Exciton recycling via InP quantum dot funnels for luminescent solar concentrators

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ABSTRACT
Luminescent solar concentrators (LSC) absorb large-area solar radiation and guide down-converted emission to solar cells for electricity production. Quantum dots (QDs) have been widely engineered at device and quantum dot levels for LSCs. Here, we demonstrate cascaded energy transfer and exciton recycling at nanoassembly level for LSCs. The graded structure composed of different sized toxic-heavy-metal-free InP/ZnS core/shell QDs incorporated on copper doped InP QDs, facilitating exciton routing toward narrow band gap QDs at a high nonradiative energy transfer efficiency of 66%. At the final stage of non-radiative energy transfer, the photogenerated holes make ultrafast electronic transitions to copper-induced mid-gap states for radiative recombination in the near-infrared. The exciton recycling facilitates a photoluminescence quantum yield increase of 34% and 61% in comparison with semi-graded and ungraded energy profiles, respectively. Thanks to the suppressed reabsorption and enhanced photoluminescence quantum yield, the graded LSC achieved an optical quantum efficiency of 22.2%. Hence, engineering at nanoassembly level combined with nonradiative energy transfer and exciton funneling offer promise for efficient solar energy harvesting.

KEYWORDS
energy transfer, indium phosphide, quantum dot, light harvesting, luminescent solar concentrator, luminescent solar concentrators (LSC)

1 Introduction
The energy delivered by the Sun to the Earth in one hour (4.6 × 10^20 Joules) is close to the global annual energy consumption [1]. Efficient collection of solar photons and subsequent conversion to electricity can significantly decrease the demand for fuel-based energy and lead to a clean and sustainable energy solution that can have positive impact on environment, economy, and health. For that, luminescent solar concentrators (LSCs) integrated with photovoltaics (PV) hold significant potential [2–6]. LSCs can advantageously absorb large-area sunlight, and the down-converted light is propagated by total internal reflection into the small-area edges, where light energy is converted into electrical energy by PV cells [7, 8]. Due to the capability of large-area light harvesting and the use of small-area PVs, LSCs can reduce the operational cost of PVs and their turn-over time [9]. Moreover, LSCs utilized as semitransparent photovoltaic windows can be a viable solution for net-zero-energy buildings in urban areas [10].

Quantum dots hold significant promise for LSCs because of their beneficial properties, such as high quantum yield, good photostability and strong absorption [11, 12]. A variety of techniques at the quantum dot and device level were investigated to increase the external quantum efficiency (EQE) of LSCs [4, 10, 13–15]. At the quantum dot level, Type-II core/shell heterojunctions [16–18], layered hybrid metal halide perovskites [19], coordination engineered perovskites [20], indirect band gap quantum dots [21], alloyed quantum dots (QDs) [22], and QDs doped with transition-metal ions [23–25] were reported to suppress losses originating from reabsorption and increase photoluminescence quantum yield (PLQY). At the device level, direct incorporation of QDs into polymeric waveguides [6, 16, 17], doctor-blade deposition of quantum dots as single QD-layer [9] and tandem architectures [26, 27] have been reported for the development of low-loss LSCs.

In this study, we nanoengineered LSCs at quantum dot assembly level and formed toxic-heavy-metal-free band gap gradient to enhance the energy harvesting efficiency. The energy funnel directs photoexcited charge carriers from smaller-toward larger-sized InP-based QDs, which recombine radiatively in localized excited states of copper-doped InP-based QDs at the final stage of non-radiative energy transfer. The exciton
recycling takes place by resonance transfer of both radiative excitons and trapped excitons from large-gap QD layer to small-gap QD layer. At the final step, the resulting near-IR emission is guided to the PV module for electricity generation (Fig. 1).

![Figure 1](image)

Figure 1 Design principle of light harvesting energy-gap gradient LSC using indium phosphide QDs. An LSC is composed of four layers of graded-gap InP QDs on a glass. The nonradiative energy transfer occurs from green-emitting to yellow-emitting (n1), yellow-emitting to red-emitting (n2), and red-emitting to near-IR emitting (n3) InP QDs. Both radiative excitons and trapped excitons are resonantly transferred to luminescent state of the larger QDs that lead to the increase of the in-device PL efficiency. Solar irradiation (Pin) is absorbed by the luminoophores (in our case, InP multilayers), down-converted to the near-IR emission, and waveguided to the edge of the LSC (Pout) for electricity production.

2 Results and discussion

As the construction blocks of the graded-gap nanoassembly, we synthesized InP core quantum dots surrounded by a ZnS shell (see the Electronic Supplementary Material (ESM)) for the detailed synthesis procedure). We initially formed InP cores using hot-injection method as we reported previously [16, 28–31], and potential traps due to surface dangling bonds – cores using hot-injection method as we reported previously [16, 28–31], and potential traps due to surface dangling bonds –

\[
E = \frac{1}{1 + \left(\frac{R^d}{R_0}\right)^2}
\]

To tune the band gap of InP/ZnS core/shell QDs, the growth temperature was varied from 220 to 280 °C. Thus, the mean particle size was increased from 2.71 ± 0.29 to 3.29 ± 0.29 nm and 3.8 ± 0.66 nm for green-, yellow- and red-emitting InP/ZnS core/shell QDs, respectively (Figs. 2(f)–2(h)). Moreover, for the introduction of the mid-gap states, host InP cores were doped with copper ions using well-known thermal decomposition method [12, 36]. Previously, we have synthesized copper doped InP core QDs and optimized their synthesis parameters [12]. Then, the copper to indium ratio was set as 1:100 to achieve maximum PLQY [12]. For the copper-doped InP/ZnS core/shell QDs, the particle size is 3.9 nm (Fig. 2(i)), which is slightly higher than the red-emitting QDs, due to the incorporation of copper-ions into the lattice [36]. QDs showed an increase in absorption below their 1S-1S excitonic transition energies, due to additional electronic transitions at higher energy levels (Fig. 2(a)). The green-, yellow- and red-emitting quantum dots showed PL peaks at 2.39, 2.18 and 2.00 eV, respectively (Fig. 2(b)). Significant PL redshift and broadening occurred upon incorporation of copper into the InP QDs (Fig. 2(b)), which enabled a large Stokes shift above 600 meV, i.e. between the first excitonic absorption and photoluminescence peaks of copper-doped InP QDs (Fig. 2(c)).

To investigate the dynamics of the copper-induced hole acceptor state (Fig. 3(a)), we used an ultrafast pump-probe spectrometer and measured the non-linear absorption variation of QDs at picosecond time scales. In the experiments, free charge carriers were first generated by using a femtosecond pump pulse at 320 nm with a fluence of 240 μJ·cm−2 and the resulting change ΔA in the nonlinear absorbance spectrum (ΔA = A_pumped − A_unpumped, A_pumped = absorbance spectrum of the pumped sample and A_unpumped = small-signal absorbance spectrum) was measured by using a femtosecond white-light probe covering the 430–800 nm spectral window. Over a time interval of 3.8 ps, the peak wavelength of the ΔA spectrum for doped-QDs shifted from 547 to 568 nm, indicating an energy difference of 84 meV (Fig. 3(b)), due to the relaxation of the hot excitons down to the band edges [37]. The ultrafast transient evolution of the nonlinear absorbance up to a delay of 80 ps (Fig. 3(c)) features an initial growth time τ1 (τ1 = 3.3 and 2.4 ps in undoped and doped QDs, respectively) due to the relaxation of the hot excitons to the band edges, followed by a bi-exponential decay characterized by decay times τ1 and τ2. The decay times τ1 and τ2 describe fast decay due to intermediate trapping states, as observed in earlier studies [38, 39]. In addition, a third longer decay exists over ns time scales due to inter-band electron-hole recombination. The measured ultrafast transition decay times of copper-doped InP QDs (τ1 = 2.5 ps, τ2 = 35 ps) at the probe wavelength of 570 nm (Fig. 3(c)) show higher electronic transition rates in comparison with undoped InP QDs (τ1 = 3.4 ps, τ2 = 52 ps) (Fig. S1 in the ESM) due to copper-induced mid-gap states. The role of copper-induced mid-gap states in increasing the decay rates is further elucidated in the contour plots (Fig. S2 in the ESM), which show the variation of ΔA (nonlinear absorbance) as a function of the probe wavelength and probe delay. As can be seen from Fig. S2 in the ESM, both ultrafast decay components become shorter for the copper doped InP QDs due to the formation of the copper-induced mid-gap states.
We constructed a graded-gap nanoassembly made of InP/ZnS core/shell QDs on a glass substrate. Layer-by-layer assembly has enabled the fabrication of graded nanoassemblies, i.e., the controlled assembly of layers of QDs, such that the layers have a gradation in energy gaps, as demonstrated in the fabrication of QD-based device applications ranging from solar cells [40–43] to biointerfaces [28]. Our QDs were assembled to produce an energy gap gradient from the top layer down to the substrate via spin-coating [28, 40, 44], starting from 2.38 eV emitting in the green and ending with 1.71 eV emitting in the near-IR, due to the copper states (Fig. 4(a), bottom panel). The thickness of each QD layer is determined to be around 14–24 nm according to our previous study [28].

To investigate the effect of energy transfer on optical properties, two different control groups, including semi-graded and ungraded nanoassemblies, were also fabricated (Fig. 4(a)). In the semi-graded device, three layers of red-emitting QDs were implemented on the Cu:InP/ZnS QDs (Fig. 4(a), middle panel).
and in the ungraded structure, all four layers consisted of Cu:InP/ZnS QDs (Fig. 4(a), top panel). The ungraded nanoassembly showed higher absorbance than the semi-graded and graded nanoassemblies, due to having more copper-doped InP QD layers, which have higher extinction coefficient in comparison with the other InP/ZnS core/shell quantum dots [45] (Fig. 4(b)). Moreover, the semi-graded nanoassembly has higher absorbance than graded nanoassembly due to the higher red-emitting layers. Device absorbance ($\eta_{abs}$) is defined as the ratio of the solar photons that are absorbed by the LSC fluorophores [10]; the device absorbance of the ungraded and semi-graded LSCs, which correspond to 5.16% and 5.03%, respectively, are higher than that of the graded LSC, which absorbs 4.91% of sunlight (Fig. 4(c)). Despite having lower absorbance, the graded nanoassembly shows significantly increased photoluminescence in the near-IR spectral region compared to inhomogeneously broadened PL of semi-graded and ungraded nanoassemblies (Fig. 4(d)). These results confirm exciton recycling in the narrow band-gap QD layer (i.e., Cu:InP/ZnS QD). The exciton recycling relies on two mechanisms: first, stepwise radiative exciton transfer from large-gap QD layers to small-gap QD layers and second, recovery of trapped excitons from large-gap QD layers to the small-gap QD layer [46]. In other words, electron-hole pairs trapped in defect states of the green-, yellow-, and red-emitting QD layers were recycled for eventual recombination in the Cu:InP/ZnS QD layer. Hence, the graded nanoassembly can emit with 61% stronger intensity, due to the super-efficient nonradiative energy transfer and exciton recycling effect. Moreover, the optical PL efficiency of nanoassemblies was measured in an integrating sphere (see the ESM) and showed a significant increase from 22.1% for the ungraded nanoassembly to 35.6% for the graded nanoassembly, due to implementation of a step-wise energy gradient profile and exciton recycling [28, 46–50] (Fig. 4(e)).

We also studied time-resolved fluorescence dynamics of the graded nanoassembly using donor fluorescence lifetime in the absence of acceptors, and for that we fabricated a donor control group consisting of four layers of only green-emitting InP/ZnS QDs. Nanoassemblies were excited with a 375 nm pulsed laser, and their decay analyses were performed (Fig. 4(f)). The average lifetime ($\tau_{avg}$) was calculated from an amplitude weighted mean [30] (Eq. (3)), and it decreased from 7.18 ns ± 0.34 (for the donor control group) to 2.41 ns ± 0.12 (the graded nanoassembly). The energy transfer efficiency was calculated based on Eq. (4) [51], in which $\tau_D$ and $\tau_{DA}$ were the donor lifetime of the donor control group and of the graded structure, respectively; the energy transfer efficiency corresponds to 66% for the graded architecture. This result shows that exciton accumulation in the Cu:InP/ZnS QDs is due to efficient nonradiative energy transfer. Because of enhanced emission characteristics and higher optical transmission, the graded nanoassembly is the best candidate for luminescent solar energy harvesting.

$$
\tau_{avg} = \frac{A_1\tau_D^2 + A_2\tau_D^2}{A_1\tau_D + A_2\tau_D}
$$

$$
E = 1 - \frac{\tau_{D}}{\tau_{DA}}
$$

We constructed a graded-gap LSC on a commercial 6 cm × 6 cm × 2 mm glass substrate. The optical properties of the LSC were investigated to evaluate its suitability for solar energy harvesting. The LSC has a high- level of light transmission in the visible spectral window (Fig. 5(a)) that well suited for solar windows. In order to investigate the optical stability of the fabricated nano-assemblies, the PL efficiency of the device with graded architecture was measured at different time intervals.
The optical performance of graded luminescent solar concentrators was investigated. They were illuminated orthogonally to the front surface by a calibrated solar simulator of AM 1.5 G, and the photoluminescence generated by the QDs was coupled to the silicon solar cell positioned at the single collection edge. The EQE was calculated using the expression in Eq. (5), where $I_{SC}$ is the short-circuit current of the solar cell, when the solar cell is directly illuminated by the solar simulator, $I_{PV}$ is the short-circuit current of the solar cell, when the solar cell is directly illuminated by the solar simulator without the LSC. Based on the measurements, the EQE of the graded LSC was 1.68%, which corresponded to a relative increase of 33.3%, compared to a semi-graded LSC having an EQE of 1.26%, due to the stepwise band-gap profiles in the graded LSC. To further increase the EQE level, nanoantennas that can harvest energy from thousands of donors to a single acceptor at molecular level and integration of back-reflectors or solar cells to all four edges of LSC can be utilized [12, 52]. Moreover, the optical quantum efficiency, which is defined as the fraction of the absorbed photons to the photons reaching the output faces [10], corresponded to 22.2% (Eq. (S2) in the ESM). This is higher than the $\eta_{OQE}$ of semi-graded LSC of 16.3%. Comparatively, the enhancement of optical quantum efficiency was higher than that of the external quantum efficiency, due to enhanced fluorescence efficiency while preserving lower absorbance [10].

$$\text{EQE} = \frac{I_{SC} \times A_{PV}}{I_{PV} \times A_{LSC}}$$ (5)

### 3 Conclusion

In summary, we have fabricated energy gradient QD nanoassemblies, based on InP/ZnS core/shell QDs emitting at green, yellow and red and copper-doped InP core/shell QDs emitting in the near-IR. The presence of the copper mid-gap states in the near-IR region was confirmed by ultrafast transient
absorption spectroscopy. The step-wise energy gradient architecture led strong exciton accumulation in the copper-doped InP/ZnS QDs, which facilitated significant optical quantum efficiency increase of LSCs due to the super-efficient energy transfer and exciton cooling. This work demonstrates that the strategy of nanoassembly engineering of QDs holds significant promise for efficient solar energy harvesting.

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