Surface modification toward luminescent and stable silica-coated quantum dots color filter

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ABSTRACT A highly pixelated and luminescent silica-coated quantum dot color filter (QDCF) was achieved by surface conjugation with epoxy functional group. Epoxy-functionalized silica-coated quantum dots (QDs) can be thoroughly mixed with SU-8 photoresist up to 25 wt.% without aggregation. The quantum yield (QY) of the silica-coated QDCF can be significantly improved from 19.3% to 36.5% after epoxy treatment. The pristine QDCF experienced a 40% QY decrease, while the epoxied silica-coated QDCF maintained its luminescence even after irradiation (300 mW cm⁻² @450 nm) for over 25 days. The well-controlled epoxy cap plays a critical role in attaining the ideal optical properties of the QDCF.

Keywords: quantum dot, silica coating, color filter, ligand exchange

INTRODUCTION

The BT.2020 color gamut standard has become a major research goal for the display industry [1]. The discovery and development of alternative highly efficient emitting materials with narrow bandgaps in the red-green-blue (RGB) spectral region is important and challenging [2]. Quantum dots (QDs) have been applied as promising emitting materials to enhance the display color gamut due to their unique properties, such as tunable bandgap, adjustable size, and controlled emission wavelength [3–6]. Generally, there are four types of QD applications in displays (including liquid crystal display (LCD) and micro light emitting diode (Micro-LED)), as summarized in Fig. S1. RGB pixels are normally obtained using traditional absorptive color filters (CFs), wherein the proper mixture of red and green QDs is excited with a blue LED chip. To achieve this, QDs can be embedded in packaged

blue LED chips or tightly deposited on the surface of packaged chips (Fig. S1a and b); however, both these methods involve heat resistance issues. Ensuring an appropriate distance between the QD layers and packaged chips can overcome this problem (Fig. S1c and d). As shown in Fig. S1d, the traditional absorptive CFs are replaced with a wavelength-converting quantum dot color filter (QDCF), which can directly provide individual red and green pixels, with high color purity, efficient luminescence, wide-angle viewing, and a wide color gamut [3]. Therefore, the QDCF is critically important for newly emerging display techniques.

In preparing efficient QDCFs, fabricating proper QD substrates is a principal factor. Fully cured cross-linking photoresists exhibiting excellent mechanical properties [7] are a good choice for QD matrices. However, QDs normally possess different surface characteristics with a photoresist [8], resulting in significant phase segregation when directly mixed with a photoresist. This phase segregation dramatically hinders the application of QD/photoresist hybrids. Extensive research efforts have been devoted to dispersing inorganic QDs in organic polymers to improve performance. Electrolyte salts can stabilize colloidal nanocrystals in solvents [9,10]. However, the presence of electrolytes in the polymer will dramatically compromise its physical properties. Specific surface modification through capping agents was directly employed with QDs to enhance colloidal stability in the polymer [11–14]. However, manipulating native surface-capping ligands of QDs will quench the nanocrystal luminescence and thus decrease their shelf life [15]. Moreover, the QY of QDs is strongly affected by their dispersion in polymers, which is closely related to the

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surface trap states. Specifically, the cross-linking unit of the photoresist polymer is hydrolyzed (or degraded or broken) by an acid generated by a photoacid generator on the exposed region [16], which severely degrades the emission properties of QDs.

A common method for circumventing these issues is encapsulating the QDs with a hydrophilic silica shell, which is a basic strategy for protecting QDs from environmental degradation. The stability of the silica-coated QDs has been significantly improved in previous studies on CdSe-SiO₂, CdSe/ZnS-SiO₂, CdSe/CdS/ZnS-SiO₂, and ZnSe-SiO₂ [17–22]. Generally, silica coating has been reported to decrease the QY [23], but increased QYs were achieved by Selvan's group [24] using a reverse microemulsion technique. In this approach, QDs were exposed to a silica precursor (tetraethyl orthosilicate, TEOS) in the presence of a surfactant. Rapid ligand exchange of the native surfactants with TEOS occurs, which is a critical step for incorporating QDs in a reverse micelle that forms a starting point for silica growth [25]. Therefore, a silica shell can prevent leakage of toxic Cd²⁺, enhance the chemical stability of the QDs [26,27], and promote surface conjugation with the ligand [15]. Moreover, it was reported that a silica shell can prolong the shelf life of QD devices: after long-term operation for 100 h at a driving current of 60 mA, a silica QD-LED retained 88% of its initial QY, while a pristine QD-LED only retained 65% of its initial value [28].

In this work, we prepared silica-coated QDs by a reverse microemulsion technique and investigated the interaction dynamics of QDs in an SU-8 photoresist. To fabricate a stable QDCF with high pixelation and luminescence, surface passivation of the silica-coated QDs in the photoresist was conducted using (3-glycidyloxypropyl) trimethoxysilane. For comparison, different samples are denoted as QDs, pristine silica-coated QDs, and epoxy silica-coated QDs. Ligand exchange at the silica surface involves replacing native –OH ligands with epoxy functional groups, which is conducive to obtaining photoresist-soluble silica-coated QDs. Therefore, after epoxy modification, silica-coated QDs can be dispersed uniformly in the matrix and are expected to be applied as stable and luminescent QDCFs.

EXPERIMENTAL SECTION

Preparation of silica-coated QDs

Unless specified otherwise, chemicals were purchased from Sigma-Aldrich and used without further purification. CdSe/ZnS QDs (denoted as QDs) were incorporated

in silica spheres by a reverse microemulsion method; 10 g IGEPAL® CO-520 surfactant, 3 mL TEOS, 640 μ L QDs/methylbenzene (50 mg mL^{−1}), and 3 mL ammonium hydroxide were added to 160 mL cyclohexane with a constant stirring speed of 500 rpm. The final mixed solution was stirred continuously for 24 h in dark.

Synthesis of epoxied silica-coated QDs

Ammonia solution (30%) was added to an ethanol solution of silica QDs (4 mg mL^{−1}) at room temperature (RT) to reach specific pH values. A molar ratio of 1:1 (3-glycidyloxypropyl) trimethoxysilane in chloroform solution (10%) was added dropwise to the silica-coated QD suspension under vigorous stirring. After 24 h, the epoxied silica-coated QDs were separated by centrifugation, washed with ethanol, and dispersed in propylene glycol monomethyl ether acetate (PGMEA).

Fabrication of QDCF

To compare the effects of silica coating and surface passivation on the QDCF, we prepared CdSe/ZnS QDs, pristine silica-coated QDs, and epoxied silica-coated QDs. These three types of QD were mixed with SU-8 3050 photoresist at the same concentration. Spin coating of SU8/QDs in PGMEA (v:v 1:1) was performed at 500 rpm for 5 s and 3,000 rpm for 30 s. Soft baking was performed for 9 min at 120°C, followed by UV exposure at 365 nm for 15 s (300 mW cm^{−2}). Post-exposure baking was performed for 3 min at 100°C. The samples were developed by immersion in SU-8 developer for 5 min to remove the unexposed areas, followed by rinsing with isopropylamine and drying with nitrogen gas.

Characterization

Transmission electron microscopy (TEM) was conducted with a Tecnai F30 microscope. Fourier transform infrared (FTIR) spectroscopy was performed using KBr tablets with a Perkin Elmer Frontier spectrometer (4,000–750 cm^{−1}). Scanning electron microscopy (SEM) images were acquired with a field-emission scanning electron microscope (FE-SEM, JEOL JSM-7401F). Photoluminescence QY (PLQY) measurements were performed at RT with a C11347-12 (HAMAMATSU) absolute QY measurement system (excitation wavelength: 450 nm). The optical output power of the blue LED (AISU 2835) for photoaging was measured using an ATA-500 Spectral Radiation Analyzer (EVERFINE Corporation) with an integrating sphere. Dividing the optical power by the sample area, we obtained the optical power density of photoaging. All optical measurements were performed at

RT under ambient conditions.

RESULTS AND DISCUSSION

We successfully fabricated QDCF thin film with well-ordered patterns *via* a process involving a typical photolithography technique, as shown in Fig. 1, where the silica-coated QDs are employed in this work. PGMEA was the developing agent for the SU-8 photoresist. As shown in Fig. 1, the QD/photoresist mixture was spin-coated on the substrate, followed by UV exposure (365 nm, 15 s) after soft baking. The masks formed the well-ordered patterns, such as circular, triangular, and square shapes with the same dimensions and spacing. After development, the non-irradiated part was removed, and the designed patterns were clearly and completely preserved. To obtain cemented QDCF thin film, hard baking is an essential stage. QDs are extremely sensitive to chemicals and thermal treatment during photolithography. The most viable strategy for protecting QDs from degrading environments is to physically encase them with chemically stable oxide phases, and the most widely used oxide for QD encapsulation is silica [28,29]. It was reported that silica coating improved the colloidal, chemical, and photostability of QDs and reduced the penetration speed of moisture and oxygen into the emission materials [21,30,31]. Therefore, silica coating is expected to preserve the QY during photolithography, as well as resist QD photodegradation.

To relieve phase segregation, which usually occurs in QD/photoresist hybrids, surface modification is adopted in this work. As far as we know, SU-8 is an epoxy-based negative photoresist, which can be conjugated with epoxy group. For silica-coated QDs, the residual hydroxyl group provided by ammonium during silica coating is condensation-cross-linkable and can be employed in the epoxy-capping reaction. Therefore, a homogeneous QD/photoresist compound can be realized through surface decoration of the silica-coated QDs using epoxy ligands. Surface modification of the silica-coated QDs was performed using epoxy-containing silane, involving ligand

exchange on the QD surfaces, as shown in Fig. 2. Replacing native $-OH$ ligands with epoxy groups is conducive to obtaining photoresist-soluble silica-coated QDs.

To optimize the ligand exchange conditions, the effects of temperature and pH on the QY of silica-coated QDs were clarified in this work, as summarized in Table S1 (see Supplementary information). At RT (20°C), the QY of silica-coated QDs increases from 46.6% to 56.6% as pH increases from 7 to 8.5, while sharply drops to 42.9% as pH reaches 10. Moreover, the QY is affected by the reaction temperature; for example, the QY decreases from 56.6% to 51.7% as the temperature increases from 20 to 45°C at pH 8.5. By comparison, the condensation reaction with hydroxyl groups can be easily conducted in a slightly basic environment, and the optimum pH is ~ 8.5 .

After surface modification and ligand exchange at silica-coated QDs, FTIR spectra were recorded to examine the epoxy ligands, as shown in Fig. 3. The characteristic peak at $1,104\text{ cm}^{-1}$ is attributed to the $O-Si-O$ stretching vibration [32], indicating that silica coating was successful. Peaks are observed at $3,435\text{ cm}^{-1}$ for the $-OH$ vibration, $3,360\text{ cm}^{-1}$ for stretching [33,34], and $1,640\text{ cm}^{-1}$ for in-plane bending [35]. Ligand exchange was conducted in a basic environment (pH 8.5) with ethanol and ammonia water. Hence, the peak at $3,435\text{ cm}^{-1}$ is ascribed to the residual solvents (ethanol, ammonia water) in the pristine silica-coated QDs. SU-8 3000 photoresists use cyclopentanone as the primary solvent [35]; hence, the peak at $3,435\text{ cm}^{-1}$ for the $-OH$ group may be due to cyclopentanone. Both SU-8 and cyclopentanone contain $-CH_2-$ bonds ($2,920\text{ cm}^{-1}$). Therefore, the absorption peaks at $1,199\text{ cm}^{-1}$ for the epoxide group [36] and $2,920\text{ cm}^{-1}$ for $-CH_2-$ indicate the presence of epoxy groups on the surface of silica-coated QDs.

The morphologies of epoxied silica-coated QDs were studied *via* TEM, as shown in Fig. 4. The monodisperse silica-coated QDs are about 50 nm in size (Fig. 4a). As shown in Fig. 4b, for epoxied silica-coated QDCF samples, the CdSe/ZnS core is separated by silica coating;

Figure 1 Schematic diagram of QDCF fabrication.

Figure 2 Schematic diagram of the ligand exchange process.

Figure 3 FTIR spectra of pristine silica-coated QDs, epoxied silica-coated QDs and SU-8 resin.

hence, the optical properties of the QDs are retained (Table S2). These epoxied silica-coated QDCFs with high silica-coated QD concentration (25%) is observed only at vertical direction, and some QDs are visually overlapped. Therefore, surface modification of silica-coated QDs by epoxy-capping effectively improves the solubility and dispersibility of QDs in the photoresist polymer.

To evaluate the effects of epoxy treatment on the developing process, the morphology evolution of pristine and epoxied silica-coated QDCFs is compared, as shown in Fig. S2 (see Supplementary information). The pristine silica-coated QDs are precipitated in PGMEA, while the corresponding epoxied samples can be easily dispersed in the solution. This illustrates that the epoxy functional groups are conducive to the dissolution of silica-coated QDs in PGMEA. The epoxied silica-coated QDCFs were then easily peeled off after the development, forming neat and orderly patterns on the substrate, as shown in

Figure 4 TEM images of pristine silica-coated QDs (a) and epoxied silica-coated QDs (25 wt.%) in SU-8 photoresist (b).

Fig. S2e and f. Conversely, the pristine silica-coated QDCFs were extremely difficult to strip, leading to a disordered pattern (Fig. S2b and c). Therefore, epoxy modulation on silica-coated QDs is beneficial to the developing process and subsequent pattern fabrication.

In this work, we prepared color filters using epoxied silica-coated QDCF thin films to produce a badge of SUSTech (Southern University of Science and Technology), as shown in Fig. 5a. The badge pattern comprises a triangle, circle, and square in order, with the same size of 100 μm . From the SEM images, we can clearly see that the patterns are clear and neat after development, conforming to the original design.

To examine the effects of the QDCF fabricated using epoxied silica-coated QDs, PL emission spectra were recorded (Fig. S3). The QYs of different samples were compared, as summarized in Table S2. The QY of the as-obtained QDs is only 11.4%, which increases to 19.3% after silica coating and reaches 36.5% after surface modification. This indicates that the prepared QDCF exhibits an enhanced performance. The PL extraction enhancement is attributed to the silica protection, the uniform QD dispersion without aggregation, and the clean surface of the unexposed area after photolithography.

To study the photostability of the silica-coated QDCFs with and without epoxy treatment, the samples were subjected to aging tests under blue light at 300 mW cm^{-2} @450 nm. As shown in Fig. 6, the epoxied silica-coated QDCF presents a QY of 36.5% and shows a stable performance even after exposure in ambient air over 25 days. The PL intensity of the epoxied silica-coated QDCF gradually increases over time (photobrightening). Surface effects strongly influence the reactivity and optoelectronic properties of the QDs [37]: this external layer reversibly dissociates from and binds to the crystal surface, resulting in dynamic chemical formulas, surface reconstruction phenomena, and QD size reduction over time [38]. Jung *et al.* [39] reported 1.3 times of the initial PL intensity of RQDs@SiO_2 after 32 h at 120°C under a tungsten halogen light. Ligands, amorphous silica, and metal complex lay-

Figure 5 (a) Pattern mask originally designed for photolithography. (b) Mask dimensions. (c) Pixelated QDCF array molded on glass with 450 nm radiation. (d) SEM images of the epoxied silica-coated QDCF with well-ordered homogeneous shapes.

Figure 6 Photoaging of pristine QDCF and epoxied silica-coated QDCF (under 300 mW cm⁻² 450 nm blue light) for over 25 days. The measured peak wavelengths are listed in Fig. S4. Note: the QDCF samples (overall size 5 mm×5 mm) are used and placed 6 mm above the blue LED chip (overall size 2.8 mm×3.5 mm).

ers affect the surface chemistry of epoxied silica-coated QDs. The photobrightening mechanisms might involve passivation of surface states and photochemical annealing [39,40]. Nevertheless, the QDCF based on pristine QDs exhibited a dramatic 40% decrease in QY. Therefore, introducing an epoxy cap in silica-coated QDs facilitates stable and efficient QDCF thin film fabrication.

CONCLUSIONS

In summary, we prepared highly pixelated and photo-stable QDCFs by photoetching. To obtain efficient QDCFs, QDs were passivated with silica and then decorated with epoxy *via* surface ligand exchange. Introducing epoxy functional groups on silica-coated QDs effectively

enhances QD solubility in SU-8 photoresist. The epoxied silica-coated QDCF retained its initial PL and exhibited strong photostability against UV radiation for over 25 days.

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- Author contributions** Zhao B and Hao J conceived the experiments.

Zhao B, Bai X and Lu R took part in detailed experiments. Zhao B and Zhang X prepared this manuscript. Liu H, Li S and Yang H were mainly responsible for the characterization in this work. Xu B provided chemicals used in this experiment. Wang L, Wang K and Sun X W carefully reviewed and modified this manuscript. All authors discussed on the results and commented on the manuscript.

Conflict of interest The authors declare no conflict of interest.

Supplementary information Supporting data are available in the online version of the paper.

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通过表面钝化实现稳定发光的硅包覆量子点滤色片

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摘要 通过表面连接环氧基团得到高亮度像素化的二氧化硅包覆量子点滤色片(QDCF). 环氧基功能化的二氧化硅包覆量子点可以与SU-8光刻胶均匀混合, 且混合比例高达25%. 经环氧基处理之后, 二氧化硅包覆QDCF的量子产率(QY)从19.3%提高到36.5%. 合成的QDs经过25天的光照辐射(300 mW cm^{-2} @450 nm), QY较初始值降低了65%, 而环氧基二氧化硅包覆的QDCF没有明显衰减, 说明环氧基调控对QDCF的光学性能起到了关键作用.