) [31], which serve as electron conduits and facilitate electron transport through the photoanode or using co-sensitizers such as PbSe [32,33], or PbS [34,35], which harvest near infrared (NIR) wavelengths and enable broader spectral utilization. Combinations of these approaches have also been used. However, these approaches are complex, in terms of materials preparation and identification of compositions for optimum performance. Here, we used a TiO_2/CdS film, and incorporated electrically conductive g-NPs in the same to increase photocurrents and thus PCEs. The g-NPs are extremely easy to prepare, and their preparation requires no capital equipment. PEC cells with a $\text{TiO}_2/\text{g-NPs}/\text{CdS}$ film, which served as the photoanode and an electrodeposited MoO_3 film

which functioned as the electrochromic counter electrode were assembled. The transmission modulation of MoO_3 and a sandwich cell with MoO_3 , achieved under different intervals of solar irradiance, by a simple external connection with the $\text{TiO}_2/\text{g-NPs}/\text{CdS}$ photoanode film, and under no external bias or current, demonstrates the potential of a QDs sensitized film in driving redox reactions of a chromogenic electrode.

2. Experimental

2.1. Materials

Cadmium acetate ($\text{Cd}(\text{CH}_3\text{COO})_2$), sodium sulfide (Na_2S), starch, sulfuric acid, acetyl acetone, liquid ammonia and solvents: toluene, isopropyl alcohol and ethanol were obtained from Merck. Titanium tetrachloride (TiCl_4), Triton X-100, and MoO_3 powder were procured from Sigma-Aldrich. TiO_2 powder (P25) was a free gift from Evonik. Ultrapure water with a resistivity of $\sim 18.2 \text{ M}\Omega \text{ cm}$ was used as a solvent. Transparent conducting electrodes of $\text{SnO}_2:\text{F}$ coated glass (FTO, sheet resistance: $25\text{--}30 \Omega^{-2}$) were obtained from Pilkington and were washed in a soap solution, ethanol, acetone (1:1 v/v) and isopropyl alcohol, in that order before use.

2.2. g-NPs and photoanode preparation

Starch, the carbon source, was dissolved in an aqueous solution of 6 M H_2SO_4 (50 mL) to form a starch solution, which was placed in a round bottom flask and refluxed at 120°C for 12 h. The resulting black suspension was filtered and the black product obtained was washed repeatedly with ultrapure water, till the discarded water attained a neutral pH. The product was dried overnight and then annealed at 350°C for 3 h, and a dry solid black powder, labeled as graphitic-nanoparticles or g-NPs was obtained, which was stored in a vacuum desiccator.

A fine paste of TiO_2 was prepared from TiO_2 powder dispersed in a solution of acetyl acetone (1.5 mL), ultrapure water (8.5 mL) and Triton X-100 (20 mg). It was applied over the cleaned FTO coated glass substrates by doctor blading. The as-fabricated TiO_2 films were heated at 60°C for 30 min, followed by annealing at 450°C for another 30 min. The TiO_2 coated FTO substrate was lastly immersed in an aqueous TiCl_4 (40 mM) solution for 30 min at 70°C and the resulting TiO_2 film was washed in double distilled water and annealed at 450°C for 30 min. 5 wt% of g-NPs were dispersed in ethanol, by ultrasonication for 15 min, and two electrodes: (i) a TiO_2/FTO plate or a blank FTO plate and (ii) a Pt rod were immersed in the black dispersion. A constant dc potential of 50 V was across the two electrodes, placed 0.5 cm apart, using a Tarsons electrophoresis power supply unit MC-01. The voltage was applied for three different time spans of 60, 90 and 120 s, and the g-NPs deposited on the TiO_2 or the FTO plate. The resulting films were labeled as $\text{TiO}_2/\text{g-NPs}$ (x s) or g-NPs films, where x=60 or 90 or 120 s. CdS QDs were deposited by a successive ionic layer adsorption and reaction (SILAR) process. In a glass beaker, a 0.1 M cadmium acetate solution in methanol and in another one, a 0.1 M sodium sulfide solution in methanol, were taken. The $\text{TiO}_2/\text{g-NPs}$ (x s) or the pristine TiO_2 films were initially immersed in the Cd^{2+} salt solution for 2 min then rinsed in methanol to remove superfluous ions, then dried at 60°C in a hot air oven. The same films were immersed in the S^{2-} salt solution for 2 min, and then rinsed in the solvent, and this is designated as 1 SILAR cycle. The plate was subjected to 3 more SILAR cycles and the resulting film is labeled as a TiO_2/CdS or $\text{aTiO}_2/\text{g-NPs}$ (x s)/CdS film (x=60 or 90 or 120 s). The films were maintained at 45°C under dark conditions for 48 h. The films were stored under dark at 45°C for 5 days prior to photovoltaic measurements. CdS films

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