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CdSe/ZnS quantum dot films for high performance flexible lighting and display applications

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Abstract

Colloidal quantum dots have attracted significant interest in recent years for lighting and display applications and have recently appeared in high-end market products. The integration of quantum dots with light emitting diodes has made them promising candidates for superior lighting applications with tunable optical characteristics. In this work we propose and demonstrate high quality colloidal quantum dots in their novel free-standing film forms to allow high quality white light generation to address flexible lighting and display applications. High quality quantum dots have been characterized using transmission electron microscopy, x-ray diffraction, x-ray photoelectron spectroscopy, steady state and time resolved photoluminescence and dynamic light scattering methods. The engineering of colloidal quantum dot composition and its optical properties in stand-alone film form has led to the experimentally high NTSC color gamut of 122.5 (CIE-1931) for display applications, color rendering index of 88.6, luminous efficacy of optical radiation value of 290 lm/W_{opt} and color temperature of 2763 K for lighting applications.

S Online supplementary data available from stacks.iop.org/NANO/27/295604/mmedia

Keywords: nanocrystals, quantum dots, lighting, light emitting diodes

(Some figures may appear in colour only in the online journal)

1. Introduction

Colloidal quantum dots (QDs) have emerged in the last few decades due to their promising optical and electronic properties, which make them one of the concrete building blocks of next generation technology. They have been serving optoelectronics in many applications i.e., in solar cells [1, 2], photodetectors [3, 4] and light emitting diodes [5, 6].

One of the key properties that the QDs offer is their tunable emission characteristic, which is due to the quantum confinement effect [7]. This has inspired the fields of lighting and display applications based on quantum dots. The quantum dots allow engineering of the output white light and sharp characteristics of emitters, which can be hybridized to generate the desired light output. Next generation displays require a wider color gamut to mimic natural appearance on displays, and lighting applications require high color performance, both of which cannot be met by the conventionally used phosphor based emitters. According to a recent report, revenues based on quantum dot emitters are projected to exceed phosphor revenue by 2020 [8]. In alignment with that, quantum dots have started emerging for lighting and display applications to boost their colorimetric performance.

To date there has been remarkable work done on the systematic tuning of white light parameters using colloidal quantum dots and their implementation for display

applications [9–12]. Lighting applications using QDs are generally based on the utilization of the different sized quantum dots hybridized on a blue light emitting diode platform to generate white light. However, this approach brings questions regarding the degradation of the emitters in direct contact with the underlying LED platforms. Due to their long-term thermal instability, the QDs cannot withstand the high temperature generated by the underlying current driven platform. This has led to research efforts on the implementation of stand-alone sheets of QDs ready to be utilized as remote phosphor agents. In this regard, the QD films can be employed at a distance from the excitation platform and can sustain their performance for longer, also allowing surface emission, rather than point like sources. However, the literature is lacking regarding possible novel approaches towards achieving flexible high performance emitting platforms for lighting and displays. In addition, stand-alone quantum dot films for high performance lighting and display applications, for which the requirements are different, have also not been shown to date. Lighting applications require a high color rendering index, warm color temperature and high luminous efficacy of optical radiation, however the design of emitters for display applications require an enhancement of the color gamut that can enhance the viewer's color vision, allowing it to provide more vivid colors at the display. In this study, we propose and demonstrate stand-alone sheets of CdSe/ZnS quantum dots of surface emitters which can be combined to generate white light by the three-color mixing approach (blue LED, green and red-emitting QDs) for display applications, and remarkable performance of a white light emitting device when a yellow-emitting sheet of quantum dots is combined as the fourth emitter.

Versatile use of the prepared QD sheets allows for a specific solution (lighting or display). In addition, our efforts have a significant contribution towards the realization of flexible lighting platforms. Synthesis of up to near unity quantum yield emitters, preparation of stand-alone sheets of QD emitters and the development of routes for the flexible lighting platforms have been performed. This has led to the achievement of blue LED excited films providing the best performing enhanced color gamut of 122.5% (as compared to the NTSC (National Television System Committee) color gamut) with remote phosphor approach based sheets in the display application mode. A color rendering index of 88.6, luminous efficacy of optical radiation value of 290 lm/W_{opt} and warm color temperature of 2763 K have been achieved in the white light application mode. (The reported values are the optimal data for achieving warm white light, however CRI $>$ 90 levels and LER $>$ 300 are possible with cold white light corresponding to color temperature ca. 2000 K using our flexible films.) The performance values achieved are the best optimal achieved experimental values for flexible remote phosphor films for lighting and display applications.

2. Sample preparation

2.1. Synthesis of CdSe/ZnS quantum dots and preparation of free-standing films

Cadmium oxide (CdO, 99.99%), zinc acetate (ZnAc, 99.99%), zinc acetate dihydrate ($ZnAc.2H_2O$, 99.999%), selenium (Se, 99.99% powder; 99.999% pellets), sulfur (S, 99.998% trace metals basis; 99.5% purum), oleic acid (OA, 99.99% technical grade), 1-dodecanethiol (DDT, 98%) 1-octadecene (1-ODE, 90% technical grade), trioctylphosphine (TOP, 90% technical grade), poly (methyl methacrylate (Mw: 996000)), anisole (99.0%), ethanol (EtOH, 99.8% absolute), acetone (Act, 99.5% absolute) and methanol (MeOH, absolute). All chemicals and reagents were purchased from Sigma-Aldrich. All chemicals were used without further purification.

2.1.1. Synthesis of green-emitting QDs. The synthesis of green-emitting CdSe/ZnS QDs was performed by using a modified version of a previously reported method [13, 14]. Individual precursor concentrations, their ratio, vacuum level and stirring speed all contributed to the quality of the synthesized colloidal quantum dots and it is possible to drastically change the quality of the end product by small modifications to the recipe [15]. 0.3 mmol of CdO and 4 mmol of zinc acetate (ZnAc) reacted with 5 ml of oleic acid in a 50 ml three-necked flask under vacuum at 150 °C for 30 min (vacuum level reaching ca. 10^{-3} Torr). Following this, the solution was cooled to 50 °C and 15 ml of 1-ODE was loaded in a 50 ml reaction flask equipped with a condenser and heated to 100 °C under vacuum. As the reaction system was maintained under Ar gas flow, the temperature was increased to 300 °C to obtain the transparent mixed solution of cadmium oleate and zinc oleate. Separately, a mixture of 0.3 mmol of Se and 3 mmol of S was dissolved in 2 ml of TOP at room temperature and stirred overnight at 800 rpm in a glove box. Afterwards, a clear solution of Se and S in TOP was obtained and rapidly injected into the reaction flask at 300 °C and the solution was kept for 10 min for the completion of the green CdSe/ZnS QDs. Then, the mixture was cooled to room temperature to end the reaction. The synthesized QDs at room temperature were transferred to a cleaning process. A precipitation and re-dispersion method was used for the cleaning of QDs. The crude solution was precipitated in the presence of absolute acetone and methanol by centrifugation at 5000 rpm for 15 min and finally QDs were re-dispersed in the hexane.

2.1.2. Synthesis of yellow-emitting QDs. The reported methods were modified for the synthesis of yellow-emitting CdSe/ZnS QDs [13, 14]. 0.8 mmol of CdO and 4 mmol of zinc acetate (ZnAc) reacted with 5 ml of oleic acid in a 50 ml reaction flask with a condenser equipped system under vacuum at 150 °C for 30 min (vacuum level reaching ca. 10^{-3} Torr). The solution was cooled to 50 °C; 15 ml of 1-ODE was loaded in the reaction vessel and reaction was heated up to 100 °C under vacuum. Later, the reaction was

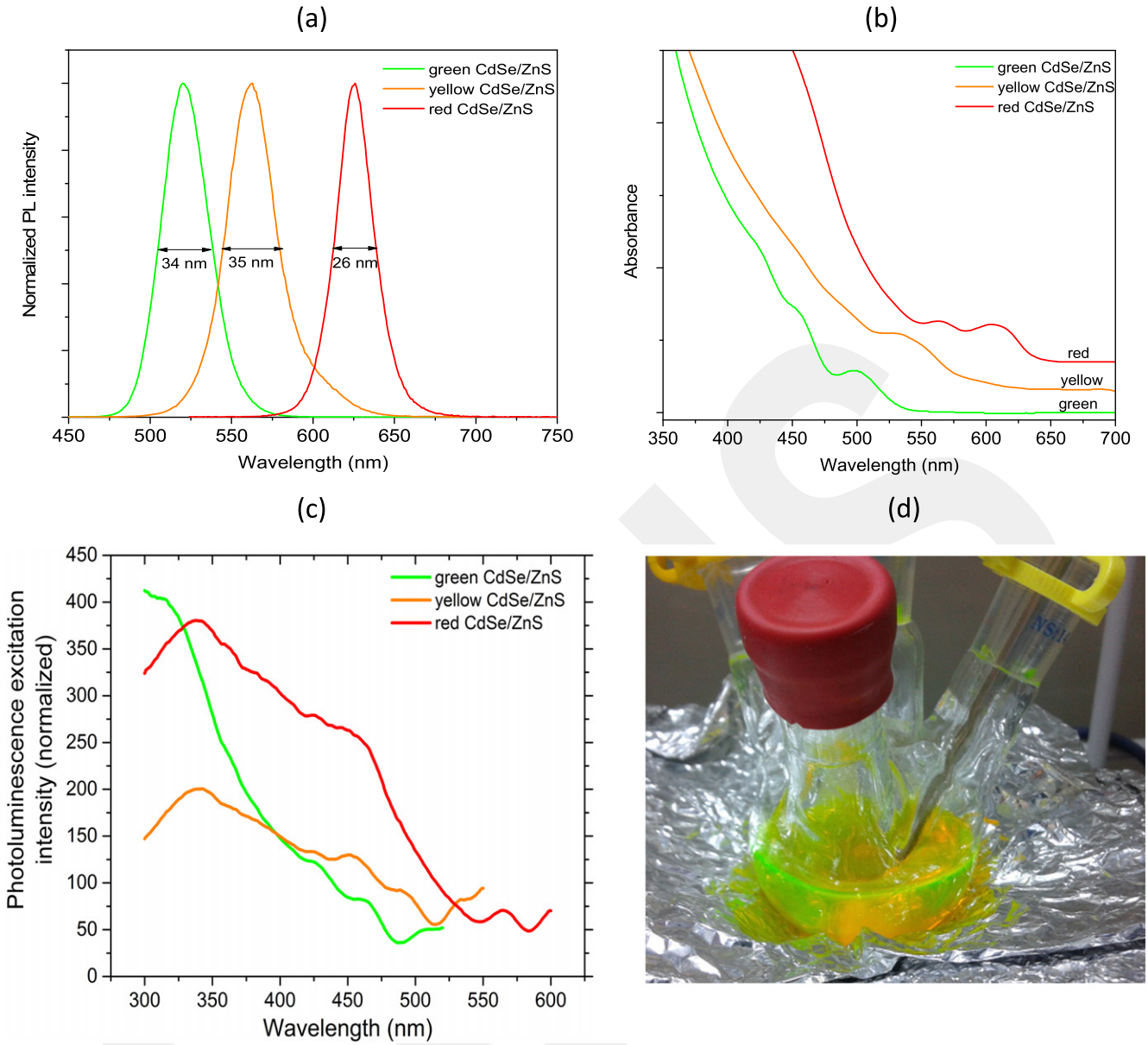

Figure 1. (a) The emission, (b) absorption, (c) photoluminescence excitation spectra of the synthesized CdSe/ZnS colloidal quantum dots. (d) A photo of the green-emitting quantum dots just after synthesis, under room light (without UV illumination).

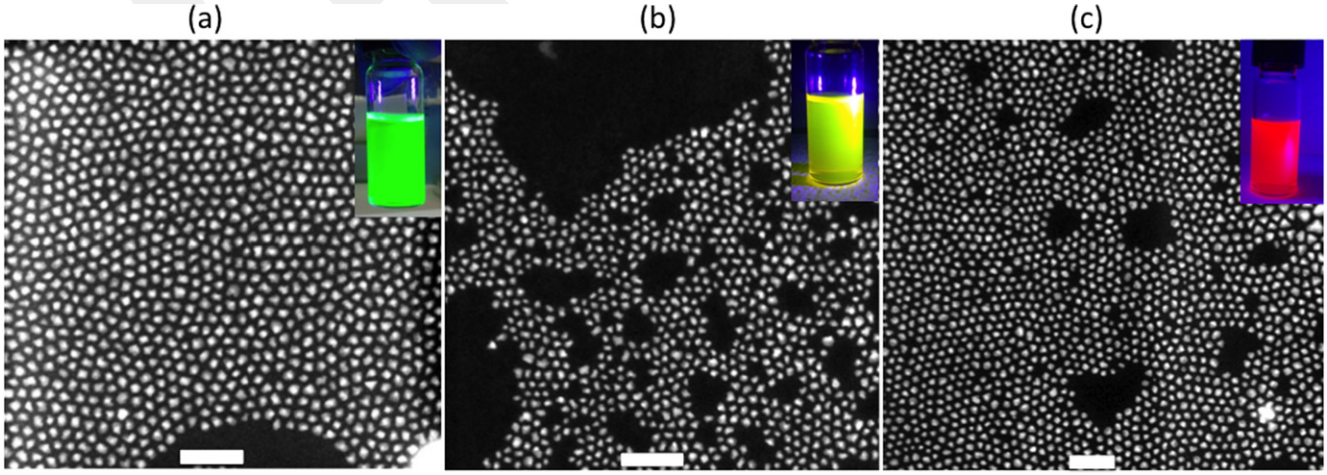

Figure 2. HAADF-TEM images of the (a) green, (b) yellow and (c) red-emitting CdSe/ZnS quantum dots. The scale bar is 50 nm.

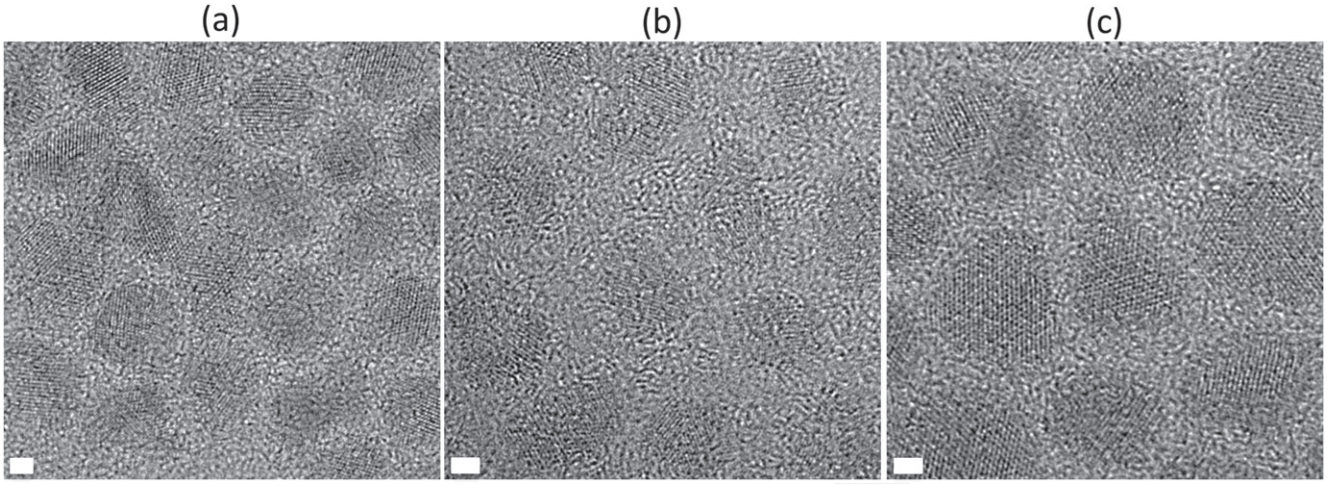

Figure 3. HR-TEM images of the (a) green, (b) yellow, and (c) red-emitting CdSe/ZnS quantum dots. The scale bar is 2 nm.

heated to 300 °C under Ar gas flow to obtain a clear and homogeneous mixed solution of cadmium oleate and zinc oleate. A mixture of 0.8 mmol of Se and 3 mmol of S were prepared by dissolving them in 2 ml of TOP at room temperature with overnight stirring at 800 rpm in a glove box. The homogeneous clear solution of Se and S in TOP was rapidly injected into the reaction system at 300 °C and the solution was kept at this temperature for 10 min in order to grow yellow-emitting CdSe/ZnS QDs. The cleaning process was carried out in the same way as explained for the green-emitting dots.

2.1.3. Synthesis of red-emitting QDs. The reported procedures [13, 14, 16] were modified for the synthesis of red-emitting CdSe/ZnS core–shell QDs. 1 mmol of CdO and 1.68 mmol of zinc acetate dihydrate and 5 ml oleic acid were added to a 50 ml three-necked flask and heated to 140 °C under vacuum for 30 min (vacuum level reaching ca. 10^{-3} Torr). Then, the solution was cooled to 50 °C and 25 ml of 1-ODE was loaded to the mixture of cadmium oleate and zinc oleate in the reaction flask. The solution was first heated to 100 °C under vacuum and then the temperature was increased up to 300 °C under Ar gas flow. Separately, the injection solution was prepared as 1 M TOP-Se (pellet) solution, by stirring overnight at 800 rpm at 100 °C in a glove box. From the stock solution of TOP-Se, 0.2 ml of solution was rapidly injected into the reaction flask at an elevated temperature (300 °C) and kept for 80 s. Then, 0.3 ml of 1-DDT in 1 ml of 1-ODE was injected into the system and the reaction continued for 20 min. Afterwards, 1 ml of TOP-S (2 M) was slowly injected to the flask at 300 °C in 10 min. Subsequently, the solution was cooled to room temperature in order to produce the red-emitting CdSe/ZnS QDs. The cleaning process was carried out in the same way as explained for the green-emitting dots.

2.1.4. Preparation of the free-standing films. Poly (methyl methacrylate) PMMA was prepared ca. 7.5% by dissolving in anisole. Upon preparation of the viscous solution, the cleaned QDs in fresh hexane were mixed with the PMMA and left for rigorous stirring to provide well mixed QDs with polymeric solution. The solution was drop cast on the pre-cleaned glass slides (pre-washed with detergents using tap water, followed by cleaning with ethanol under sonication for 20 min) and left to dry under controlled evaporation overnight. The fully dried films were peeled off from the glass surface and used as the flexible sheets of QDs ready for further experiments.

3. Results and discussions

The CdSe/ZnS QDs were synthesized using recipes explained in the previous section. By changing the composition of the colloidal quantum dots, tunability of their optical properties was achieved. In our experiments we used colloidal quantum dots with in-solution peak emission wavelengths of 520, 562 and 626 nm along with their 34, 35, 26 nm full width at half maximum (FWHM) values, having photoluminescence quantum yields of 90%, 50% and 78%, respectively. Absorption and photoluminescence spectra were collected using a UV-1800 Shimadzu and Cary Eclipse Fluorometer. Green, yellow and red-emitting quantum dots' quantum efficiency was measured by comparison with an organic dye using the methodology explained in the literature [17]. Green and yellow-emitting dots were compared with rhodamine 6 G in ethanol (QE:95%) and red-emitting dots with sulphorhodamine 101 in ethanol (QE:90%). Intersecting the absorption curves of the quantum dot and respective dye at the 460–490 nm range for the rhodamine 6 G and 510–560 nm range for sulphorhodamine 101, around 0.1–0.2 absorbance value to prevent reabsorption, the photoluminescence measurements were performed at the intersection wavelength (chosen as excitation wavelength for photoluminescence measurements) and the spectral areas were compared to calculate the quantum efficiencies. The photoluminescence and absorption spectra of the quantum dots and the respected dyes are given in the supplementary information stacks.iop.org/NANO/27/295604/mmedia.

Table 1. Energy dispersive x-ray spectroscopy data for the green, yellow and red-emitting quantum dots.

	Green QD			Yellow QD			Red QD		
Element	Weight (%)	Atomic (%)	Uncertainty (%)	Weight (%)	Atomic (%)	Uncertainty (%)	Weight (%)	Atomic (%)	Uncertainty (%)
S	20.51	36.71	0.48	14.63	29.80	0.51	17.95	40.25	0.71
Zn	54.13	47.49	1.03	38.60	38.54	0.98	9.39	10.32	0.36
Se	13.23	9.61	0.52	18.28	15.11	0.91	10.95	9.96	0.58
Cd	12.10	6.17	0.50	28.46	16.53	1.63	61.70	39.45	2.63

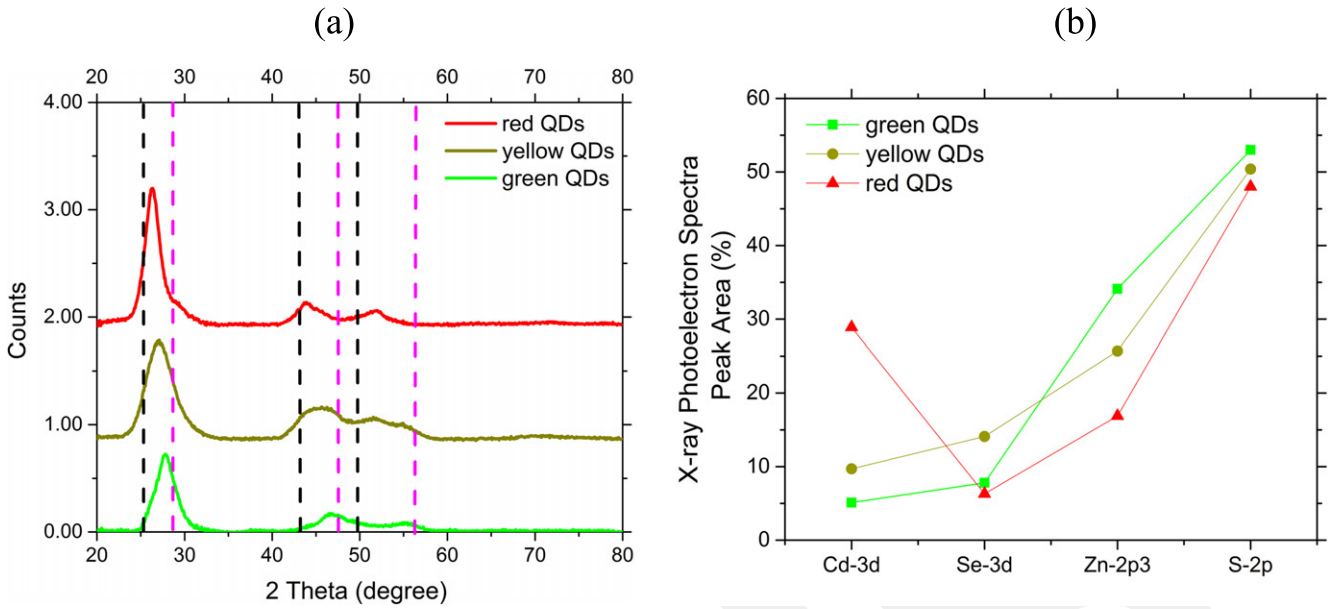

Figure 4. (a) X-ray diffraction (XRD) spectra of CdSe/ZnS core–shell QDs. Dashed black and purple lines represent the corresponding (111), (220) and (311) planes of zincblende CdSe and ZnS respectively. (b) X-ray photoelectron spectra (XPS) peak area percentages of each QD for Cd-3d, Se-3d, Zn-2p3 and S-2p peaks.

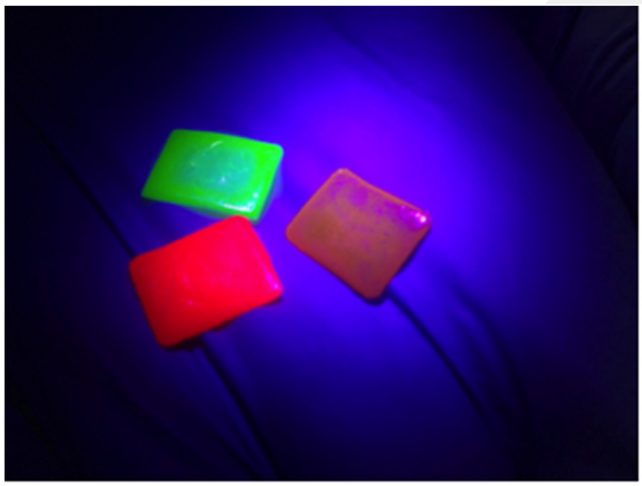

Figure 5. Free-standing sheets of CdSe/ZnS quantum dots under UV light.

The optical properties of the synthesized quantum dots are shown in figure 1, along with the high-angle annular dark field transmission electron microscopy images in figure 2, and high-resolution transmission electron microscopy images in figure 3 for the green, yellow and red-emitting CdSe/ZnS quantum dots (using FEI—Model: Tecnai G2 F30 (with EDX)). An HR-TEM image of an individual red-emitting CdSe/ZnS quantum dot is given in the supplementary information.

Regarding the generation of white light for display or lighting applications, there is a definite need for the careful optimization of the emission peak wavelength and FWHM of the nanoemitters. Whereas the narrow emitters provide an enhanced color gamut for the display applications, optimization for lighting requires satisfying the desired white coordinates along with high color performance (i.e., providing color temperature < 3000 K, color rendering index > 90 and luminous efficacy of optical radiation > 300 lm/W_{opt}). In that regard, although it is possible to achieve a larger color gamut using a three-color mixing approach utilizing emitters with narrow FWHM, for the lighting applications, the narrow green and red emitters themselves are not sufficient to address the requirements of high quality lighting. This then leads us to the use of the yellow emitters to fill the spectral content. In addition to that, the peak positions of the emission peaks and their spectral contribution to the output white light also affect the performance figures of merit of the white light generated, which requires fine tuning of the optical properties of the nanoemitters.

The energy disperse x-ray spectroscopy experiment results achieved from the STEM measurements are given in table 1, which presents the Cd, Se, Zn and S content for the different sized QDs. In accordance with the synthesis approach explained previously, the color tuning of the QDs was achieved by modifying the Cd and Zn content of the synthesized particles. The average size of the quantum dots was found to be 5.42, 4.60, 6.00 nm for green, yellow and red quantum dots, respectively, using dynamic light scattering (Malvern-Zeta Sizer). The dynamic light scattering measurements and size histograms for each QD are presented in the supporting information.

The particles were further investigated using x-ray diffraction (XRD) and x-ray photoelectron spectroscopy (XPS). Figure 4(a) demonstrates the XRD of the colloidal quantum dots taken by a PANalytical: X'pert Pro MPD. The diffraction peaks of CdSe/ZnS quantum dots shift to (111), (220), (311) planes of the ZnS for green-emitting QDs, which is in good agreement with the EDX measurement analysis given in table 1. Due to their more pronounced Cd content, the red-

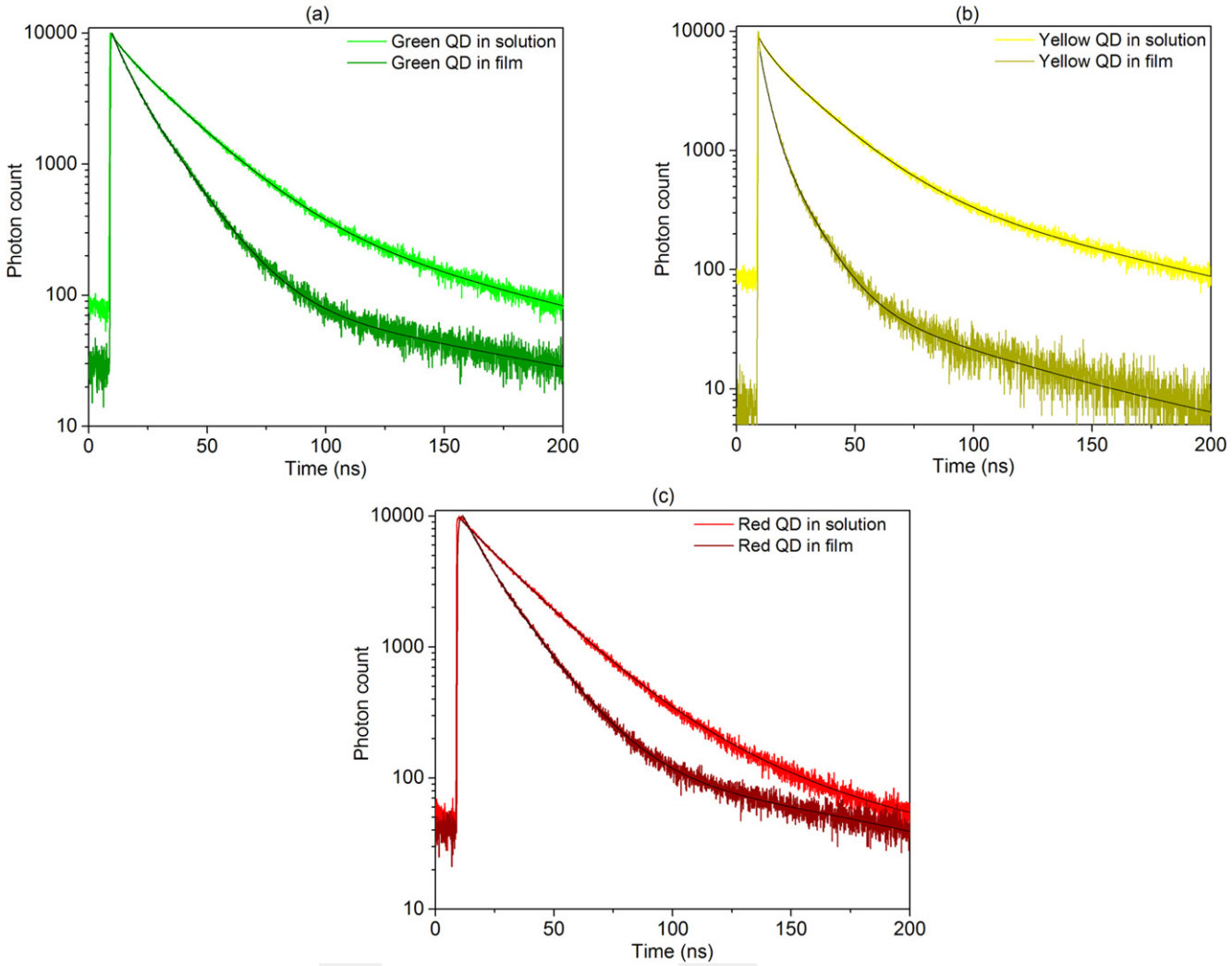

Figure 6. (a) Time resolved photoluminescence decays of (a) green, (b) yellow, (c) red-emitting CdSe/ZnS colloidal quantum dots.

Table 2. Time resolved fluorescence fit data for the green, yellow and red-emitting quantum dots.

	τ_1 (ns)	τ_2 (ns)	τ_3 (ns)	$\tau_{\text{amp-average}}$ (ns)
Green QD in solution	113.56 (4.77%)	23.379 (81.78%)	4.925 (13.45%)	25.19
Green QD in film	161.4 (1.18%)	15.435 (64.70%)	5.277 (34.12%)	13.70
Yellow QD in solution	100.2 (6.81%)	21.365 (73.13%)	4.202 (20.06%)	23.29
Yellow QD in film	67.91 (1.05%)	10.694 (26.88%)	3.266 (72.07%)	5.94
Red QD in solution	227.31 (3.16%)	24.136 (91.69%)	5.970 (5.15%)	29.63
Red QD in film	398.3 (2.20%)	17.165 (65.69%)	7.354 (32.11%)	22.40

emitting quantum dots' XRD spectra resemble the peaks of bulk CdSe. However in green-emitting QDs, where the Zn and S content is comparatively higher, we observe that the XRD peaks are closer to the bulk values of ZnS crystal structure. Figure 4(b) depicts the XPS peak area percentage for Cd-3d, Se-3d, Zn-2p3 and S-2p performed by K-Alpha x-ray photoelectron spectroscopy. In this comparison, the oxygen, carbon and nitrogen peaks were not included in the analysis. The results have a similar pattern with the atomic percentages shown for EDX analysis (table 1). In agreement with our synthesis approach, the areal comparison of the XPS also depicts the compositional nature of the synthesized QDs. The XPS spectra for each QD including oxygen, carbon and nitrogen are given in supporting information.

As has been outlined in the introduction, the importance of using the remote phosphor approach stems from the fact that the colloidal quantum dots cannot withstand high temperatures when in direct contact with the underlying LED platform. This effect has also been observed for the existing phosphor material used as the color converter. This has led to research efforts on the fabrication of free-standing color converting films that can allow for their versatile use and in

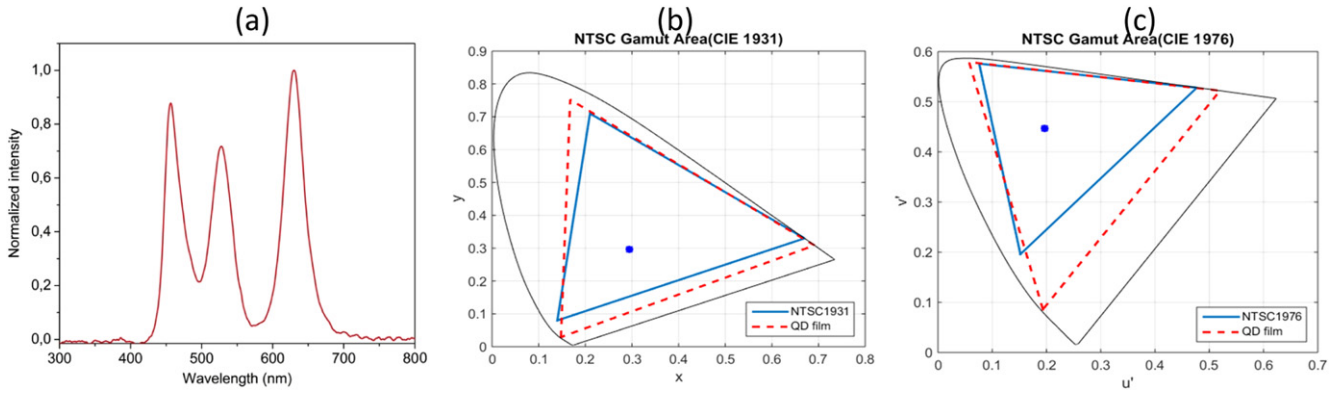

Figure 7. (a) The output light intensity as the red and the green emitters are hybridized with blue LEDs. The color gamut enhancement in (b) NTSC 1931, (c) in NTSC 1976 coordinates.

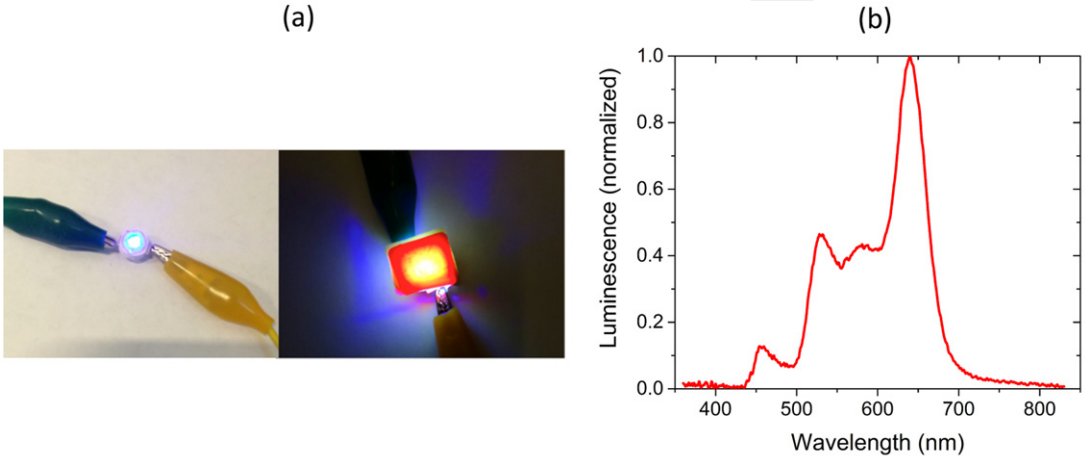

Figure 8. (a) Blue LED hybridized with and without CdSe/ZnS quantum dot flexible sheets. (b) Spectral light output from the free-standing sheets of CdSe/ZnS quantum dots hybridized with blue LED.

that respect, very large areas of quantum dot free-standing films have previously been shown [18]. Free-standing films of CdSe/ZnS QDs have been achieved by using a polymethylmethacrylate (PMMA) matrix as the host polymer material. Figure 5 shows QD films made up of green, yellow and red emitters that offer flexible, foldable solutions to be used for solid state lighting and display applications.

In order to investigate the effect of the photoluminescence emission kinetics of the free-standing films, we performed time resolved photoluminescence measurements both for in-solution and film samples, excited using a pulsed diode laser at 375 nm (using PicoQuant Fluo Time 200). Regarding the emission kinetics, we observed that the lifetime decays of the in-solution quantum dots get faster in their film form (figure 6). This is due to the nonradiative channels being dominant in the film form of the colloidal quantum dots. As the nanocrystals are transferred to a film structure, we observe that the amplitude averaged lifetime values (fit by three exponentials) of the green QDs decrease from 25.19 ns to 13.70 ns, the yellow QDs' lifetime from 23.29 to 5.94 ns, and the red QDs' lifetime from 29.63 to 22.40 ns. The decrease in the average lifetime of the QDs is mostly attributed to the increase in the contribution of τ_3, the fastest decay component, which has been shown to increase from 13.45% to 34.12% for green, 20.06% to 72.07% for yellow and 5.15% to 32.11% for red-emitting QDs. The details of the decay analysis are given in table 2. It is also worth noting here that the energy transfer (nonradiative) within the ensemble of quantum dots may also contribute to the change in QD lifetime.

Figure 7 shows the intensity of the output light generated when the green and red-emitting films have been hybridized on the blue emitting LED platform. As a blue LED excitation source, we use 456 nm emitting commercially available InGaN LEDs, which are driven by using a Keithley-2400 sourcemeter. The spectral content of the output light is used to determine the performance of the output light generated.

For the three-color mixing approach, the color coordinates were determined as (x, y) (0.2939, 0.2958) along with a CRI value of 49.0, CCT of 8357 K and LER value of 239 lm/W_{opt} (figure 7(a)). As also has been seen in the output light content of the three-color mixing, there is a gap to deliver the high quality white light. Although the color mixing of the green and the red emitters allows for the generation of white light for a non-optimized performance in a lighting perspective, it provides a wide color gamut to enhance the color

gamut to 122.5% (NTSC-CIE 1931) and to 148% (NTSC-CIE 1976). The corresponding color gamut enhancement visualization is given in figures 7(b) and (c) for the CIE (International Commission on Illumination) 1931 and 1976 diagrams. In the calculation of the color gamut enhancement, the peak emission wavelengths and FWHM values read (456 nm, 28 nm) for the blue LED, (527 nm, 37 nm) for the green and (629 nm, 33 nm) for the red emitters. The emission properties of the QDs are slightly different (red-shifted) in their film form as compared to the in-solution optical emission properties due to environmental effects when the medium is changed.

In order to address for the high quality white light parameters, we have further incorporated an additional yellow-emitting sheet to the existing green and red emissive layer and have performed the experiment for the spectral light output. The collected output light from the fiber (StellarNet Inc. Spectrometer with fiber optic output) is investigated to calculate the figures of merit of the white light generated.

Using a four-color mixing approach we achieved coordinates of (x,y) (0.4709, 0.4393) along with a CRI value of 88.6, luminous efficacy of optical radiation value of 290 lm/W_{opt} and color temperature of 2763 K, which suits the warm white light spectrum well. Figure 8(a) shows the blue LED hybridized with and without CdSe/ZnS quantum dot flexible sheets and figure 8(b) shows the spectral output of the color converter hybridized with the blue LED, with 2.65 V potential difference applied to the end terminals of the device.

It is worth noting here that a careful optimization of the color coordinates and the emission intensity of the individual emitters is necessary in order to optimize the light output, and thus the color coordinates. It is evident from the literature that the red component has a great influence on the quality of the white light achieved [19]. In our experiment we have incorporated a red-emitting free-standing sheet in an orientation such that not only the blue emission but also the green and yellow emission contribute to the excitation of the red-emitting species. The quantum dot films were used as separate sheets rather than mixing them in a film form to prevent Förster-type resonance energy transfer among them. The physical distance between different optical sheets therefore does not affect the overall output spectrum.

4. Conclusions

In conclusion, we have demonstrated free-standing sheets of versatile CdSe/ZnS quantum dots as light generating agents to be implemented for lighting and display applications. Starting from the synthesis of high quality colloidal quantum dots with in-solution peak emission wavelengths of 520, 562 and 626 nm along with their 34, 35, 26 nm full width at half maximum (FWHM) values, and in-solution photoluminescence quantum yield of 90%, 50% and 78%, we have extensively characterized QDs using transmission electron microscopy, x-ray diffraction, x-ray photoelectron spectroscopy, steady state and time resolved photoluminescence and dynamic light scattering. By further incorporating quantum dots to flexible polymeric films, we have utilized green and red emitters and achieved an enhanced NTSC color gamut reaching 122.5%. The incorporation of the yellow-emitting sheets has allowed us to target high performance lighting allowing warm white light with a high CRI level (88.6) and color temperature around 2700 K. The realization of high-efficiency and high performance quantum dot sheets for flexible lighting and display applications will pave the way for the sustainable growth of lighting and display industry.

Acknowledgments

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