

## Electronic Supplementary Information (ESI)

### **Facile fabrication of two-dimensional inorganic nanostructures and their conjugation to nanocrystals**

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#### **Preparation of inorganic nanocrystal solutions**

**Preparation of dihydrolipoic acid (DHHLA)-capped Au nanoparticle (NP) solution** 20  $\mu\text{mol}$  hydrogen tetrachloroaurate hydrate ( $\text{HAuCl}_4$ ) was dissolved in 10 mL deionized (DI) water with stirring at room temperature and the pH was adjusted to 7.2 using 2 M NaOH aqueous solution. 1  $\mu\text{mol}$  ( $\pm$ )- $\alpha$ -lipoic acid ( $\text{C}_8\text{H}_{14}\text{O}_2\text{S}_2$ ) and 40  $\mu\text{mol}$  sodium borohydride ( $\text{NaBH}_4$ ) were added to the solution and the solution was stirred at room temperature for 3 h. For preparing an additional aqueous DHHLA solution, 4  $\mu\text{mol}$  ( $\pm$ )- $\alpha$ -lipoic acid and 8  $\mu\text{mol}$  sodium borohydride was dissolved in 0.8 mL DI water at room temperature for 30 min. The aqueous DHHLA solution was added to the reaction solution. The reaction solution was stirred at room temperature for 3 h. After the reaction, the solution was dialyzed three times using Amicon ultra 30 kDa Mw cutoff centrifugal filters for purification.

**Preparation of thioglycolic acid (TGA)-capped CdSe quantum dot (QD) solution** TGA-capped CdSe QDs were prepared following a method described in the literature, with slight modifications.<sup>S1</sup> 2 mmol cadmium chloride hemi(pentahydrate) ( $\text{CdCl}_2 \cdot 2.5\text{H}_2\text{O}$ ) was dissolved in 100 mL DI water in a three-neck flask, and 0.5 mL TGA was added while stirring. The solution was adjusted to pH 11.0 with NaOH solution. 0.1 g  $\text{NaBH}_4$  and 1 mmol selenium dioxide was added successively into the solution. The solution was then refluxed at 100°C for 20 min. After the reaction, the solution was dialyzed three times using Amicon ultra 30 kDa Mw cutoff centrifugal filters for purification.

**Preparation of CdSe/CdS/ZnS (core/shell/shell) QDs solution** CdSe/CdS/ZnS QDs were prepared by the following procedures.<sup>S2</sup> CdSe bare nanocrystals were prepared by the following procedures. To obtain a cadmium precursor, 1.2 mmol cadmium acetate was dissolved in 6.0 mmol oleic acid at 100°C under vacuum. The solution was cooled to room temperature, then the cadmium precursor solution was mixed with selenium precursor. The selenium precursor was previously prepared by dissolving 6.0 mmol selenium shots in 6 mL trioctylphosphine (TOP) in a glove box. 40 mL 1-octadecene (ODE) and 6 mmol oleylamine

were placed in a three-neck flask and heated to 300°C under nitrogen gas flow. At this temperature, the mixture of cadmium and selenium precursors was quickly injected into the reaction flask and the temperature was maintained at 280°C. The reaction mixture was kept stirred until CdSe nanocrystals of the desired size were obtained. Upon completion, the mixture was cooled to room temperature and diluted by hexanes. For purification, the product mixture was precipitated by adding excess methanol, collected by centrifugation, and redispersed in a small amount of hexanes. CdS and ZnS shells were deposited onto CdSe bare nanocrystals by the following procedure. To obtain the cadmium precursor, 0.3 mmol cadmium acetate was dissolved in 1.5 mmol oleic acid at 100°C under vacuum. The solution was cooled to room temperature, then the cadmium precursor was mixed with sulfide precursor. The sulfide precursor was previously prepared by dissolving 45  $\mu\text{L}$  bis(trimethylsilyl)sulfide in 3 mL TOP in a glove box. For precursor of zinc and sulfide, 130  $\mu\text{L}$  diethylzinc and 240  $\mu\text{L}$  bis(trimethylsilyl)sulfide were dissolved in 5 mL TOP. 45 mL ODE was placed in a four-neck flask. Under nitrogen gas flow, bare CdSe nanocrystals ( $9.0 \times 10^{-4}$  mmol) were added to the reaction flask. When the temperature of the reaction flask reached 120°C, the mixture of Cd and S precursors was slowly added using a syringe pump. After allowing 30 min for the CdS shell growth, the temperature was raised to 140°C then the mixture of Zn and S precursors was added dropwise. The temperature was maintained with stirring for 30 min to allow ZnS shell growth. Upon completion, the mixture was cooled to room temperature and diluted by hexanes. For purification, the product mixture was precipitated by excess methanol, collected by centrifugation, and redispersed to a small amount of chloroform. The CdSe/CdS/ZnS QDs were ligand exchanged by sulfonate surface ligands.<sup>S3</sup> Excess amount ( $10^6$  times excess of the moles of QD) of the surface ligands (oxidized form) were dissolved in DI water. 2 equimolar sodium borohydride was added to the solution and vigorously stirred for 30 min under  $\text{N}_2$  gas flow at room temperature. The QD chloroform solution was mixed to the aqueous solution and further stirred for 4 h at room temperature. When QDs in chloroform were introduced to the disulfide-ligands in the aqueous solution, no active sodium borohydride were thought to present in the solution considering the reactivity and decay properties of the reductant. As the ligand exchange proceeds, the QDs were transferred from the organic chloroform layer to the aqueous layer. To remove excess free surface ligands, the QD solution was dialyzed twice using Amicon ultra 30 kDa MW cutoff centrifugal filters.

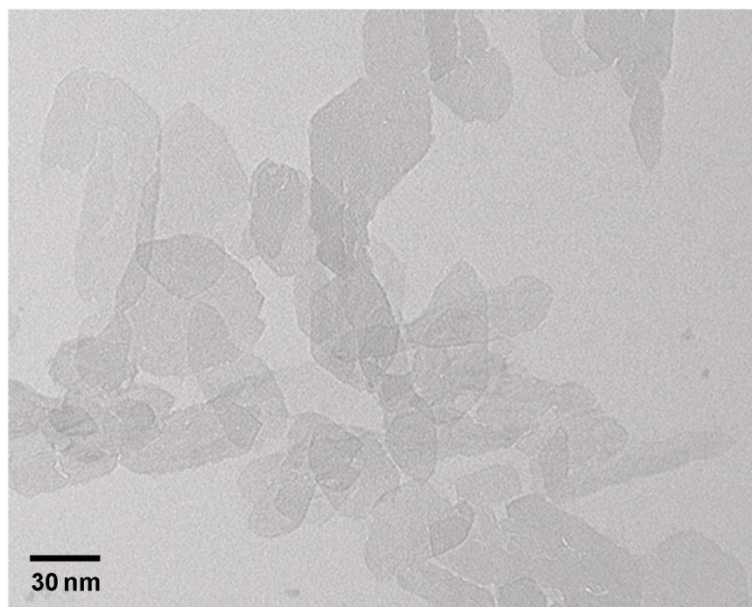
**Preparation of cetrimonium cations and poly(styrene sulfonate) polymers (PSS)-capped Au nanorods (NRs) with solution** Au NRs were prepared following a seedless growth method described in the literature, with slight modifications.<sup>S4</sup> A 20 mM aqueous solution of  $\text{HAuCl}_4$  (125 mL, 2.5 mmol) was mixed with 5 mL aqueous solution of cetrimonium bromide (CTAB) (0.162 g, 0.44 mmol) and stirred for 10 min. Then, a 10 mM aqueous solution of  $\text{AgNO}_3$  (40 mL, 0.4 mmol) was added to the mixture and stirred for 5 min, followed by addition of 100 mM ascorbic acid (30 mL, 3 mmol). The color of the solution abruptly changed from orange to transparent as the gold ion was reduced from  $\text{Au}^{3+}$  to  $\text{Au}^+$  by ascorbic acid. Finally, a 1.6 mM aqueous solution of  $\text{NaBH}_4$  (2 mL, 3.2 nmol) was added to the reduced-gold ion solution and vigorously stirred overnight. The resultant Au NR solution was centrifuged at 14000 rpm for 30 min to get rid of excessive free CTAB molecules. The supernatant was discarded. The 1 mg/mL PSS solution of 200 mL and 1 mM NaCl solution of 20 mL were added and stirred for 1 h. The Au NR solution was centrifuged at 14000 rpm for 30 min. The sediments were redispersed in DI water.

**Preparation of DHLA-capped CdSe NR solution** 0.4 mmol of cadmium oxide ( $\text{CdO}$ ), 0.8 mmol of tetradecylphosphonic acid (TDPA) and 1.615 g of trioctylphosphine oxide (TOPO)

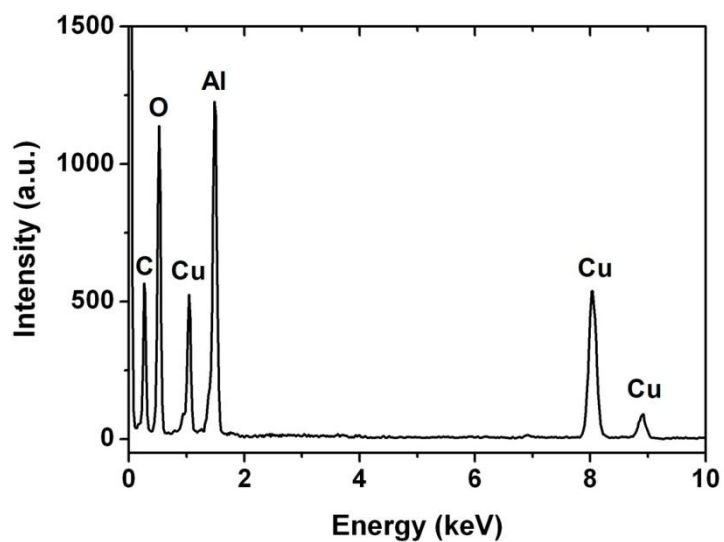
were placed in a three-neck flask. The flask was under vacuum at 110°C for 2 h, and then heated to 320°C under nitrogen for 30 min. The reaction mixture was cooled to 120°C and evacuated for 1 h to remove water. When the flask was heated back up to 320°C, 0.4 mmol of trioctylphosphine selenide (TOPSe) was injected into the flask. The nanocrystals were grown for 45 min at 315°C. After cooling, the CdSe NRs were washed several times by adding hexane and methanol followed by centrifugation to precipitate the NRs. The supernatant was removed, and the precipitated NRs were redispersed in chloroform. The CdSe NRs were ligand exchanged by DHLA. The NRs, dissolved in chloroform, were added to DHLA ( $10^5$  times excess of the moles of NR). DHLA was generated in situ by reduction of ( $\pm$ )- $\alpha$ -lipoic acid in chloroform with  $\text{NaBH}_4$ . The mixture was shaken for 30 min. 1 mL DI water was added and stirred vigorously until the organic layer became colorless. Excess free ligands were removed by repeated centrifugal dialysis using Amicon ultra 30 KDa Mw cutoff centrifugal filters.

## References

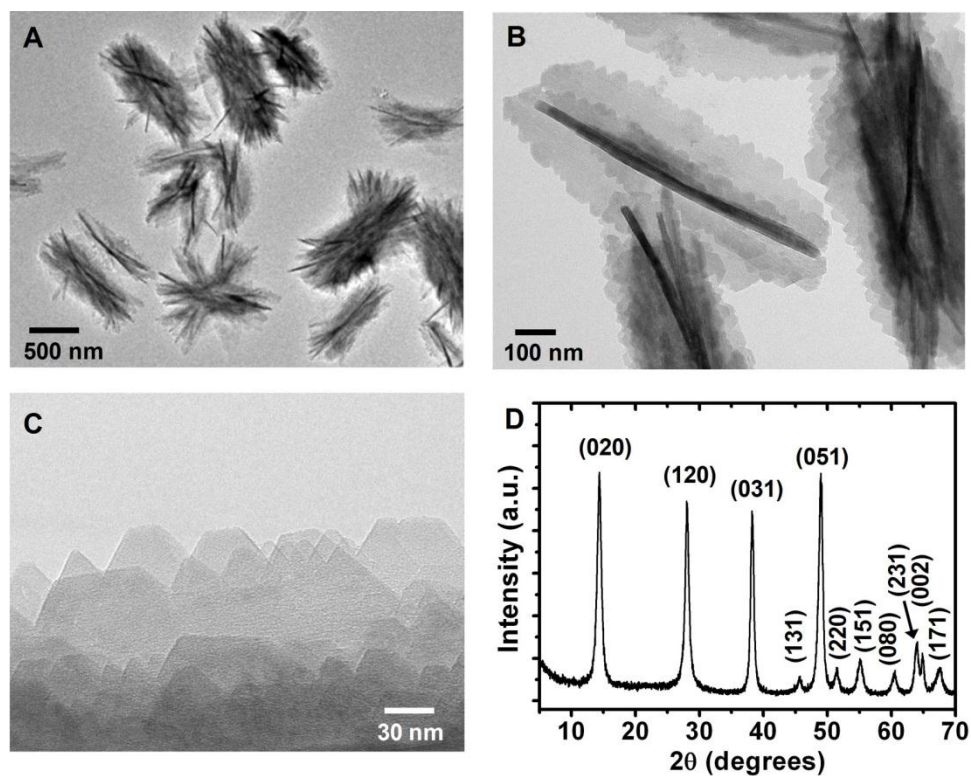
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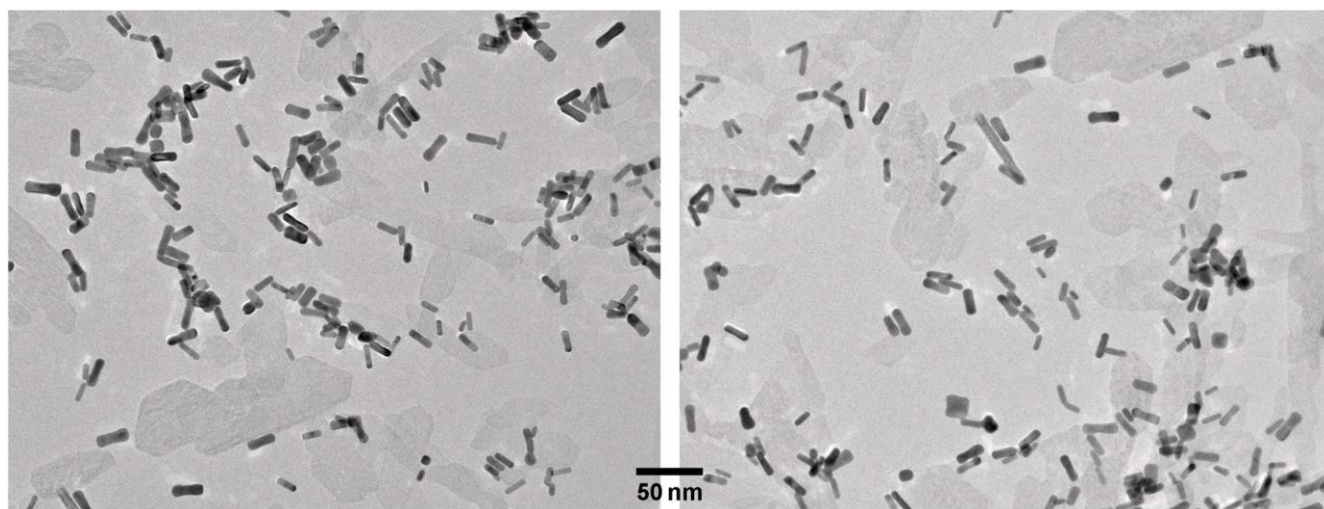
**Figure S1.** TEM image of the structures synthesized from the reaction of 3 mM aluminum nitrate nonahydrate, 0.05 M urea, 35% hydrogen peroxide solution at 220°C for 24 h.



**Figure S2.** EDX spectrum of the structures synthesized from the reaction of 3 mM aluminum nitrate nonahydrate, 0.05 M urea, 35% hydrogen peroxide solution at 220°C for 24 h.



**Figure S3.** (A, B and C) TEM images of structures synthesized from the reaction without  $\text{H}_2\text{O}_2$ . (D) XRD pattern of the synthesized powders.



**Figure S4.** TEM images of products of the mixing (vortexing) of Au nanorods capped with cetrimonium cations and  $\gamma\text{-AlOOH}$  for 1 h at room temperature.