Physical approaches to tuning the luminescence color patterns of colloidal quantum dots

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Abstract. Localized surface plasmon resonance (LSPR) and photoactivation (PA) effects are combined for the tuning of fluorescent colors of colloidal CdSe quantum dots (QDs). It is found that LSPR with QD emitters intensely enhances surface state emission, accompanied by a remarkable red-shift of fluorescent colors, while PA treatment with colloidal QDs leads to a distinct enhancement of band-edge emission, accompanied by a peak blue-shift. Furthermore, the LSPR effect on QD emitters can be continuously tuned by the PA process. The combination of the post-synthetic approaches allows feasible realization of multi-color patterns from one batch of QDs and the approaches can also be compatible with other micro-fabrication technologies of QD embossed fluorescent patterns, which undoubtedly provides a way of precisely tuning the colors of light-emitting materials and devices that use colloidal QDs.
1. Introduction

Colloidal semiconductor quantum dots (QDs) as fluorescent materials have attracted much attention in optoelectronics, biotechnology and other basic research fields because of small sizes and high quantum yields (QYs). The large surface-to-volume ratio of colloidal QDs makes the surface defects greatly impact the optoelectronic properties. To improve QYs of the colloidal QDs, much effort has been made to suppress the surface states [1]. In contrast, the surface states of colloidal QDs could potentially be applied in white light-emitting diodes (LEDs) because their radiative surface state emissions (SSEs) are just in the visible light region and display variable broad emission bands [2–6]. These QDs are becoming competitive candidates for the next-generation white LEDs. Colloidal CdSe QDs are bright fluorescent emitters and have been widely used. There are two main luminescence peaks in the visible range for these QDs: the band-edge emission and the SSE. As the fluorescent colors of the emitters are determined by a superposition of the two peaks, different fluorescent colors can be obtained from one group of such QDs through modulating the wavelength and the intensity ratio of the two peaks. Baker and Kamat tuned the SSE peak and the intensity ratio of the band edge/SSE of CdSe QDs, via altering the 3-mecaptopropionic acid concentration, thereby obtained different colors [6]. But these chemical methods are only able to change the fluorescent color of a whole bottle of colloidal QDs rather than selectively changing colors in the designated area.

Localized surface plasmon resonance (LSPR) with QDs emitters could effectively enhance luminescence intensity [7, 8]. However, the colors of light emission via LSPR were primarily the same or close to those without LSPR such that the fluorescent patterns usually show high brightness contrast rather than high hue contrast [9, 10]. It has been demonstrated that the ultraviolet (UV) photoactivation (PA) process could effectively passivate the CdSe QDs surface and remarkably enhance the band-edge emission [11–13], which indicates that PA could change the fluorescent colors of the CdSe QDs continuously toward ones that are dominated by the band-edge emission. In this paper, the LSPR and PA are combined to realize multi-color patterns with both high brightness contrast and high hue contrast. It is found that oleic acid (OA) on the surface of CdSe QDs plays an important role in the formation of Ag nanostructures that show a...
strong coupling of localized surface plasmon (LSP) with QDs emitters. The physical approaches enhance the brightness contrast via increasing the luminescence efficiency, and modulate the hue contrast via changing the intensity ratios of the two emission peaks of QDs. Most important of all is that LSP on metallic nanostructures coupling with QD emitters can be continuously tuned by UV irradiation. The fundamental operations could be either individually used or combined to tune the luminescent colors of the QDs elaborately at designated areas and realize multicolor patterns.

2. Experimental methods

2.1. Synthesis of CdSe quantum dots (QDs) capped with oleic acid

In this study, two typical types of CdSe QDs (types #1 and #2) that were capped with OA were synthesized. #1CdSe and #2CdSe were used for PA processing and photoluminescence (PL) characterization and #1CdSe for coupling of QDs with Ag particles. Cadmium oxide (CdO; 99.95%), selenium powder (Se; 99.999%), OA (90%), 1-octadecane (ODE; 90%), tri-n-octyl phosphine (TOP; 90%) and polymethylmethacrylate (PMMA; average MW 350 000) powders, which were purchased from Alfa Aesar. Toluene, ethanol and acetone were used as received.

The synthesis route of type #2 CdSe QDs with rich SSE is described as follows. A mixture of 0.0775 g CdO powder (0.6 mmol), 3 ml OA and 30 ml ODE in a 50 ml three-neck flask was heated at 150 °C under Ar gas flow for 30 min to remove the air and water. Then the temperature was elevated to 300 °C and a transparent Cd precursor solution was formed. Then 0.5 ml selenium precursor solution TOPSe (containing 0.6 mmol selenium) was rapidly injected into the flask at 230 °C, while the Cd precursor solution was stirred by a magnetron vigorously. When the color turned red, the flask was cooled by a water bath to stop the growth of the CdSe QDs. After being cooled to room temperature, the ODE phase was mixed with an equal volume of hexane/methanol for extracting the unreacted precursor. Then CdSe QDs in ODE phase were precipitated with three times the volume of ethanol. Our experiments show that the lower injection temperature and concentration of the precursor solution will promote the SSE as well. Type #1 CdSe QDs that present weak SSE were prepared via increasing the concentration of Cd precursor and the Se/Cd ratio; that is, 4 mmol CdO, 8 ml OA, 30 ml ODE and 3 ml TOPSe (containing 6 mmol selenium) were used in the synthesis.

2.2. Preparation of polymethylmethacrylate–QDs films and Ag–QDs coupling systems

The synthesized CdSe precipitates were dispersed in toluene and then the QDs/toluene solution was added with PMMA powders in a weight ratio of 30:1. The mixture was placed in a dark ambient for days to dissolve the PMMA powders. Si wafers were cleaned in acetone, ethanol and deionized water in order by a sonication bath, followed by drying with a nitrogen jet. PMMA–QDs/toluene solution was coated on the Si wafers by a spin coater at a speed between 1500 and 3000 rpm. Consequently, uniform and smooth PMMA–QDs films were prepared.

The preparation of QDs–Ag coupling systems was as follows. Half the area of a Si wafer was coated with 60 nm Ag film by thermal evaporation. The thickness was controlled by a crystal monitor. The CdSe QDs/toluene solution was spin-coated on the wafer with half-coated Ag films. Each wafer was cleaved to two pieces along the orientation perpendicular to the boundary between the area with Ag and the area without Ag. Then one of the pieces was kept
Figure 1. PL spectra of the neat QDs (types #1 and #2) and their PMMA–QDs blends.

as a reference sample, while the other one was annealed in air at 150 °C in an oven for 30 min. PL spectra were measured separately on the areas with and without Ag for all the samples.

2.3. Photoactivation (PA) processing and measurements of photoluminescence spectra and quantum efficiency

In the PA processing and PL measurements, an excitation laser beam (λ = 325 nm) was used as both an excitation line for PL and a UV light source for PA. The laser beam just stayed at the same area of the sample surface and recorded PL intensity for an interval. PA processing for fabrication of color patterns was performed by UV light irradiation for different periods using a 20 W UV lamp (central wavelength λ = 365 nm). PL measurements were carried out using an Edinburgh FLS920 PL system. Photographs of the color luminescence patterns were taken by a digital camera under the UV spot of a xenon lamp at 325 nm. The absolute QYs of the two groups of PMMA and QD blends were measured by the FluoroMax-4, HORIBA. The QYs of the UV-irradiated samples, annealing-processed samples and the QDs–Ag coupling structures were obtained by quantitative comparison of the integrated areas of the PL spectra to the reference spectra area.

3. PA effect for tuning the emission of CdSe QDs

Figure 1 shows the PL spectra of the neat QDs films and PMMA–QDs blending films with different QDs (types #1 and #2). The two batches of PMMA–QDs exhibit different PL intensity ratios of the band edge/SSE owing to different synthetic parameters described above. #1QDs samples show a dominant band-edge emission peak (562 nm) with a weak shoulder at about 650 nm which originated from optical transition related to surface states. In contrast, #2QDs samples show a weak band-edge emission peak with a strong SSE band at about 700 nm. The observed differences in peak intensities and wavelengths of SSE can be explained by the different densities of surface states involved in the two types of QDs. Difference in densities of surface states arose from different Cd precursor concentrations in the synthesis of QDs,
i.e. #1QDs had a higher Cd concentration than #2QDs. It is known that when the density of defect states is low, their energy levels are discrete lying in the band gap of CdSe. As the density of surface states increases, the discrete energy levels expand into a band in the band gap; consequently, a longer wavelength SSE is observed in #2QDs. Although the neat QDs films and PMMA/QDs blending films display basically the same luminescent characters for each group of QDs, after forming thin films, however, PMMA–QDs present more stable mechanical characters with smooth and hard surfaces, which is vital for subsequent device processing and storage.

Figure 2 shows the PL spectra corresponding to the PA processed (UV irradiations) PMMA–QDs blending thin films made of type #1 and #2 QDs. The PL spectra for the as-deposited films are also plotted by black lines in figures 2(a) and (c). Both samples display band-edge emission blue-shifts after PA treatment in air ambient. The blue-shifts originated from photo-assisted oxidation of QDs surfaces, which generates Cd–OH and SeO$_2$ layers on the QDs surfaces. The oxide layers formed by the photochemistry process can effectively reduce the size of QDs and isolate the connection of QDs, which leads to an apparent blue-shift and intensity enhancement in the PL spectra [13].

As the fluorescent colors are determined by the spectral characteristics in the visible light range, the color of the type #1 QDs is green, which is dominated by the band-edge emission, whereas the second one is orange as determined by both the band edge and SSE. Figures 2(b) and (d) plot the PL intensity variations of band-edge emission and SSE versus UV irradiation times. The QYs versus UV irradiation times are shown as well. There are obvious similarities between the two samples: as irradiation time is prolonged, the band-edge emission intensities first rise and afterwards fall down, accompanied by peak blue-shifts. The change of QYs for the two groups of QDs after PA processing is consistent with the band-edge emission. For #1QDs, the QYs rise to 7.2% in 90 min and then fall down slowly. For #2QDs, the QYs are much lower and decrease faster than #1QDs because higher density of surface states are involved in #2QDs. Figure 2(e) shows a schematic diagram of the PL intensity versus UV irradiation times, which is divided into three regions, I, II and III. High brightness contrast for color luminescence patterns can be realized by UV irradiation corresponding to point B, whereas point A (insufficient irradiation) or C (overexposure) corresponds to the dark ones.

To intuitively illustrate the modulation of brightness contrast for fluorescent patterns by PA, we chose type #1 QDs with weak SSE to fabricate periodic color patterns using a glass photo mask and irradiation of UV light (365 nm). The two insets in figure 2(e) show the photographs of fluorescent patterns after the UV irradiation. The dark area in the left color photograph corresponds to non-irradiation (region I), whereas the bright green patterns fall into region II. When a PMMA–QDs film was wholly irradiated by UV light for a suitable period falling into region II prior to the UV irradiation with the glass photo mask, the overexposed area (patterns) became darker than the surrounding region. In the right color photograph of figure 2(e) the light area corresponds to region II, whereas the dark patterns are the result of overexposure (region III). The tempered exposure and excessive exposure are similar to the positive and negative photoresist technology. More complex fluorescent patterns with multiple colors were fabricated through multi-pass exposure operations using a photo mask as shown in figure 3. Figure 3(a) illustrates schematic diagrams of the operation procedure with multiple exposures. Figures 3(b)–(d) are the fabricated fluorescent color patterns with double and triple irradiations. Evidently, the fluorescent patterns made with the PA effect by modulating the intensities and wavelengths of both band edge and SSE show high brightness contrast. However, it is found in figure 3 that the colors realized by PA are clearly not rich or in low hue contrast. The reason is
that the UV irradiation leads to similar PL intensity changes for both the band edge and SSE. To highlight hue contrast of the fluorescent patterns, as a next step we adjusted the PL intensity ratios of the two peaks via coupling QDs emitters with LSPs excited on Ag nanostructures.

4. Combination of localized surface plasmon resonance and PA

To demonstrate the changes of the luminescent characters of the QDs emitters via coupling with LSPs in metallic nanostructures, 60 nm Ag films were first deposited on a silicon wafer,
followed by spin coating of PMMA–QDs blends with #1QDs. The wafer was cleaved into three pieces along a direction perpendicular to the boundary between the areas coated with Ag film and without Ag film. One of the pieces was kept as a reference sample, while other two were annealed in air ambient at 150 °C in an oven for 30 min. Afterwards, one of the two annealed samples was irradiated by UV light for 6 h. The experimental details are shown in figure 4(a). Here, the annealing process is an essential step to realize the effective coupling of LSPs on Ag films with QDs. As demonstrated in [13], annealing treatment decreased PL intensities of both band edge and SSE, whereas PA processing enhanced (or recovered) the PL intensities. Therefore UV irradiation processing was carried out on one piece of the annealed samples.

The measured PL spectra for the reference and processed samples are shown in figures 4(b)–(d). By comparison of PL spectra of QDs samples without Ag as shown in figures 4(b) and (c), we find a band-edge emission red-shift (from 562 to 564 nm) after the annealing. When a sample was annealed the OA ligand on QDs desorbed from the QD surface. The loss of ligand made coalescence of the adjacent quantum dots, i.e. two or more QDs approaching or coming into contact with each other. Consequently, electron coupling between these QDs became possible, which is equivalent to the effective increase of QD size. This quantum size weakening effect leads to the red-shifts and quenching of the band-edge emissions [13].

As shown in figure 4(b), the PMMA–QDs with Ag film display a higher PL intensity than those without Ag film and the inset photograph also shows that the area with Ag is brighter than the area without Ag. The QY for the CdSe QDs on Ag film is about 3.5%, whereas the QY for the area without Ag is only 1.25%. As shown in 4(c), the annealing treatment of the area without Ag causes PL intensity decay accompanied by a band edge peak red-shift, and QY is lowered to 0.73%. A dramatic result is that the annealing treatment leads to a large enhancement of the SSE of the PMMA–QDs/Ag area in comparison with the reference area and the QY increases to 14.9%. Consequently, the inset photograph in figure 4(c) demonstrates vivid brightness and

Figure 3. (a) Schematic diagrams of the fluorescent patterns fabricated by multiple exposures; (b) and (c) the patterns produced with double irradiations; (d) the patterns produced with triple irradiations.
Figure 4. (a) Schematic diagrams of the fabrication of the PMMA–QDs on Ag nanostructures; (b) PL spectra for type #1 PMMA–QDs; (c) PL spectra for the annealed samples; (d) PL spectra for the annealed and then UV-irradiated samples. The QYs of the PMMA–QDs reference and Ag-coupled samples are labeled in (b)–(d).

hue contrast between the two areas. This phenomenon is assigned to the enhanced coupling effect of the fluorescent emission from QDs with LSPs on the Ag nanostructures because annealing at a proper temperature induced the modification of Ag morphology, which was catalyzed by the PMMA–QDs layer in the annealing process. The SSE at about 640 nm from the PMMA–QDs/Ag area is enhanced much more than band-edge emission, indicating that SSE is more effectively coupled with LSPs on Ag nanostructures than band-edge emission. Compared to a conventional Ag-island film, the modified Ag film adhering to the PMMA–QDs layer is favorable for enhancing the SSE of the CdSe QDs. This wavelength-selective enhancement is in agreement with the observation of ZnO–Ag and CdS–Ag nanostructures [14, 15]. Our further experiments indicate that the sizes of QDs, the thickness of the Ag films and the annealing temperature could slightly affect the SSE–SPs coupling.

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As shown in figure 4(d), after UV irradiation the band-edge emission intensities for both areas (with or without Ag) are distinctly enhanced in comparison with figure 4(c). The QYs for PMMA–QDs/Ag and without Ag are 21.0 and 5.8%, respectively. In the area without Ag, the color (green) was dominated by band-edge emission, whereas the color (orange) of the area with Ag was dominated by the enhanced SSE. It is thus clear that the LSP enhancement and PA effect can be combined for tuning the color of QDs.

Stability was further examined with both PMMA–CdSe QDs films and PMMA–CdSe QDs/Ag structures. Optical characterizations show that the as-coated PMMA–CdSe QDs films are stable over a one-year observation period, whereas the annealed PMMA–QDs/Ag structures present stable PL intensity over a seven-month observation period, which is shown in figure 5.

We make a comparison of the morphology of Ag films on PMMA–QDs layers and that on a Si wafer after annealing in order to explore the large luminescence enhancement. Figure 6 shows that after annealing the morphology of Ag on a PMMA–QDs layer is quite different from that on the surface of the Si wafer. The as-deposited Ag film was composed of tiny Ag particles with ∼30 nm size, as shown in figure 6(a). For the annealed Ag film on the PMMA–QDs layer shown in figure 6(b), crystallized polyhedron morphology of Ag was formed. In contrast, annealing the Ag films on Si formed the conventional Ag islands by tension of Ag in the air or vacuum. The polyhedron morphology of Ag was modified mainly by the residual OA in the PMMA–QDs layer because OA can influence the shape of nano-materials as a surfactant and an etchant [16, 18]. It is known that OA as a sort of ligand on CdSe QDs provides rich surface states. In the annealing process, as temperature rises, the OA ligand leaves CdSe QDs and moves to the Ag surface; consequently, it plays the role of catalyst on the Ag surface at annealing temperatures. As a surfactant, OA could restrain the Ag from aggregating in the conventional islands at the annealing temperature. As an etchant, OA could change the morphology of Ag film by selective etching in a small scale on the surface of Ag films. To verify this hypothesis of OA being a surfactant of Ag, we performed further verification tests by annealing a piece of Ag/Si sample in OA at 150 °C for 30 min. Then the sample was cleaned by toluene to remove the residual OA on the Ag surface and observed using a scanning electron microscope (SEM), shown in figure 6(b). Therefore, it is concluded that OA ligand on CdSe QDs plays a critical role in modifying Ag morphology and consequently impacts the coupling of QDs emitters with LPs on Ag nanostructures.
5. Multiple color luminescence patterns

To demonstrate the tuning of the luminescence colors at designated areas via coupling CdSe QDs emitters with LSPs, we fabricated the luminescence patterns by deposition of Ag on the selective areas with hollow masks, followed by thermal annealing and UV irradiation. Figure 7 shows the colorful fluorescence patterns after subsequent processing steps: spin coating of PMMA–QDs blends on the patterned Ag film (left column: figures 7(a), (d) and (g)), thermal annealing (middle column: figures 7(b), (e) and (h)) and UV irradiation (right column: figures 7(c), (f) and (i)). Evidently, color differences with a high hue contrast between the areas with and without Ag nanostructures are seen. For example, in figures 7(a)–(c), the color letters of ‘ZJU’ are the outcome of QDs sitting on Ag nanostructures, which is brighter than the background area without Ag coating. It is seen that after annealing the color of the letters changes from yellow to red, whereas the background becomes dark (the ratio of PL intensity 20). After UV irradiation, the letters change to orange color, while the background area becomes bright. The diffusion (or leakage) of Ag vapor to the area beyond the hollow ‘ZJU’ mask made a red blear shadow-like special effect.

Colloidal CdSe QDs with smaller sizes were used such that we could fabricate color patterns with shorter wavelength. Figure 7(d) shows vertical strip patterns in which smaller QDs were coated on selectively evaporated Ag nanostructures. Distinct color changes occur after annealing as shown in figure 7(c). The PMMA–QDs/Ag films were then selectively irradiated under the hollow strip mask that was aligned in the horizontal direction, which is perpendicular to Ag strips (a similar operation to figure 3(a)). Evidently, annealing and UV irradiation greatly highlight the brightness contrast and hue contrast of the color luminescence patterns.
**Figure 7.** Fluorescent photographs produced by patterned Ag deposition, annealing and UV irradiation. Left column (a, d, g): spin-coated PMMA–QDs blends on patterned Ag nanostructures; mid-column (b, e, h): after annealing; right column (c, f, i): after UV irradiation. (g) The pattern was deposited twice with Ag strips that were across each other; (h) after annealing; (i) the pattern was irradiated twice by UV light.

| As deposited | Annealing | Photoactivation |
|-------------|-----------|----------------|
| ![Image](image1) | ![Image](image2) | ![Image](image3) |
| ![Image](image4) | ![Image](image5) | ![Image](image6) |
| ![Image](image7) | ![Image](image8) | ![Image](image9) |

**Figure 8.** Schematic diagram to combine the effects of PA and LSPR for color tuning and the potential applications of the technology.

More complex color patterns can be realized by multiple evaporation of Ag films and multiple UV irradiations. Figures 7(g)–(i) illustrate that the colors of luminescence patterns can also be tuned by the variation of Ag film thickness. In figure 7(g) the vertical strips correspond to 30 nm Ag, while the horizontal ones correspond to 15 nm Ag. Clearly, the annealing process...

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greatly highlights the brightness contrast and hue contrast of the multiple color patterns shown in figure 7(h). In particular, as demonstrated in figure 7(i), double UV irradiations made the patterns colorful. Even more colorful patterns can be produced by the magic combination of the coupling of QDs emitters with LSPs on Ag nanostructures and UV irradiation.

6. Conclusions and perspectives

In conclusion, we have systematically studied physical approaches of PA and LSPR for modulating the band-edge emission and SSE and tuning the luminescence colors of CdSe QDs. PA treatment of PMMA–QDs blends results in the enhancement of luminescence intensity accompanied by blue-shift of the emissions. The coupling of LSPs on Ag nanostructures with QDs mainly enhances SSE and causes a remarkable red-shift of the colors. Most important of all is that the LSPs-enhanced PL spectra can be continuously tuned by UV irradiation. It is found that OA on the surface of CdSe QDs plays an important role in the formation of Ag nanostructures that show strong coupling of LSPs with QDs emitters. These fundamental approaches can be easily combined to attain multi-color patterns from only one batch of QDs. The methods presented here are also compatible with other micro-fabrication technologies of QDs embossed emission patterns, such as stamp mold [19, 20], e-beam lithography [21, 22] and flow coating [23]. The combination of PA, LSPR and annealing undoubtedly provides an effective way of precisely tuning the colors of light-emitting materials and devices that use colloidal CdSe QDs. Figure 8 shows perspectives on the potential applications of the combined methods.

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