

Large-Scale and Electroswitchable Polarized Emission from Semiconductor Nanorods Aligned in Polymeric Nanofibers

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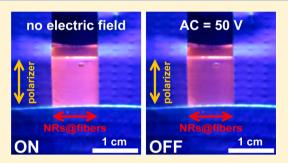
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Supporting Information

ABSTRACT: We demonstrate the fabrication of a large-scale device allowing for electrical switching of polarized light with a polarization ratio of 0.45 over a 1.5 cm² area. To achieve this, silica-coated semiconductor nanorods were embedded in polymeric nanofibers by electrospinning. The uniaxial extensional flow experienced by the particles induces alignment of the nanorods within the nanofibers. A subsequent parallel alignment of the nanorods that can conveniently be processed further. We demonstrate this by integrating the aligned nanofibers in a liquid crystal cell with a polarizer where applying a voltage allows switching the emitted light on and off.



KEYWORDS: quantum dots, microemulsion, electrospinning, liquid crystal, display, LED

C emiconductor nanostructures such as colloidal quantum dots (QDs) offer interesting optical properties, including tunable emission spectra and high photoluminescence quantum yield. These characteristics make them very promising candidates as the next generation of light emitters in a wide variety of applications ranging from biology to photonics. Anisotropic structures such as nanorods (NRs) are of further interest, as they also show polarized emission along their long axis.^{1,2} To fully take advantage of this unique property in lighting or display applications for instance, these colloidal objects need to be ordered on a large scale. To this end, several approaches and techniques have been developed to organize and align NRs. Examples involve slow solvent evaporation at a water/air interface,³ coffee stain evaporation dynamics,⁴ impregnation of 3D templates,⁵ mechanical rubbing of spin-coated NR layers,⁶ stretching of polymer films,^{7,8} or electrically driven alignment.^{9–11} Although in most of these techniques the NRs are aligned over areas in the micro- to millimeter range at best, some have already shown to be suitable for relatively large scale alignment. For instance, Amit et al. have obtained a polarization ratio of 0.55 (contrast ratio of 3.5) over a several cm² area by mechanical rubbing of CdSe/CdS NRs.⁶ We recently reported a novel technique for the alignment of CdSe/ CdS NRs by applying an alternating electric field during a dipcoating procedure, achieving a very high polarization ratio of 0.60.12 Nevertheless, these techniques can result in a heterogeneous distribution of the NRs over the substrate. In addition, the aligned NRs obtained by these techniques cannot

be conveniently manipulated and processed, which would be a great asset for display and lighting applications. As a further requirement, the NRs should also be aligned horizontally in order to be able to collect the polarized emission perpendicularly to the substrate.

In this context, a very interesting approach has been developed for the alignment of gold NRs in polymeric nanofibers made by electrospinning.¹³ During electrospinning, the polymer solution undergoes a strong uniaxial extensional flow, which aligns the NRs along the nanofiber axis and prevents rotational diffusion from randomizing the particle's orientation. This technique has proven to efficiently align even NRs with low aspect ratios.¹³ Generally, the electrospun nanofibers are collected on a conductive, grounded plate as nonwoven mats. The reason for this is the random movement of the charged jet caused by the bending instability that originates from the interactions between the electrostatic charges within the jet and between the electrostatic charges and the applied electric field. However, using different types of collectors and/or manipulating the electric field enables ordered structures to be formed, which is an additional asset of the technique.¹⁴

In this work, we employ electrospinning to produce films made of aligned polyvinylpyrrolidone (PVP) nanofibers containing silica-coated CdSe/CdS NRs oriented parallel to

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the nanofiber axis. The uniaxially aligned nanofibers were obtained by simply replacing the standard grounded plate collector with a parallel plate collector, consisting of two free-standing stripes of electrical conductors separated by a gap. When the randomly moving parts of a nanofiber get close to the edges of the conducting plates, they are "pulled" by the electrostatic forces that stretch the nanofiber across the gap and orient it perpendicularly to the parallel stripes.^{15,16} Using this type of collector we produced a free-standing film of 1.5 cm² that showed polarized emission with a large-scale polarization ratio of 0.45.

The alignment of NRs in such polymeric films offers important advantages for their use in light-emitting devices. With this approach the NRs are aligned within a protective, low refractive index matrix, whereas other techniques often produce rather compact films of NRs, which can result in a selfquenching of their emission.¹⁷ In addition, such flexible and free-standing films can be manipulated and further processed for the realization of a working device, which we also demonstrate here. Indeed, besides the large-scale alignment of NRs we go one step further toward a realistic lighting application through the integration of this film in a liquid crystal (LC) cell. Liquid crystals are electro-optic materials that are commonly used in display applications. In such applications a voltage across the liquid crystal layer is used to change the direction of the liquid crystal, which in turn has an effect on the polarization state of light passing through. It appears that the role of the aligned nanofibers not only is limited to the alignment of the NRs but can also be used to induce alignment of the nematic LC. With this approach we have fabricated a simple device emitting polarized light over a large area in which the initial direction of polarization can be switched over 90°.

CdSe/CdS Dot-in-Rods. The CdSe/CdS NRs were synthesized according to a procedure described in the literature (see the Supporting Information, paragraph 1, for the complete synthesis details).¹⁸ The first step is the synthesis of spherical CdSe QDs with a wurtzite crystalline structure. The QDs are then used as the cores for the seeded growth of an anisotropic CdS shell, resulting in a so-called dot-in-rod heterostructure. The CdSe/CdS NRs involved in this work have an average diameter of 4.1 nm and an average length of 29.3 nm (see the Supporting Information, Figure S1, for a transmission electron microscopy (TEM) image). The NRs show an emission spectrum centered at 600 nm (see the Supporting Information, paragraph 2 and Figure S2, for the optical characterization of the NRs).

Silica Encapsulation of the NRs. The CdSe/CdS NRs are typically dispersed in apolar solvents, such as toluene or chloroform, which are not suitable for electrospinning due to their incompatibility with the polymers typically used for electrospinning or their fast evaporation rate. In order to transfer them to a polar solvent (ethanol), the NRs were coated with a thin silica shell through a reverse microemulsion process. This technique has already proven to be very efficient for the silica coating of initially hydrophobic nanocrystals, including CdSe/CdS dot-in-rods.¹⁹ Here, 10 nmol of NRs was mixed with 100 mL of n-heptane (99%, VWR) and 32 mL of polyoxyethylene (4) lauryl ether (Brij L4, formerly known as Brij 30, Sigma-Aldrich). After 15 min under magnetic stirring, 5 mL of Milli-Q H₂O and 0.5 mL of NH₄OH (28% in water, VWR) were slowly added to form the microemulsion. After 1 h under stirring, 0.25 mL of tetraethyl orthosilicate (TEOS, 98%, Sigma-Aldrich) was added to the microemulsion. The reaction

was left stirring for 2 days to form the silica shell. The CdSe/ CdS@SiO₂ NRs were then further functionalized with polyethylene glycol (PEG) to provide them with enhanced colloidal stability in polar solvents and to prevent aggregation. PEGylation of the CdSe/CdS@SiO₂ NRs was done in a onepot process, following the growth of the silica shell, by adding 120 mg of methoxy-PEG-silane (mPEG-silane, $M_w \approx 1000$ g/ mol, Gelest) to the microemulsion. After 3 additional days of stirring, the reaction was stopped by adding a large volume of ethanol, resulting in the destabilization of the microemulsion and precipitation of the particles, which were collected by centrifugation. The PEGylated CdSe/CdS@SiO₂ particles were further purified twice with *n*-heptane and redispersed in ethanol. Figure 1a shows the TEM image of the CdSe/CdS@

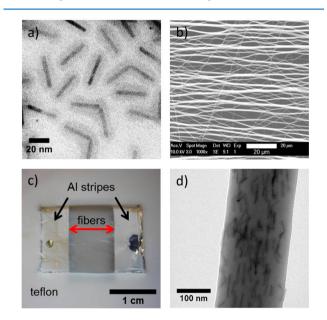


Figure 1. (a) TEM image of the PEGylated CdSe/CdS@SiO₂ NRs. (b) SEM image of the aligned nanofibers. (c) Photography of the whole aligned nanofiber sample (red arrow indicates the alignment direction of the nanofibers). (d) TEM image of a CdSe/CdS@SiO₂@ PVP nanofiber.

 SiO_2 NRs, evidencing that a homogeneous silica coating of about 6 nm in lateral thickness is formed. The rod shape of the objects is largely preserved, albeit with a reduced aspect ratio. The initial aspect ratio of 7.1 of the CdSe/CdS NRs is reduced to 2.4 for the CdSe/CdS@SiO_2 NRs. Figure 1a also shows that some of the objects actually consist of two CdSe/CdS NRs connected within the silica matrix, resulting in some cases in V shapes with an angle of 110°. The connections between rods could not be avoided in our experiments and are believed to occur already when the CdSe/CdS NRs are in their apolar solvent prior to the silica encapsulation. This assumption is supported by TEM observations of the CdSe/CdS NRs deposited on a Formvar layer prior to their silica encapsulation (see the Supporting Information, Figure S3).

Electrospinning of the Nanofibers. The NRs were mixed with a polymer by adding 240 mg of polyvinylpyrrolidone (M_w = 1 300 000 g/mol, Sigma-Aldrich) to a solution containing 4.65 nmol of the CdSe/CdS@SiO₂ NRs in 2.81 g of ethanol. Even though the NRs were coated with silica, we avoided using water since this is a rather harsh environment that tends to deteriorate the optical properties of the semiconductor NRs.¹⁹

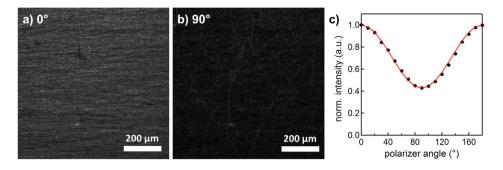


Figure 2. Fluorescence microscopy images of the aligned CdSe/CdS@SiO₂@PVP nanofibers with the polarizer oriented (a) parallel (0°) and (b) perpendicular (90°) to the nanofibers. (c) Normalized integrated intensities depending on the polarizer angle (black markers) fitted with a \cos^2 function (red line).

The relatively high molecular weight has been chosen for the ease of nanofiber formation by electrospinning.²⁰ The nanofibers were made using a climate-controlled electrospinning chamber (EC-CLI) from IME Technologies. The temperature and relative humidity were set to 25 °C and 30%, respectively. The flow rate was 0.55 mL/h (controlled with a Harvard syringe pump). The tip-to-collector distance was 15 cm, and the voltage was set to 14 kV. The nanofibers were electrospun from a needle with an inner diameter of 0.25 mm and an outer diameter of 0.52 mm. In order to obtain aligned nanofibers, a parallel plate collector was used.¹⁶ The collector consists of two aluminum stripes separated by a void gap of 1 cm wide and 1.5 cm long. In order to ensure that the nanofibers are deposited within the desired area and that they have support, the gap and the space around the stripes have been covered with Teflon sheets. With this type of collector, the electrostatic forces generated by the two conductive stripes induce a uniaxial alignment of the charged spun nanofibers across the insulating gap.¹⁵ The collected nanofibers, denoted as CdSe/CdS@ SiO₂@PVP, show a high degree of alignment, as evidenced by scanning electron microscopy (SEM) observations (Figure 1b). Electrospinning was carried out up to 5 min, producing a freestanding film of 1.5 cm² that could then be manipulated (Figure 1c). Nevertheless, the electrospinning technique used here can easily be adapted to produce even larger films by increasing the length of the stripes. The TEM image of a single nanofiber in Figure 1d shows a relatively high load of CdSe/CdS@SiO₂ NRs embedded in the polymeric nanofiber. Moreover, this image already evidences that the NRs are indeed aligned along the axis of the nanofiber. Even NRs with a V shape are found to be oriented at their best in the nanofibers, resulting in a maximum angular deviation of 35° with respect to the nanofiber axis. The TEM image in Figure 1d also shows that the NRs are evenly distributed within the fiber. The NRs do not appear to aggregate (keeping in mind that TEM observations provide only a 2D projection), which confirms the good colloidal stability of the silica-coated NRs in the polar polymeric solution and the good affinity of the polymer for their PEGylated surface. The absence of aggregation is an important parameter to avoid self-quenching of the NRs' emission in lighting applications.17

Large-Scale Polarized Emission. To measure polarized emission with a fluorescence microscope, the film of aligned $CdSe/CdS@SiO_2@PVP$ nanofibers was detached from the collector and placed between two glass slides. The nanofibers were excited with green light, using a xenon lamp with a bandpass filter (510–560 nm), in order to avoid emission from the PVP or the LC, which also absorbs in the UV range (see the

Supporting Information, Figure S4, for their optical characterization). The emitted light was collected by an objective $(10\times)$ after passing through a rotatable linear polarizer and was detected with an Andor CCD camera. One should note that in our experiments the excitation light is not polarized. This is preferred in order to avoid preferential absorption by the NRs that have the best alignment and allow for a better assessment of the true polarization ratio of the sample. Figure 2 gives the fluorescence microscopy images and integrated intensities depending on the polarizer angle. This angle is defined as the difference between the nanofibers' long axis and the polarizer axis. As clear evidence of the alignment of the NRs inside the nanofibers and of the parallel alignment of the nanofibers, a bright image is obtained when the polarizer is parallel to the nanofibers (Figure 2a) and a dark image is obtained when the polarizer is perpendicular to the nanofibers (Figure 2b). Figure 2c further evidences that the variation in intensity, when rotating the polarizer, follows a classic cos² function.^{5,6,12} From these results one can calculate the polarization ratio (PR), defined by PR = $(I_{\parallel} - I_{\perp})/(I_{\parallel} + I_{\perp})$, where I_{\parallel} and I_{\perp} are the integrated intensities when the polarizer is parallel and perpendicular to the nanofibers respectively, i.e., at 0° or 90° . One should keep in mind that the polarization ratio of a single CdSe/CdS NR with a spherical core is limited to 0.75, so that a polarization ratio of 1 cannot be achieved.^{8,21,22} From the largescale images shown in Figure 2a and b, we obtain a polarization ratio of 0.45. For measurements on individual nanofibers, the highest polarization ratio was found to be 0.55 (see the Supporting Information, Figure S5, for fluorescence microscopy images of a single fiber). This indicates that the polarization is relatively well preserved when aligning the nanofibers over a large area. The few nonaligned nanofibers, which become visible in Figure 2b when the polarizer is in the 90° position, have a negligible contribution to the overall integrated intensity. The 0.45 polarization ratio obtained here has the same order of magnitude as obtained with other techniques for the alignment of NRs at large scale. This is comparable for instance with the 0.34 obtained by Rizzo et al. through solvent evaporation at a water/air interface³ or the 0.55 obtained by Amit et al. through mechanical rubbing.⁶ Although we have previously demonstrated that polarization ratios up to 0.6 could be obtained through electrical-driven alignment,¹² the present approach offers important advantages with regard to scale-up and further processing as demonstrated in the following section. In addition, the polarization ratio of 0.45 obtained here can most likely be improved, especially if one can avoid the connection between NRs. It should also be noted that the excitation wavelengths used in the experiment (510–560 nm)

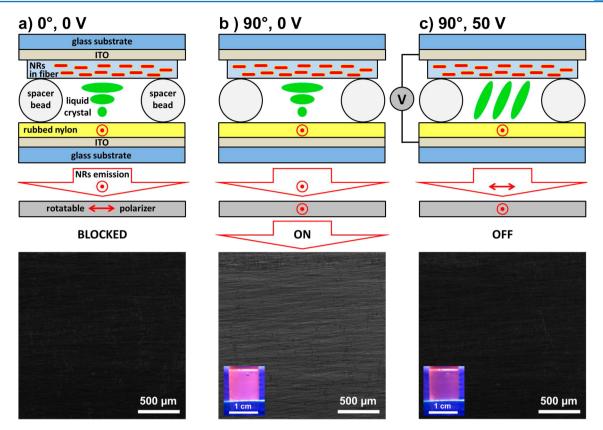


Figure 3. LC cell with CdSe/CdS@SiO₂@PVP nanofibers and corresponding fluorescence microscopy images depending on the polarizer orientation and electric field. (a) Polarizer is parallel to the nanofibers (0°) , and no electric field is applied. (b) Polarizer is rotated perpendicularly to the nanofibers (90°) , and no electric field is applied (ON-position). (c) Polarizer is maintained perpendicular to the nanofibers (90°) , and a 50 V electric field is applied (OFF-position). Insets of the fluorescence microscopy images in (b) and (c): corresponding pictures of the LC cell using a portable UV lamp for the excitation.

actually correspond to absorption by the CdSe cores in the CdSe/CdS NRs (see the Supporting Information, Figure S2, for the absorption spectrum of the NRs). This excitation range was initially chosen in order to avoid emission from the PVP and LC. However, additional experiments using excitation in the UV range (330–380 nm) also resulted in a polarization ratio of 0.45 (see the Supporting Information, Figure S6, for fluorescence microscopy images under UV excitation). This observation is in line with previous reports indicating that in such heterostructures the dipole moments—responsible for the polarized emission property—are aligned in the same direction in the CdS core and in the CdS shell.⁵

Integration in a Liquid Crystal Cell. One of the advantages of this method of aligning the NRs in polymeric nanofibers is that the resulting large-scale flexible film can be conveniently manipulated and further processed. Here, a film of aligned CdSe/CdS@SiO2@PVP nanofibers has been integrated in a liquid crystal cell, which is illustrated in Figure 3. The cell consists of two glass substrates coated with indium-tin-oxide (ITO) layers as the electrical contacts. The bottom substrate, i.e., on the side where emission is collected, is also coated with a 300 nm thick layer of mechanically rubbed nylon-66. The film of aligned nanofibers was placed in the cell so as to have a 90° angle between the alignments of the nylon layer and the nanofibers. The cell was then sealed with glue containing spacer beads of 10 μ m (Sekisui Chemicals) to ensure a uniform cell gap. An opening was left on the side of the cell to infiltrate it with a commercial E7 (Merck) LC. The director of the liquid crystal follows the alignment of the nanofibers and of the nylon

at both sides, and in between it makes a twist of 90°. This device was then analyzed by fluorescence microscopy under green excitation, with the emitted light passing through a rotatable polarizer. The different experimental conditions and the resulting fluorescence microscopy images are shown in Figure 3. The microscopy image in Figure 3a evidences that almost no light is collected when the polarizer is oriented parallel to the nanofibers (0° position). Since both the NRs and the polarizer are oriented in the same direction, this can only be explained by a 90° rotation of the initial polarization direction of the NR emission. The 90° rotation of the polarization direction is caused by the fact that the linear polarization direction is following the twist of the LC director. Indeed, for the parameters in this work the twist of the director is slow and the change of polarization is according to the Maugain regime as demonstrated in the Supporting Information (paragraph 7).²³ It confirms that the nematic phase of the LC is aligned by the nanofibers. Aligned nanofibers have already been reported to orient twisted nematic LC phases as efficiently as rubbed polymer layers.²⁴ This could be explained by the shape of the nanofibers, as waveguides and edges have been reported to align LCs.²⁵ In addition, the long polymer molecules within the nanofibers undergo a coil–stretch transition in the extensional flow of the spinning process,^{26,27} of which a residual alignment along the nanofiber axis has been reported in the solid nanofiber state,^{28,29} and could also contribute to the LC alignment similarly to a rubbed polymer layer. The fact that the same template matrix can be used for the alignment of the NRs and of the LC constitutes a considerable added value of this

approach. In Figure 3b, the polarizer is rotated perpendicularly to the nanofibers (90 $^{\circ}$ position) and therefore lets the polarized light emitted by the NRs go through. This is the "on" position. Here again a polarization ratio of 0.45 is found between the fluorescence microscopy images of Figure 3a and b, confirming that the large-scale polarized emission is not affected by the integration of the aligned nanofiber film in the LC cell. In Figure 3c an ac electric field (sine wave, 50 V, 1 kHz) is applied over the ITO layers, resulting in a change of the orientation of the LC director perpendicular to the substrates, along the electric field. As a consequence, the polarization of the light emitted by the NRs is not rotated anymore and is blocked by the polarizer. This is the "off" position. Here, a polarization ratio of 0.43 is found, which has been calculated using I_{0V} (Figure 3b) and $I_{50 \text{ V}}$ (Figure 3c) instead of I_{\parallel} and I_{\perp} , respectively. This value is very close to the 0.45 of the aligned nanofiber film itself, which shows the high efficiency of the electrical switching of this polarized light emitting device. If the polarizer is rotated to the 0° position, i.e., parallel to the nanofibers, the emitted light is collected again (see the Supporting Information, Figure S7). In this case, the amount of light collected corresponds to 77% of the "on" position, which indicates a relatively good vertical alignment of the LC director in the presence of the 50 V electric field. Nevertheless, the small decrease in collected light still suggests that the vertical alignment is not perfect, which is most likely the case at the vicinity of the alignment layers (rubbed nylon on one side, fiber film on the other). This could result in a distortion of the linearly polarized light originating from the NRs, which then becomes slightly elliptical.

In conclusion, we have successfully aligned silica-coated CdSe/CdS NRs in PVP nanofibers by electrospinning, in spite of the relatively low aspect ratio (2.4) of the NRs. Aligning the nanofibers themselves parallel resulted in a flexible film of 1.5 cm^2 showing a polarization ratio of 0.45. Such a self-supporting film, similar to a foil, can be manipulated and combined with any kind of substrate, which is an important asset of the approach undertaken here for the realization of a device. Through its integration in an LC cell we have shown that the film of aligned nanofibers can be used to induce alignment of a nematic LC. Finally, we have demonstrated that this simple device, combining aligned nanofibers and liquid crystal, constitutes an efficient way of electrically switching on and off polarized light while fully preserving the polarization ratio over a large area. The strategy developed here can readily be applied to a large variety of colloidal objects, taking advantage of the high versatility of the electrospinning technique, and paving the road for the design of novel devices where the largescale organization of nanosized building blocks matters.

ASSOCIATED CONTENT

Supporting Information

Synthesis details for the CdSe/CdS NRs, additional TEM and optical characterizations, additional fluorescence microscopy analyses, condition for the Maugain regime. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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