

# Reversibly Electroswitched Quantum Dot Luminescence in Aqueous Solution

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**D**esign of stimuli-responsive materials, which are reversible in response to various external stimuli through their specific properties, has been an attractive topic among scientists in recent years.<sup>1</sup> Particularly, developing luminescence switches in nanostructures draws increasing attention because of their amazing potential applications in optoelectronic and memory materials.<sup>2,3</sup> A large number of luminescence modulations based on light-controlled nano-hybrids have been designed and studied.<sup>4–6</sup> With functionalized photochromic organic molecules or assembled inorganic polyoxometalate, the luminescent intensity of these systems would be reversibly regulated *via* energy transfer or electron transfer effect. Until now, very few electricity-dependent luminescence-controllable systems have been carried out,<sup>7</sup> though these electric luminescence switches were predicted to have important applications in fields of electro-optical devices and information displays.<sup>8</sup>

Colloidal luminescent semiconductor QDs have emerged as one of the most important classes of luminescence materials for over two decades. Due to the high photo- and chemical stability and high quantum yield as compared to traditional organic dyes, the QD nanomaterials represented a revolution in the application of fluorophores in luminescence system design.<sup>9–13</sup> Besides, these unique optical properties from QDs are also proved charge tunable.<sup>14,15</sup> Although much effort on expanding the application of this distinctive electrochromic character has been made,<sup>16–19</sup> the demanded condition in realizing the potential direct switching luminescence operations was still very rigorous. It requested both the absolutely anhydrous organic solvent and quite negative applying potentials (*ca.*  $-1.2$  V). Therefore, to overcome these

**ABSTRACT** In this study, we fabricated a novel hybrid film system, in which reversible electroswitching quantum dot (QD) luminescence was realized in aqueous solution for the first time. On the basis of an electrochromic material, poly(methylene blue) (PMB), QD luminescence could be switched effectively in a narrow potential range of  $-0.4$  to  $0$  V *via* the corresponding luminescence quenching effect. The luminescence switching operation was reversible and reproducible, and no noticeable changes in both “on” and “off” luminescence intensities were observed in 20 cycles. This simple system not only effectively overcame the harsh operation environment that generally existed in previous reports but also provided an easy method for the design and fabrication of other novel QD electroswitchable hybrid components.

**KEYWORDS:** quantum dots · poly(methylene blue) · electroswitch · luminescence · electrochromic

bottlenecks, we were interested to see if reversible luminescence electroswitching can be carried out easily by introducing another electrochromic species. Like other light stimuli-responsive systems,<sup>4–6</sup> the conversion of absorbance from the additional electrochromic component is expected to be capable of modulating the emission of QDs effectively. Herein, a typical electrochromic material, poly(methylene blue) (PMB), was adopted, and by using the CdTe QDs and PMB composed hybrid system, for the first time, reversible electroswitching of QD luminescence was achieved in aqueous solution by modulating the potentials between  $-0.4$  and  $0$  V.

## RESULTS AND DISCUSSION

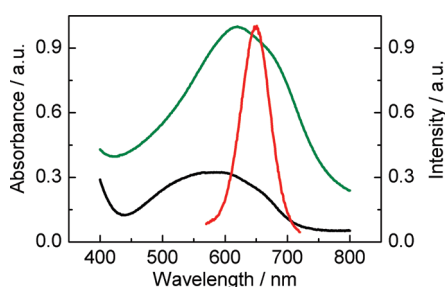
In this study, PMB is selected as a prominent candidate for the additional electrochromic species based on the following considerations: First, the absorption band of PMB oxidation states overlaps greatly with the emission band of QDs (Figure 1), which is particularly favorable for the occurrence of highly efficient energy transfer that would ensure the successful luminescence switching in our system. Second, PMB exhibits

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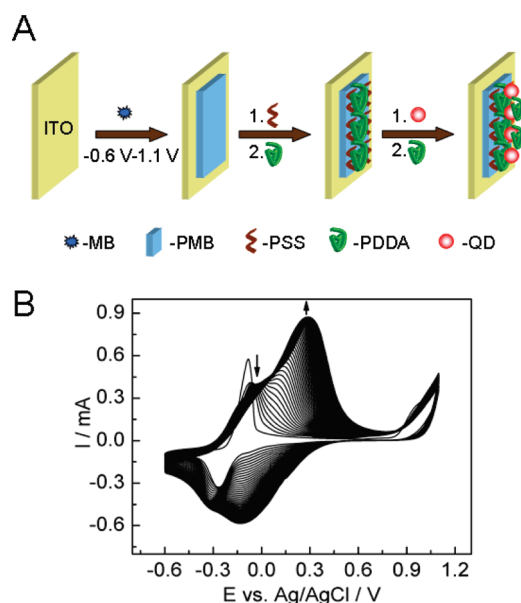


**Figure 1.** Absorption spectra of PMB film on ITO electrode under open circuit (green line) and at applied potential of  $-0.4$  V (black line) and fluorescence emission spectra of QD film (red line,  $\lambda_{\text{ex}} = 390$  nm) in 0.1 M phosphate buffer solution (PBS, pH = 7).

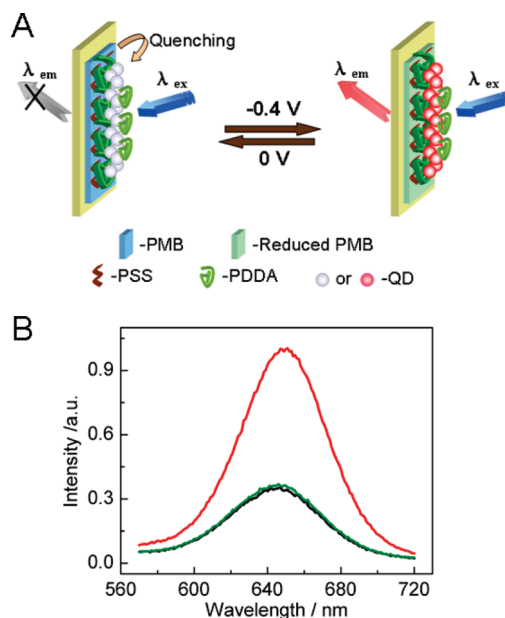
excellent reversible electrochromic behaviors in the QDs' suitable potential range ( $-0.8$  to  $0$  V). According to our previous study, applying the prescribed potential ( $-0.8$  to  $0$  V) on the CdTe QD films would pose negligible influence on their luminescence in  $\text{N}_2$ -saturated aqueous solution.<sup>20</sup> In the present experiment, in order to prevent oxidation of QDs at a positive potential as well as to effectively implement oxidation–reduction reaction of PMB,  $0$  and  $-0.4$  V were chosen as the oxidation (“off”) and reduction (“on”) potential, respectively. The contrast absorbance ratio of PMB at  $0$  and  $-0.4$  V was 3.3, indicating a good contrast between the two states of PMB that was favorable for switching the luminescence of QDs.

The scheme of fabricating PMB and QD hybrid films is shown in Figure 2A. Briefly, PMB film was first prepared on the indium tin oxide (ITO) electrode by the traditional electropolymerization method.<sup>21</sup> The cyclic voltammograms (CVs) of PMB growth is depicted in Figure 2B. The peak current at  $-0.08$  V obtained for monomer oxidation of the methylene blue (MB) molecule clearly decreased, and a pair of new redox peaks at  $0.3$  V appeared, with the peak currents increasing in cyclic potential scanning, suggesting the successful formation of PMB on the electrode surface.<sup>22</sup> After that, the synthesized CdTe QDs were spin-coated on the surface of PMB by using polystyrene sulfonate (PSS) and polydiallyldimethylammonium chloride (PDDA) as the cross-linker. Finally, a hybrid thin film composed of PMB/PSS/PDDA/QDs/PDDA was constructed for the following study. The thickness of the prepared film determined by Dektak 6 M stylus profiler was estimated to be *ca.* 23–31 nm.

The representation of the luminescence switching process is illustrated in Figure 3A. Note that no obvious luminescence variation was observed when applying negative potential on single PDDA/QDs/PDDA films (Figure S1 in the Supporting Information). However, when QDs incorporated with PMB in the hybrid film, the QD luminescence is endowed with the “on–off” function. As shown in Figure 3B, in the open circuit, the as-prepared hybrid films possess a broad absorption band in the range of 500–750 nm. At this time, the QD

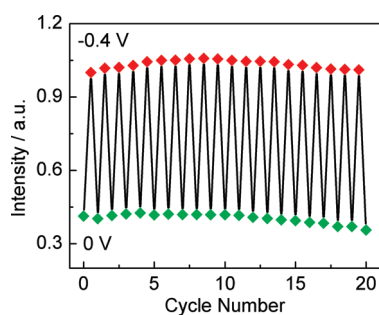


**Figure 2.** (A) Scheme of preparation of PMB/PSS/PDDA/QDs/PDDA hybrid films on the ITO electrode. (B) CVs of electropolymerization PMB in 0.05 M PBS (pH = 7), 0.5 mM MB, potential range of  $-0.6$  and  $1.10$  V,  $\nu = 0.1$  V/s.



**Figure 3.** (A) Schematic representation of luminescence modulation by applied potential on the PMB/PSS/PDDA/QDs/PDDA hybrid films. In the open circuit, the QD emission is reduced owing to the quenching effect from PMB. When a more negative potential was applied, the electrochromic PMB was converted to its reduced form, which then recovers the QD emission. (B) Luminescence switching of the hybrid thin film from open circuit (green line), to applied a potential of  $-0.4$  V (red line), to an applied oxidized potential of  $0$  V (black line). The experiments were performed in  $\text{N}_2$ -saturated 0.1 M phosphate buffer solution (PBS, pH = 7) ( $\lambda_{\text{ex}} = 390$  nm).

emission was weak due to the luminescence quenching by PMB possibly *via* energy transfer and inner filter effect (Figure 3B, green line). After a reduced potential

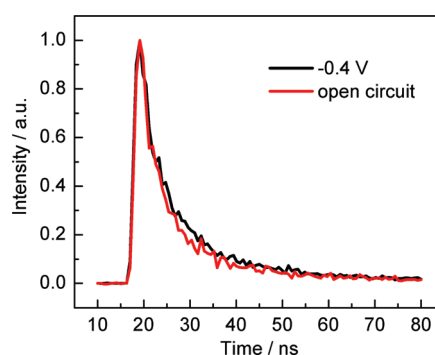


**Figure 4.** Normalized luminescence intensity of hybrid film in the double-potential cycles as a function of cycle number at an applied potential of 0 and  $-0.4$  V. The experiments were performed in  $N_2$ -saturated 0.1 M PBS (pH = 7) ( $\lambda_{\text{ex}} = 390$  nm).

of  $-0.4$  V was applied, PMB was converted to its reduced form. The changes in  $\pi$ -electron conjugation length in the molecules induced the distinct decrease of PMB absorbance,<sup>23</sup> and its original blue color turned rather transparent. Correspondingly, the luminescence from the hybrid film was increased obviously following the change of the absorbance (Figure 3B, red line). While the PMB film was reoxidized by further applying a potential of 0 V, the luminescence intensity would be almost completely recovered to its original state (Figure 3B, black line). The above spectral response to the potential was observed to be quite rapid. Both absorbance and luminescence intensity was steady within 30 s after applying the potentials (Figure S2 in the Supporting Information). The relative short circular duration (60 s) greatly improved the respond time of the switching operation, while it usually took more than 10 min to restore the original QD luminescence state in previous reports.<sup>14,15</sup>

Furthermore, the stability and reversibility of the switching operation were also evaluated by repeating reduction of the hybrid film and its subsequent oxidation numerous times. As can be seen from Figure 4, the luminescence switching operation was quite reversible and reproducible, and no noticeable changes in both "on" and "off" luminescence intensities were observed in 20 cycles. Such excellent fatigue resistance of the luminescence switching was thought to be ascribed to the good photostability possessed by both QDs and PMB (Figure S3 in the Supporting Information), while the tiny decrease of the luminescence intensity (*ca.* 8%) might be due to slight QD corrosion after the long-term electroswitching operations. In addition, the contrast luminescence ratio of the system at  $-0.4$  and 0 V was calculated to be about 2.7. This crucial value in the current work is proportionate to the corresponding contrast absorbance ratio because of the quenching mechanism. More efficient switching operation is expected to be achieved by engineering other more intertwined electrochromic materials and suitable QDs together.

In the present system, potential-induced absorbance change of PMB was considered to be a key factor for



**Figure 5.** Fluorescence decay of the hybrid film as a function of time. The red line represents the fluorescence decay under open circuit, while the black line is for the fluorescence decay after applying the potential of  $-0.4$  V.

the luminescence switching process. While the absorption band of PMB overlaps greatly with the emission band of QDs, fluorescence quenching could happen *via* either inner filter effect (IFE)<sup>24,25</sup> or energy transfer effect between PMB and QDs.<sup>26,27</sup> The interdistance between PMB and QDs was estimated to be about 4–7 nm, which just fell into the effective distance for the energy transfer effect.<sup>28,29</sup> Furthermore, the IFE was also expected to be responsible for the quenching while the emitted light from QDs travels through the PMB film to the detector. In the open circuit, the oxidized state of PMB possesses strong absorbance that could effectively absorb and block the emission coming from the front QD layer, as well as lead to effective energy transfer from QDs to PMB. As a result, the detected emission intensity was rather weak. Upon applying a reduced potential to PMB, the absorption of the hybrid films would decrease, which then weaken both the energy transfer and IFE process between PMB and QDs. Correspondingly, the concomitant detected luminescence intensity was increased dramatically. Once applying another oxidized potential, both the absorbance and luminescence intensity would be restored to the original states. Furthermore, the fluorescence decay dynamics of QDs in the hybrid films under different applying potential were also performed. As shown in Figure 5, the average fluorescence lifetime of QDs in the hybrid films under open circuit and applied potential of  $-0.4$  V were found to be  $5.51 \pm 0.164$  and  $6.57 \pm 0.126$  ns, respectively. The change of the fluorescence lifetime further suggested the contribution of the nonradiative energy transfer mechanism to the luminescence switching process.<sup>30</sup>

## CONCLUSIONS

In summary, we have successfully realized electroswitching QD luminescence in aqueous solution with the assistance of the electrochromic dye, PMB. Through modulating the luminescence quenching effect, QD luminescence could be effectively tuned at

the “redox” potential of PMB in the hybrid films. The switching operation exhibits good reversibility, and no noticeable change was obtained even after 20 cycles. Hopefully, by adopting a much more electrical fatigue resistance of QDs, a better long-term stability could be

achieved in the future work. We anticipate that the present work would offer important hints and a basis for the promising applications of QDs in the field of photoelectric devices such as electrically pumped lasers, optical displays, memories, and smart windows.

## METHODS

**Chemicals.** Mercaptosuccinic acid (MSA) was purchased from Aldrich. Sodium borohydride was purchased from Acros. Na<sub>2</sub>TeO<sub>3</sub> was bought from Schering-Kahlbaum in Germany. Cadmium chloride, trisodium citrate dehydrate, and methylene blue were bought from Beijing Reagent Company. Polydiallyldimethylammonium chloride (PDDA, 30 wt % in water,  $M_w = 200\,000$ ) and polystyrene sulfonate (PSS) were obtained from Sigma. All other chemicals were of analytical grade. All aqueous solutions were prepared with ultrapure water (>18 M $\Omega$ ). Phosphate buffer solution (PBS) containing 0.1 M Na<sub>2</sub>HPO<sub>4</sub> and 0.1 M NaH<sub>2</sub>PO<sub>4</sub> (pH = 7.0) was used as the electrolyte throughout the experiments.

**Instrumentation.** Electrochemical experiments were conducted with a CHI832 electrochemical workstation (Shanghai Chenhua Instrument Corporation, China). Luminescence measurements were carried out on a Fluoromax-4 spectrofluorometer (Horiba Jobin Yvon Inc., France) with excitation and emission slit widths of 2.5 nm. Absorption measurements were performed on a Cary 500 UV–vis–NIR spectrometer (Varian). The thickness of the film was determined by Dektak 6 M stylus profiler. Photoluminescence decay was measured on a Photon Technology International (PTI) Time master fluorescence lifetime spectrometer equipped with GL-302 dye laser pumped by PTI GL-3300 nitrogen laser and a GL-303 frequency doubler. The ITO plates (surface resistance of 30–60  $\Omega/\text{cm}^2$ ) with a geometric area of  $\sim 1\text{ cm} \times 5\text{ cm}$  were purchased from Nanbo Display Technology Co., Ltd. (Shenzhen, China).

**Preparation of PMB Film on the ITO Electrode.** Before modification, the ITO chips were washed with acetone, ethanol, and water in an ultrasonic bath sequentially. Then ITO chips were immersed in a solution of 1:1 (v/v) ethanol/NaOH (1 M) for 15 min to activate the surface. After being rinsed with pure water and dried under N<sub>2</sub> flow, in  $5 \times 10^{-4}$  M MB in 0.05 M PBS (pH = 7), the ITO electrodes were electropolymerized at 0.1 V/s in the potential range of  $-0.60$  to  $1.10$  V for 100 cycles to form a PMB film structure. Before being using, the PMB–ITO electrode was thoroughly rinsed with water to remove the physically adsorbed MB.

**Preparation of PMB/PSS/PDDA/QDs/PDDA Films on the ITO Electrode.** Stable water-soluble CdTe QDs were prepared as described in a previous paper.<sup>31</sup> The typical absorption and luminescence spectra of the synthesized CdTe QD aqueous solution are shown in Figure S4 in the Supporting Information. A particle size of 3.7 nm is estimated from the spectra, based on the first absorption peak wavelength.<sup>32,33</sup> Then, 50  $\mu\text{L}$  of 0.5 mg/mL PSS solution, 0.5 mg/mL PDDA, and 2  $\mu\text{M}$  CdTe QDs were pipetted and dropped on the modified ITO chips followed by spin-coating at 2700 rpm for 30 s. Herein, the relative luminescence intensity of the hybrid films could be controlled by adding an amount of QDs, while the QD content should be sufficient to keep away from nonrecoverable luminescence that is caused by the absorption of incomplete reduction of PMB. Figure S5 recorded the change of absorbance during the spin-coating operation.

**Spectroelectrochemical Experiments.** Spectroelectrochemical measurements (*in situ* fluorescence) were carried out in a modified fluorescence cell according to the previous report (1 cm length quartz cell) at room temperature.<sup>34</sup> The cell was capped with a Teflon plate, which was also served as the electrode support. ITO electrode, platinum wire, and Ag/AgCl (saturated KCl) were used as the working electrode, the counter electrode, and the reference electrode, respectively. Details of

the device are displayed in Figure S6 of the Supporting Information. Figure S7 shows UV–vis absorption spectra and luminescence spectra of the hybrid film as a function of applying different potential. Unless otherwise stated, the obtained UV–vis absorption and luminescence spectra results were acquired 30 s after the potential step to the films.

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**Supporting Information Available:** Additional information as noted in the text. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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