Efficient Charge Extraction from CdSe/ZnSe Dots-on-Plates Nanostructures

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ABSTRACT: An efficient and a selective charge extraction from a new type of heterostructured material is demonstrated: the quasi-type-II structure formed upon deposition of ZnSe quantum dots on CdSe nanoplatelets, termed as CdSe/ZnSe dots-on-plates (DoPs) heterostructures. Insights into the charge extraction mechanism are gained from the present studies. Quenching experiments on nanoplatelets (NPLs) and DoPs using electron (benzoquinone) and hole (pyridine) quenchers show the possibility of electron extraction leaving behind the hole in the nanostructures. These outcomes indicate more labile electron extraction in comparison with the hole from these DoP structures vis-à-vis the plate only nanostructures, thereby enabling materials for devices requiring only one type of charges. In CdSe NPLs, the excitons are short-lived making them difficult for various applications involving charge separation. The CdSe/ZnSe DoPs could be alternate candidates for overcoming the difficulties involved with NPLs.

INTRODUCTION

Over the last few decades, there has been tremendous interest in the synthesis of colloidal semiconductor nanomaterials (NMs) and their applications in various areas, such as photovoltaics, light emitting diodes, biological imaging and sensors, transistors, and lasers.1−9 The unique features of these NMs are due to the quantum confinement effect, which results into size-dependent optoelectronic properties. Initially, research was more focussed on the optimized syntheses of various sizes of these colloidal nanocrystals (NCs), followed by the development of different synthetic strategies to synthesize NMs of varied shapes and compositions as well. To date, various shapes of the semiconductor NMs, including spherical quantum dots (QDs), nanorods, tetrapods, and nanoplatelets (NPLs)10−12 have been synthesized successfully. The QDs, nanorods, and NPLs exhibit confinement of excitons in three, two, and one dimensions, respectively. Out of the NMs mentioned above, the most recent addition to the family are the colloidal two-dimensional (2D) materials, that is, NPLs, nanoribbons, nanosheets, quantum belts, or quantum disks.13−16 Two-dimensional nanostructures of cadmium chalcogenides (CdE, E = S, Se, and Te) have been synthesized with control of their thickness with atomic precision. These show sharp absorption and emission peaks with very small Stokes shifts and short radiative fluorescence lifetimes. The optical properties of these NPLs vary strongly with their thickness. Because the thickness, which is in the order of a few monolayers (MLs) of the material, is typically less than the bulk exciton Bohr radius, effects of quantum confinement can be exploited to tune the optical absorption and photoluminescence (PL) spectra.17 Two-dimensional NPLs have precise quantum confinement throughout the sheet along the thickness and not in all directions as in the 0D nanoparticles. Because of their unidirectional confinement and large lateral extension, 2D NPLs are perfect candidates for charge transport owing to reduction in the number of hopping steps as a result of a high ratio of their lateral size over their confined direction.18−20 These properties make 2D NPLs promising candidates for light harvesting and charge transport materials in photovoltaics.21,22

An additional epitaxial growth of a semiconductor layer in the vicinity of the starting semiconductor material and decoration of another semiconductor or metal on their surface leads to various architectures of heterostructures. By doing so, the properties can be altered precisely by controlling the spatial delocalization of the charge carriers across the heterostructures. They may carry properties exhibited by each semiconductor component as well as new properties generated as a result of their combination. These colloidal nanoheterostructures are possibly the best candidates for excitonic engineering and present fascinating opportunities for enhanced platforms of colloidal photonics.2,23−30 On the basis of the relative alignment of the conduction band (CB) and valence band (VB) edges of the two materials, these NMs can be differentiated as type-I or type-II. In type-I structures, the
electron and hole reside in the same material of the structure leading to maximum overlap of electron and hole wave functions. In type-II structures, the lowest CB and highest VB edges of the two components lie in different semiconductors. Therefore, the electrons and holes localize in different parts of the heterostructure resulting in charge separation. In the case of cadmium chalcogenide NPLs, another semiconductor material can be grown epitaxially as well as laterally. The NPL—semiconductor heterostructures that result from the epitaxial growth of another material are termed as core/shell nanoplatelets, whereas they are called core/crown nanoplatelets when the semiconductor material is grown laterally. The first report on core/shell NPLs involved the synthesis of CdSe/CdS and CdSe/CdZnS, in which they were shown to exhibit red-shifted absorption and emission properties compared to those of the core NPLs. From then on, these systems have been studied extensively by various groups. For synthesis of core/shell NPLs, besides one pot synthesis, the colloidal atomic layer deposition technique has also been employed and multiple quantum wells have been synthesized successfully. Also, several varieties of core/crown heterostructures, such as type-I CdSe/CdS and CdS/CdSe and type-II CdSe/CdTe and CdTe/CdSe, have been synthesized and studied recently. In core/crown structures, the crown layer grows only in lateral directions and this does not result into any noticeable change in the emission spectrum with respect to that of core NPLs because the confinement in the vertical thickness, which dictates the electronic structure, does not change.

To the best of our knowledge, we have not come across a single report that employs ZnSe for purposes of charge separation in CdSe NPLs although other semiconductors and metals have been used. Here, we report the synthesis and characterizations of CdSe/ZnSe dots-on-plate (DoP) NPLs having a quasi-type-II structure. For this purpose, initially we have synthesized 4 and 5 ML CdSe NPLs, which are used further as seeds for the growth of ZnSe over these NPLs to result into CdSe/ZnSe DoPs. We have carried out structural and optical studies of these DoPs. The PL measurements show an emission feature at low energy in the red region, quite different from that of core NPLs, which exhibit green emission. Time resolved photoluminescence (TRPL) studies have also been done to understand their decay behavior, which results into a longer average lifetime for CdSe/ZnSe DoPs in comparison to that of the core CdSe-only NPLs. The NPLs possess zinc blende (ZB) crystal structure, and this is preserved even after the coating of ZnSe, confirmed from the powdered X-ray diffraction (PXRD) patterns. The spatial separation of electrons and holes across the CdSe/ZnSe heterojunction effectively suppresses recombination processes, leading to a long-lived charge transfer exciton state. If it is possible to suppress recombination, it might well be possible to extract the charges from the excited state. Following this hint, we performed quenching experiments of charge carriers with electron and hole quenchers, namely benzoquinone (BQ) and pyridine (Py). It was realized that although the electron can be extracted easily from the charge-separated state and can be utilized, the hole prefers to remain stuck in the NMs, most likely in the traps. This is also supported by photocatalytic dye degradation studies (see Supporting Information (SI)).

### RESULTS AND DISCUSSION

In this study, we have synthesized 4 and 5 ML thick CdSe/ZnSe DoP heterostructures. Figure 1A depicts the formation of DoPs from core CdSe NPLs in the presence of zinc and selenium precursors. On the basis of the band alignment, as illustrated in Figure 1B, the CdSe/ZnSe forms a quasi-type-II system where CdSe is a narrow band gap semiconductor whose CB lies much below that of ZnSe, whereas the VB lies very close to that of ZnSe. The electrons are strongly localized in CdSe due to a large CB offset. However, the holes are delocalized in the CdSe/ZnSe system owing to the very small VB offset, making it a quasi-type-II structure.

We employed seed-mediated growth to synthesize the CdSe/ZnSe DoP heterostructures. For this purpose, at first, CdSe NPLs of 4 and 5 ML thickness were synthesized with the procedure described in the Experimental Section. The assignment of the number of MLs has been extensively worked out and detailed in the literature. The absorption and PL spectra of 4 and 5 ML thick CdSe NPLs are given in Figure 2a,d, respectively. The first two absorption maxima correspond to the heavy-hole electron (HHE) and light-hole electron (LHE) transitions. In Figure 2a, 4 ML CdSe NPLs exhibit absorption transitions due to HHE and LHE at 512 nm (2.42 eV) and 481 nm (2.58 eV), respectively. These show a very sharp and narrow emission at 515 nm (2.40 eV), with a full width at half-maximum (FWHM) of 8 nm (38 meV), and the narrow emission from these CdSe NPLs is due to their pure one-dimensional confinement. CdSe NPLs (5 ML) have a sharp emission peak at 554 nm (2.24 eV), with a FWHM of 9 nm (36 meV); HHE and LHE absorption transitions occur at 551 nm (2.25 eV) and 520 nm (2.38 eV), respectively, as shown in Figure 2d. These CdSe NPLs have been used as seeds for further decoration with ZnSe dots. The procedures are outlined in the Experimental details.

The absorption and PL spectra of the synthesized 4 and 5 ML CdSe/ZnSe DoP heterostructures are given in Figure 2b,e, respectively. In Figure 2b, we see that some of the excitonic features of 4 ML CdSe NPLs are still preserved after the growth of ZnSe dots around the NPLs but are not as sharp as they were in the pristine case; they are slightly broadened. Also, an additional excitonic feature appears at a lower energy after incorporation of the ZnSe dots. The increased absorption at high energy is due to the ZnSe coating as it absorbs in the region below 450 nm that corresponds to its bulk band gap of 2.7 eV. It suggests that the NPL structure is still intact and coating with ZnSe is not affecting the original structure but providing additional structural and optical characteristics. The PL spectrum shows the emission maximum shifted to a lower energy of 2.06 eV (600 nm) compared to that in the bare NPL emission at 2.41 eV (515 nm). In 5 ML thick CdSe/ZnSe NPLs, similar features are observed, as shown in Figure 2e. The PL spectrum shows the emission at 2.02 eV (612 nm) red-shifted from the bare CdSe NPLs that emit at 2.24 eV (554 nm).
The FWHM of the PL peak from DoPs is larger than that of the PL peak from bare NPLs: 113 meV versus 37 meV for 4 MLs and 109 meV versus 36 meV for 5 MLs. Also, the absence of the NPL emission peaks suggests almost complete DoP formation; bare CdSe NPLs would have shown the respective emissions.

The PL decay curves for CdSe NPLs and CdSe/ZnSe DoP heterostructures are shown in Figure 2c,f. Both 4 and 5 ML CdSe NPLs show faster decays with short average lifetimes, whereas the growth of ZnSe dots around these NPLs results in a slow recombination of charge carriers, with longer average lifetimes. The PL decay curves are fitted by a three-exponential decay function, suggesting multiple decay channels. The fitted parameters such as lifetimes, coefficients of each lifetime, and average lifetimes, are presented in Table 1. The average lifetime of emission from DoP heterostructures is enhanced 4-fold in comparison to bare CdSe NPLs. It has been suggested that CdSe NPLs involve complex decay kinetics that is hard to interpret. However, different lifetime components can be attributed to the radiative and nonradiative channels, which can be associated with electron or/and hole trapping.

Table 1. Lifetime Components of CdSe NPLs (4 and 5 ML) and of Corresponding CdSe/ZnSe DoPs

| system               | τ_1  | α_1  | τ_2  | α_2  | τ_3  | α_3  | ⟨τ⟩  |
|----------------------|------|------|------|------|------|------|------|
| 4 ML CdSe           | 0.59 | 0.86 | 5.37 | 0.12 | 36.5 | 0.019| 15.81|
| 4 ML CdSe/ZnSe      | 4.97 | 0.56 | 30.34| 0.38 | 164.55| 0.054| 78.68|
| 5 ML CdSe           | 3.24 | 0.56 | 7.67 | 0.43 | 37.7 | 0.019| 10.13|
| 5 ML CdSe/ZnSe      | 5.92 | 0.38 | 27.36| 0.58 | 113.93| 0.039| 42.41|

We discuss the effects of the lifetime changes below. Before that is discussed, to confirm the formation of NPLs and DoPs, structural characterization is necessary. In Figure 3, PXRD patterns of 4 and 5 ML NPLs and DoPs are presented. The patterns for bulk CdSe in ZB and wurtzite (WZ) phases and ZnSe in ZB phase are also provided. The CdSe NPLs possess ZB crystal structures, as seen from the comparison with the bulk diffraction patterns. All of the reflection peaks can be indexed with the ZB crystal structure of CdSe and the absence of any peak at 35 and 45° that would correspond to the (102) and (103) reflections, respectively, rules out the possibility of a WZ structure. It is observed that the crystal structure of NPLs is preserved after decoration with ZnSe except that a little shift of the peaks toward the higher angle is noticed, indicating the smaller size of the ZnSe lattice that grows on the CdSe NPLs. It is reported that the NPLs have their thickness in the 100 direction. The TEM images of CdSe NPLs are shown in Figure 3b,f. The average dimensions of 4 ML NPLs are 17 ± 2 nm of length and 8 ± 2 nm of width, whereas for the 5 ML NPLs these are 20 ± 2 and 10 ± 2 nm. When the CdSe NPLs are coated with ZnSe, the resulting structures are shown in Figure 3c,g. It is seen that some dark patches are present around the NPLs, and these are of ZnSe, which is also confirmed by EDX, shown in the SI. The high-resolution transmission electron microscopy (HRTEM) images in Figure 3d,h for 4 ML CdSe/ZnSe DoPs show that the dark patches at the NPL edges correspond to the ZnSe dots as the d-spacing of 3.2 Å matches very well with the ZB ZnSe (111) plane. Now that these nanostructures are well-characterized, we come back to the fact that the lifetime increases over 3-fold upon decoration with ZnSe dots that we discussed above. This prompted us to see whether it is possible to extract the charges...
from these DoPs and whether it is easier compared to that from NPLs.

Electron–Hole Quenching Studies. To gain an insight into the charge carrier dynamics, we performed experiments involving quenching of charge carriers. These are established techniques\(^{45,46}\) that have been reported earlier using BQ as an electron acceptor and Py as a hole acceptor to understand the accessibility of charge carriers (electron or hole). The ease of accessibility of the charge carriers also throws light on the trap states present in the materials. Charges associated with deep traps should be difficult to extract.\(^{19,47}\)

Figure 4a presents the PL data of 4 ML CdSe NPLs in the absence and presence of a quencher. From these, it is clearly observed that the PL intensity is almost completely quenched after the addition of BQ, whereas there is a very small decrease in the PL intensity in the presence of Py. Knowing that BQ is an electron quencher and Py, a hole quencher, the data suggests that electron extraction from CdSe NPLs is easier compared with extraction of holes. The lifetime decay (Figure 4b) becomes faster in the presence of BQ, whereas there is hardly any change in the presence of Py. Similar trends, which are seen in Figure 4c,d, are observed with the CdSe/ZnSe DoP nanostructure having quasi-type-II band alignment. The presence of ZnSe providing the additional delocalization of hole over the entire structure results in a slower recombination of charge carriers. One must keep in mind that the CdSe surface is also accessible to the quencher as ZnSe just decorates the edges and does not encapsulate the entire CdSe surface. It is thus seen that the electrons are accessible but the holes are difficult to extract from these nanostructures. This is shown schematically in Figure 5. Further, in future, ultrafast time-resolved measurements of the excited state will enable to pin down the states responsible for trapping the holes.

Following from the above results, we have carried out the photocatalytic dye degradation experiments taking methylene blue (MB) and rhodamine B (Rhb) as model compounds. The CdSe/ZnSe DoPs exhibit a higher catalytic activity in comparison to that of the CdSe NPLs. Also, these nanostructures selectively degrade MB and not RhB. The data and discussion have been presented in SI.

### CONCLUSIONS

We report a new system, CdSe/ZnSe DoPs, exhibiting a quasi-type-II structure, based on CdSe NPLs. The DoPs have been well characterized for optical and structural aspects. Nearly all of the NPLs get converted into DoPs, as evident from the disappearance of the NPL emission in these samples. The ease of electron extraction from these nanostructures is demonstrated, whereas the hole remains stuck within the trap states.

### EXPERIMENTAL SECTION

**Chemicals.** Cadmium acetate dihydrate (Cd(OAc)\(_\text{2}\))·2H\(_2\)O, 98%, Fisher Scientific), zinc acetate dihydrate (Zn(OAc)\(_\text{2}\))·2H\(_2\)O, 98.5%, Qualigens), trioctylphosphine (TOP, 90%, Sigma-Aldrich), 1-octadecene (ODE, 90%, Sigma-Aldrich), oleic acid (OA, 90%, Sigma-Aldrich), selenium powder (99.99%, Sigma-Aldrich), cadmium nitrate tetrahydrate, myristic acid (99.5%, Spectrochem), sodium hydroxide (Fischer Scientific), MB (Qualigens), and RhB (Sigma-Aldrich) were used as received. All of the solvents were dried and were purchased from Merck Chemicals.

**Preparation of Cadmium Myristate.** Cadmium myristate (Cd(Myr)\(_\text{2}\)) was prepared by following a recipe given in the literature.\(^{39}\) Cadmium nitrate tetrahydrate (1.542 g, 5 mmol) was dissolved in anhydrous methanol (50 mL). A sodium myristate solution was prepared by dissolving sodium hydroxide (0.600 g, 15 mmol) and myristic acid (3.420 g, 15 mmol) in anhydrous methanol (500 mL). Then, the cadmium nitrate solution was added dropwise into the sodium myristate solution, with vigorous stirring. After the addition of all of the cadmium nitrate solution, the reaction was continued for 30 min until completion of the reaction. The resulting white precipitate was washed with methanol three times to remove unreacted precursors and then dried at 65 °C under vacuum overnight.

**Preparation of 4 ML Thick CdSe NPLs.** CdSe NPLs (4 ML) were synthesized following a method from the literature.\(^{48}\) In a 50 mL four-neck flask, 170 mg (0.3 mmol) Cd(Myr)\(_\text{2}\) and 12 mg (0.15 mmol) Se powder were mixed in 15 mL of ODE. The
reaction mixture was degassed under vacuum at room temperature for 30 min, and then, under an argon flow, the temperature was set at 240 °C. When the temperature reached 195 °C, 40 mg (0.15 mmol) Cd(OAc)₂ was swiftly introduced into the flask. When the temperature reached 240 °C, the reaction was continued for 10 min, then quenched by the addition of 1 mL of OA, and the mixture was rapidly cooled to room temperature. Thus, the obtained product contained a mixture of CdSe QDs and CdSe nanoplatelets. At first, hexane was added to the product and centrifuged at 5000 rpm for 5 min. The supernatant containing QDs was discarded and the pellet being settled at the bottom of the centrifuge tube was dispersed in hexane. The NPLs were purified by the same procedure as described above for purification of 4 ML CdSe NPLs. The final product was redispersed in hexane and used as for coating of zinc selenide on its surface.

Preparation of 5 ML Thick CdSe NPLs. 5 ML CdSe NPLs were synthesized following a recipe from the literature.⁴⁸ In a 50 mL four-necked flask, 170 mg (0.3 mmol) of Cd(Myr)₂ in 15 mL of ODE was mixed. The mixture was degassed under vacuum at room temperature for 30 min, and then, under an argon flow, the temperature was set at 240 °C. When the temperature reached 240 °C, 1 mL solution of Se sonicated in ODE (0.15 M) was quickly injected and after 10 s, 80 mg (0.30 mmol) of Cd(OAc)₂ was introduced into the flask. The reaction was continued for 10 min then quenched by the addition of 1 mL of OA, and the mixture was rapidly cooled to room temperature. The NPLs were purified by the same procedure as described above for purification of 4 ML CdSe NPLs. The final product was redispersed in hexane and used as for coating of zinc selenide on its surface.

Preparation of CdSe/ZnSe DoPs. CdSe NPLs dissolved in 5 mL of ODE, 238 μL (0.75 mmol) OA, and 109.7 mg (0.5 mmol) zinc acetate were introduced in a 25 mL, four-necked round bottom flask and degassed under vacuum for 30 min at room temperature. Then, the reaction mixture was heated to 235 °C under Ar flow. When a reaction temperature of 235 °C was attained, 50 μL of 1 M TOPSe (0.05 mmol Se) in 1 mL of ODE was swiftly injected into the reaction mixture. After 15 min, the reaction was quenched by adding 0.5 mL of OA and was cooled to room temperature in water bath. The product is dispersed in hexane and purified with ethanol followed by centrifuging twice and finally dispersed in hexane for further characterizations.

Characterization. The CdSe NPLs and CdSe/ZnSe DoPs were dispersed in hexane and used for further characterizations. The absorption spectra of these samples were collected on a Perkin-Elmer Lambda 1050 UV/vis/NIR spectrophotometer in a quartz cuvette. The PL emission spectra of the same solution were recorded on a Perkin-Elmer LS-55 spectrophotometer, with the excitation and emission slit widths fixed at 5 nm each, at an excitation wavelength of 377 nm. The lifetime decay curves were recorded at the PL peak maximum for each sample using time-correlated single photon counting on Edinburgh Instruments FLSP920. A pulsed laser diode (377 nm) with a pulse repetition rate of 500 kHz was employed for the lifetime measurements. The instrument response function was determined using a Ludox scattering solution in water. All of
the decay curves were fitted multieexponentially using the equation, \( I(t) = \sum \alpha_i e^{-t/\tau_i} \), where \( I(t) \) is the total intensity remaining at time \( t \), \( \alpha \), and \( \tau \) are the amplitude and decay time of \( i \)th component, respectively. The average lifetime of the sample is measured using the equation \( \tau_{av} = \sum (\alpha \tau_i^2 / \alpha \tau) \). The PXRD data were collected on a Bruker D8 Advance Diffractometer equipped with Ni-filtered Cu Kα radiation. For PXRD, the sample was prepared by evaporation of the solvent from concentrated dispersions directly onto the glass sample holder. TEM images and EDAX were taken on a JEOL JEM 2010 electron microscopy using 200 kV electron source. Samples were prepared on 200-mesh carbon-coated Cu grids by dropping the solution of NCs dispersed in hexane.

**PL Quenching of Nanostuctures.** CdSe NPLs and CdSe/ZnSe DoPs were dispersed in dried chloroform. The steady state and time-resolved PL measurements were carried out with and without quencher. Here, equimolar para-BQ and Py were introduced as electron and hole quenchers, respectively.49,50

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**NOTES**

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