

## Simple Preparation Strategy and One-Dimensional Energy Transfer in CdTe Nanoparticle Chains

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One-dimensional aggregates of CdTe nanoparticles were prepared by an exceptionally simple method of self-assembly initiated by partial removal of the stabilizing shell. The driving force of the particle self-organization is likely to be the dipole–dipole attraction between the nanoparticle cores. The obtained nanoparticle chains can be easily transferred on any substrate. Steady-state and time-resolved luminescent spectroscopies revealed strong Forster resonance energy transfer (FRET) between the particles, which led to the migration of excitation along the chain similarly to the waveguiding of light observed for chains of metal nanoparticles. The efficiency of FRET quenching in this one-dimensional system is comparable to that in three-dimensional packed nanoparticle solids despite a substantially smaller number of adjacent particles. The strong coupling of the donor and acceptor excited states is likely due to short inter-nanoparticle distances and partial ground-state dipole alignment taking place during the chain formation.

Organized nanoparticle (NP) assemblies reveal fundamentally interesting collective physical properties<sup>1,2</sup> and different effects that can be put in the foundations of new technologies in optics and electronics. For instance, one-dimensional (1D) arrays of semiconductor NPs make theoretically possible the directional transfer of photons and/or electrons, which, in turn, can interface different nanoscale devices<sup>1,3</sup> and serve as a transduction mechanism for (bio)sensors. However, the practical preparation of 1D assemblies of nonmagnetic NPs revealed several challenges. Unlike two-dimensional (2D) and three-dimensional (3D) arrays,<sup>4–11</sup> 1D assemblies typically require templates such as nanotubes, nanowires, polymer/DNA chains, or inorganic crystals.<sup>12–16</sup> Long chains of metal NPs templated by lattice plane terraces were made using this technique.<sup>17,18</sup> Similar linear superstructures of a lower degree of organization were also seen as a result of the specific interaction of organic stabilizers on the NP surface.<sup>19</sup> As applied to semiconductor NPs, one of the challenges is the considerable spacing between them in 1D assemblies, which often prohibits efficient electronic and energy transfer along the chain.<sup>13</sup> Another challenge is the electronic coupling between NPs and templates, which strongly affects their energy levels and diminishes the quantum confinement effects.<sup>14</sup> Variability of the substrates, and therefore, the degree of coupling, are the degrees of freedom much needed for fundamental studies of transport phenomena in NP assemblies.<sup>20</sup>

In this Letter, a method of preparation of NP chains via their spontaneous organization is reported. The technique is exceptionally simple and does not require any templates. The self-organization process leads to closely spaced 1D superstructures of NPs, which eventually can merge into semiconductor rods.<sup>21–23</sup> The NP chains can be successfully deposited without disruption on a solid support of any nature, for instance, wide band-gap insulator, roughened gold surface, lithographically patterned substrate, etc., for subsequent studies. Of equal

importance, the particle-to-particle electronic energy transfer can be easily observed in such assemblies, suggesting surprisingly efficient 1D transfer of photons along the NP chains previously observed only in organic systems.<sup>24</sup> This energy transfer can be compared to light waveguiding previously seen for chains of metal nanoparticles. This effect originates from the sequential excitation of surface plasmons in the neighboring nanocrystals, which results in the diffusion of the light quanta along the NP chain.<sup>25,26</sup>

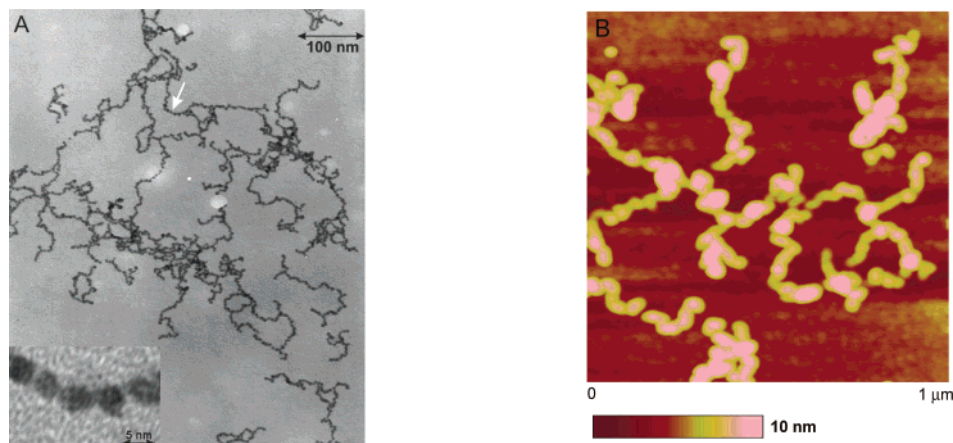
Luminescent CdTe NPs, stabilized by thioglycolic acid, were produced by the method of arrested precipitation.<sup>27</sup> They were partially stripped off the stabilizers by washing in methanol and separated from solution, as described in the Experimental Section. In a previous study,<sup>21</sup> the NP chains were observed as a fairly short-lived intermediate. It is believed that they are forming due to high dipole moments of NPs.<sup>28,29</sup> Partial removal of thioglycolic acid from the NP surface leads to the decrease of the surface charge, because the stabilizers are ionized in the basic media. The mutual electrostatic repulsion of NPs is reduced, and the dipole–dipole attraction manifests itself in the formation of the NP chains.<sup>30</sup> Transient stages of this process and involvement of other forces are still under investigation.

To make stable NP chains, the temperature during the self-assembly step of activated CdTe NPs was reduced to 4 °C. Now, the thermal energy is not sufficient enough to cause or initiate the recrystallization into rods. NPs in the prepared 1D aggregates have a mean size of 3.7 nm (10–15% size distribution) whereas the interparticle distance is  $0.5 \pm 0.2$  nm. The relationship between sizes and optical properties (Figure 2A, curve 1) of CdTe NPs (3.8 nm vs 610 nm) is also very consistent with a recent report by Yu et al.<sup>31</sup> The mean size of the particles in the chain may be varied by synthesizing starting CdTe NPs of different diameters. The chains can be easily transferred from solution to a substrate, optically inert or active, by simple dipping. Notably, the morphology of the chains deposited on the substrate is identical on both the carbon-coated transmission electron microscopy (TEM) grid surface and the silica wafers

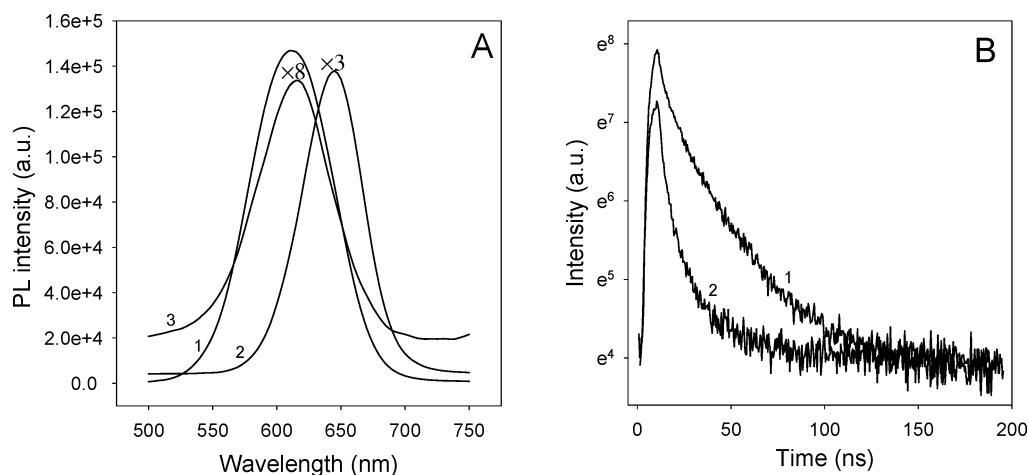
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**Figure 1.** TEM (A) and AFM (B) images of CdTe NP chains. Inset in (A) is the enlarged image of the part of the chain marked by the arrow. Single particles in (B) are not clearly resolved due to the tip broadening effect.



**Figure 2.** (A) Photoluminescence spectra for free CdTe NPs (1), NP chains (2),  $\times 3$ , and ultrasonically broken chains (3),  $\times 8$ . (B) Time-resolved photoluminescence of decays curves of free CdTe NPs (1) and NP chains (2).

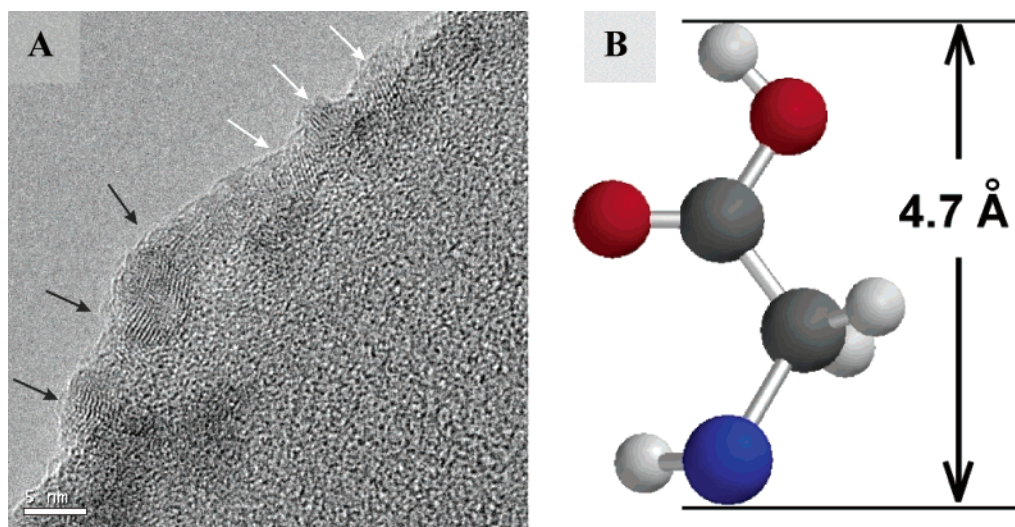
modified by poly(diallyldimethylammonium chloride), PDDA (Figure 1B).<sup>32</sup> The formation of NP chains cannot be attributed to drying of droplets because the hydrophobic TEM grid and the hydrophilic wafer have very different wettabilities.<sup>32</sup> Previously, short linear agglomerates from CdTe NPs have been observed to form at the domain borders of Langmuir–Blodgett films.<sup>33</sup> The extended linear aggregates from self-assembly process have been also seen in the case of Ag nanoparticles made in the presence of *N,N*-dimethylformamide.<sup>34</sup>

The absorption spectra of NP and their chains are virtually identical (Figure S1, Supporting Information), which correlates well with other data obtained for 2D and 3D NP assemblies.<sup>4,35</sup> The absence of the red shift of adsorption spectra affirms the constancy of the particle diameter before and after the chain formation. It also indicates that no exchange interaction between proximal CdTe NPs, such as wave function overlap and electronic delocalization, play a significant role in this system.<sup>36–38</sup> At the same time, the band-edge photoluminescence (PL) peak of CdTe NP chains (Figure 2A, curve 2) shows a strong red shift of 35 nm versus free CdTe NPs (Figure 2A, curve 1), i.e., from  $\lambda_{fl} = 610$  nm to  $\lambda_{fl} = 645$  nm. To our surprise it was noticeably greater than the red shift seen in CdTe nanowires (12–18 nm),<sup>21</sup> which is difficult to rationalize in terms of simple quantum confinement effects. Additionally, the PL peak for NP chains is significantly narrower: the half-maximum width decreases by 22 nm from 75 nm in free NP: 75 to 53 nm in chains. All these features are consistent with the presence of

Forster resonance energy transfer (FRET)<sup>39</sup> arising from coupling the transition dipoles of the excited and ground states of a luminophore. Spectral changes similar to those seen in Figure 2 were previously observed for 2D and 3D NP assemblies where excitation could spread in all three dimensions.<sup>35,40–44</sup>

Calculations show that the FRET can take place at the interparticle center-to-center separations as long as 10 nm,<sup>35,40,41</sup> whereas the center-to-center distances between adjacent CdTe NPs in the nanochains are only ca. 4.2 nm ( $3.7 \pm 0.5$  nm) (Figure 1A). Considering the closeness of the particles, this process is likely to involve the direct transfer not only to the first but also to the second nearest neighbor when the resonant conditions in the closest NPs are not satisfied.<sup>30,35</sup> Note that a similar linear arrangement of dye molecules would prohibit the efficient FRET along the chain because the characteristic distance for them is 2 nm.

The NPs of relatively bigger diameters forming the chain have a narrower band gap and serve as exciton acceptors. The natural result of FRET is selective concentration of the excitation on bigger NPs. Consequently, the observed PL peak red shifts and the width of the emission peak decreases. In this framework, red shift greater than in CdTe nanowires can be understood as dominant emission from a few NPs with a diameter significantly above average acting as energy sinks in the chain. The evidence of energy transfer in the NP chains can also be seen in time-resolved luminescence measurements (Figure 2B). The PL lifetime of NP chains (Figure 2B, curve 2) is much shorter than



**Figure 3.** (A) HRTEM image of CdTe chains with NP aligned over short distances. Almost parallel orientation of identical lattice planes in trains of several NPs can be observed (two series of crystal lattice orientations can be clearly distinguished by two-color arrows). The experimental lattice fringe spacing, 0.38 nm, is consistent with the 0.374 nm separation between two (111) planes in bulk zinc blend CdTe crystal. The chains have been imaged at the edge of the graphite matrix to get the best resolution of the lattice planes. They form in solution independently of the carbon coating of the TEM grids. (B) The calculated optimized geometry of TGA (Hartree–Fock model).

that of free NPs (Figure 2B, curve 1). When the decay kinetics are treated as two-exponent curves,<sup>45,46</sup> both time constants of PL rapid and slow decay components decrease from 7.4 ns and 24.4 ns (free NP) to 4.3 ns and 19.8 ns (chains), respectively. The value of 24.5 ns registered in the parent solution here is almost equal to the emission lifetime determined for CdTe NP synthesized in identical procedure ( $\lambda_{\text{fl}} = 565$  nm), i.e., 21 ns. In closely packed aggregates, the longer component is attributed to the native radiative lifetime of the largest NPs in the ensemble. In ideal mixture of non-interacting absolutely mono-dispersed NPs of two diameters, it should presumably be the same in both solution and agglomerates, but it decreases slightly in chains because of greater statistical probability to find even bigger neighboring NP in some vicinity. The short component describes dynamics of the luminescence in smaller particles. The drastic reduction of its lifetime is due to enhanced FRET quenching of smaller particles, which becomes more efficient upon chain formation.

The quantum yield of nanochains is 3.2%, whereas the free NPs emit with a quantum yield of 13.6%. The reduction of emission efficiency is brought about by an increased probability of radiationless electron–hole recombination rising according to the power law with each FRET event.

Note that the NP chains exhibit rather strong FRET, especially when compared to closely packed 3D arrays of CdSe NPs.<sup>42</sup> Despite the decrease of the number of nearest neighbors from 12 to 2 and the number of second nearest neighbors from 44 to 4, the effect of agglomeration on the luminescence decay of the smaller NPs—the most sensitive to FRET—is comparable to that in 3D quantum dot solids reported by Crooker et al.<sup>42</sup> and Boev et al.<sup>43</sup> Lifetime of NP emission decreased from radiative 22–24 ns to 1.9 ns for CdSe 3D solids,<sup>42,43</sup> which can be compared to 4.3 ns in CdTe chains observed here. The PL red shifts between the solution and solid state are also close in values: 111 meV for CdTe chains (Figure 2) and 50–110 meV for 3D CdSe solids. One of the plausible explanations for the comparable FRET efficiency in 1D CdTe chains and 3D solids from CdSe despite substantially smaller number of neighbors can be shorter inter-NP distances in chains. Previous

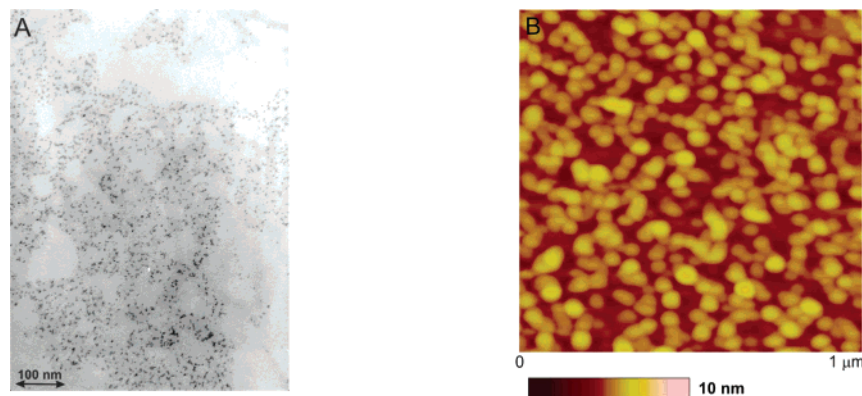
report has suggested that the FRET efficiency ( $E$ ) can be expressed as<sup>47,48</sup>

$$E = \frac{nR_0^6}{nR_0^6 + r^6} \quad (1)$$

where  $n$  is the average number of acceptor NPs interacting with one donor,  $R_0$  is the separation distance that yields 50% energy transfer efficiency, and  $r$  is the distance between donor and acceptor NPs. Obviously, the FRET efficiency is dependent not only on the number of interacting NPs but also on the distance between interacting NPs. A slight change of inter-NPs distance has a considerable effect on the FRET efficiency owing to their relationship as the sixth power. For CdSe NPs stabilized by trioctylphosphine oxide or octylamine, the closest distance between two adjacent NPs is 1.1 nm that is the length of single stabilizer molecules, when stabilizers adopt the interdigitated configuration. However, for TGA-stabilized CdTe NPs, the closest distance between CdTe NPs decreases to 0.47 nm, as calculated from Hartree–Fock ab initio model (Figure 3B), which is identical to the shortest distance between the NPs assessed from the TEM images (Figures 1A and 3A). The short separation between CdTe NPs improves the FRET efficiency and, therefore, offsets the reduction of the total number of neighbor NPs involved. Indeed, the great enhancement of FRET efficiency by shortening the inter-NP distances for CdSe 2D or 3D assemblies has recently been explored by Achermann et al. with the compression in the Langmuir–Blodgett through, for example, the lifetime of the NP 2D monolayer decreases to 50 ps.<sup>44</sup>

Several other factors may also elevate the FRET efficiency in nanochain structures. The size distribution of the CdTe NPs used here is slightly wider than that of CdSe assemblies, 10–15% vs 5%. According to ref 44, the relatively wide size distribution of CdTe NPs in nanochains may benefit the FRET process.<sup>44</sup> Additionally, because the FRET transfer rate,  $\Gamma_{\text{FRET}}$ , is proportional to  $k^2$ , where  $k$  is the orientational factor of the dipoles, prealigning of the NP with respect to each other should affect  $\Gamma_{\text{FRET}}$  very strongly.<sup>34</sup> High-resolution TEM reveals that the short-range orientation of crystal lattices of NP crystal





**Figure 4.** TEM (A) and AFM (B) images of NP chains disassembled by ultrasonication.

lattices over distances 15–30 nm appeared in CdTe chains (Figure 3A), which is a clear indication of the mutual orientation of NP with respect to each other in the chain, stemming from the permanent dipolar attraction of semiconductor NPs.<sup>28,29</sup> An analogous effect was observed by Pacholski et al. and Giersig et al. in ZnO and Ag NPs.<sup>22,34</sup> The close-range ordering of the NPs eventually translates in the complete reorientation/recrystallization to the single crystalline nanowire.<sup>21</sup> In the chains, the alignment of particle lattices is indicative of the alignment of the transitional excited-state dipoles forming during FRET transfer and enhancing its efficiency, when an excited NP is placed in the near-field of a ground-state NP. Presently, the experimental data are insufficient to analyze these details of FRET in the chains; nevertheless, the possibility of the orientation effects needs to be pointed out.

The network of NP chains in solution (Figure 1) can be disassembled by ultrasonication, as can be seen from the corresponding TEM and atomic force microscopy (AFM) images composed of only free NPs (Figure 4). After such treatment, both the position and width of the PL peak become restored to the original values (Figure 2A, curve 3). The spectral recovery of PL substantiates FRET and rules out an increase of NP diameter as a reason for observed optical effects. The delocalization of excitation resulting from FRET makes CdTe NP chains in Figure 1 an inorganic analogue of conjugated luminescent polymers demonstrating exceptional potential for sensing and information technologies.<sup>24</sup> A comparison of optical effects observed upon the polymerization of aromatic monomers is a conjugated polymer and those reported here for the assembly of single NPs into chains reveal multiple similarities and substantiate the analogy. Some similarity can also be seen with light waveguiding in the chains of noble metal NPs due to excitation of surface plasmons.<sup>25,26</sup> The diffusion length of light quanta in these chains was found to be in the range of 500 nm. The presented CdTe NP chains have two advantages over the chains of metal particles. First, the distance between the neighboring NPs in linear aggregates in Figure 1 is shorter than chains reported by Meier et al.<sup>25,26</sup> This feature facilitates energy transfer and reduces damping. Second, the control over the diameters of semiconductor NPs opens the theoretical possibility of directed energy transport in particle chains, as can be seen in the gradient NP films reported previously.<sup>9</sup> Admittedly, one needs to learn how to organize the NPs, according to their diameter in these chains, which will be the subject of our future work.

In conclusion, CdTe NP chains were fabricated and transferred onto different substrates in a simple process of spontaneous self-assembly. 1D FRET in the closely spaced NP chains has been demonstrated. This method of preparation of chains

with strong electrostatic coupling between the particles can be extended to other materials because the presence of the dipole moments is a likely attribute of different types of NPs.<sup>49</sup>

### Experimental Section

CdTe dispersions were prepared following the procedure described elsewhere.<sup>27</sup> To get rid of the excess stabilizer, as-prepared CdTe NPs were precipitated by methanol and then redissolved in water at pH 9 in air. The dispersion of stabilizer depleted CdTe NPs was kept in a refrigerator at 4 °C for 1 month. During this period the dispersion became slightly turbid, indicating the formation of 1D aggregates. Subsequently, the NP chains could be centrifuged and separated from unreacted NPs by gradual addition of isopropyl alcohol. The precipitated chains can be redissolved in water at pH 9.

The PDDA modification of Si wafer was described elsewhere.<sup>32</sup>

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**Supporting Information Available:** Figure S1. UV spectra of free CdTe NPs (1) and NP chains (2). This material is available free of charge via the Internet at <http://pubs.acs.org>.

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