Forcespinning fabrication of CdTe-quantum dot/polyactic acid microfibers for novel surface-engineered application

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Abstract
The aim of this work was to prepare and characterize color-tunable luminescent nanocomposite fibers composed of CdTe quantum dots (CdTe-QDs) and polyactic acid (PLA) via nanoprecipitation. This method provides a simple procedure to incorporate QDs with various emission wavelengths onto nanofibers’ surfaces. A one-pot microwave irradiation method was employed to prepare green- to red-emitting 3-mercaptopropionic acid (MPA)-capped CdTe-QDs with high luminescence. Microfiber PLA membranes were produced via forcespinning. These nanofibers can integrate the luminescent properties of CdTe-QDs and extend their advantages to fiber-based structures. Ultraviolet-visible spectroscopy (UV–vis) and transmission electron microscopy (TEM) were used to characterize the CdTe-QDs. TEM images revealed that the CdTe-QDs were 4 nm in size. The CdTe-QDs’ effect on the microfibers were characterized morphologically via scanning electron microscopy (SEM), which showed that the average diameter of the CdTe-QDs/PLA nanofibers was 3.5 µm. They were optically analyzed by photoluminescence (PL), absorption spectroscopy, Fourier-transform infrared spectroscopy (FTIR), and confocal fluorescence microscopy. FTIR revealed strong interactions between the MAP stabilizing agents and PLA fibers. These preliminary results demonstrate that these microfibers have potential for biological labeling and diagnostics based on their properties.

1. Introduction
Quantum dots (QDs) are spherical nanoparticles with physical dimensions smaller than the exciton Bohr radius that may be produced using most semiconducting metals, including CdSe, CdS, CdTe, PbS, and ZnS with high crystallinity and water-dispersed characteristics [1, 2]. Their excellent electrical, nonlinear optical, and band gap tunability properties can change the size of particles. The larger the size, the longer the light absorbed and emitted wavelength. CdTe-QDs are important semiconductor materials with sizes ranging from 2 to 10 nm in diameter. They have been widely studied since their tunable emission changes from green to red due to quantum confinement [3]. They have also attracted extensive attention for numerous applications such as in solar cells [4], biological labels [5], photocatalysts [6], chemosensors [7], and light-emitting diodes [8].

One of these nanomaterials’ drawbacks is their inherent instability due to their high specific area. That is why they are commonly coated with organic agents during their nucleation and growth process [9]. Controlling these
materials’ growth leads to a narrow size distribution, improving their optical properties. The stabilizing agent also allows the formation of an organic shell that covers the particles. Therefore, recent studies have focused on anchoring the QDs to polymeric matrices to increase their biomedical applications [10]. This is justified because some studies have reported that QDs such as CdTe are toxic [11, 12], limiting their biomedical applications. To avoid this problem, CdTe-QDs are encapsulated with biocompatible polymers (hydrogels) or coated with functionalized polymer chains, preserving their photoluminescent properties [13, 14]. Two-stage photoluminescence has been reported in 3 nm and 6 nm CdTe-QDs; the small QDs are excited with ultraviolet light to produce photoluminescence that in turn excites the larger QDs, which present photoluminescence in the electromagnetic spectrum’s red region [13]. CdTe-QDs’ photoluminescence is exploited in biology and medicine as cellular and tissue biomarkers [15].

For these reasons, matrices such as polyvinyl alcohol [16], PMMA [17], polyfluorene [18], and PLA [19], among others [20], have been used as support matrices for CdSe, CdSe/ZnCdS, InP/ZnSeS, CdS, and CdSe-ZnS quantum dots. Some matrices have been used in the form of micro- and nanofibers obtained via different methods, among which electrospinning is the most common [21]. However, improving the interfacial adhesion between inorganic QDs and the polymer matrix remains a key issue in QD/polymer nanocomposites to address the surface modification of QDs. Among polymeric matrices, PLA is commonly used as a support for QDs due to its biodegradability, biocompatibility, and bioresorbability [22–24]. In this context, many studies have described using PLA micro- and nanofibers for different medical applications [25], but CdTe has seldom been reported due to its toxicity, which limits its biomedical applications.

In this study, CdTe-QDs were assembled on the surface of force-spun PLA microfibers. PLA microfibers were developed via the force-spinning technique (FS), which has proven to be a viable alternative to mass produce microfibers [26]. Although electrospinning is very popular, it has various disadvantages that have prevented its industrial application and their practical applications. Electrospinning produces minute amounts of fibers (~0.1 g h⁻¹), requires highly toxic organic solvents with specific dielectric properties, and has a high operational cost due to the high electric fields necessary [27]. FS was recently introduced to reduce the disadvantages of electrospinning. FS utilizes centrifugal forces instead of electric fields, and fibers can be produced using either a polymeric solution or molten polymer. When a polymeric solution is used to produce fibers, the solvent does not require specific dielectric properties since there is no electric field, which increases the number of materials that can be used to produce fine fibers. Its yield has been reported as high as 50–100 g h⁻¹ on laboratory scales and hundreds of meters per minute on industrial scales [28].

In this study, semiconductor QDs with low loading were sufficient to produce interesting electrical and optical properties that imparted green, yellow, orange, and red luminescence. The interactions of the quantum points’ stabilizing agent with the PLA matrix was also evaluated to anchor them to the matrix to increase their use in a wide range of applications such as biomedical applications, biosensing, biomarker platforms, and biomimetic smart nanocomposites. CdTe-QDs were produced using a one-pot microwave irradiation reduction method in an aqueous solution with 3-mercaptopropionic acid (MPA) as a capping agent. Using assisted impregnation, PLA composite fibers with CdTe quantum dots were obtained.

2. Materials and methods

2.1. Materials

The materials used to synthesize the CdTe-QDs were sodium tellurite (Na₂TeO₃, 99% Sigma-Aldrich), trisodium citrate dihydrate (Na₃C₆H₅O₇·2H₂O, Sigma-Aldrich), cadmium chloride (CdCl₂, anhydrous 99.0% Fluka), sodium borohydride (NaBH₄, ≥96.0% Aldrich), 3-mercaptopropionic acid (MPA, 99%), and sodium hydroxide (NaOH, Fermon). Poly-L-lactic acid (PLA-3251D) was purchased from Nature Works LLC (Blair, NE, USA), and chloroform was purchased from Sigma-Aldrich.

2.2. Microwave-assisted synthesis of CdTe-QDs

To synthesize aqueous MPA-coated CdTe-QDs, 40 ml of CdCl₂ (0.04 M) was diluted to 420 ml with water, and then 1.073 g of trisodium citrate dihydrate (Na₃C₆H₅O₇·2H₂O) was added to the solution. Subsequently, 40 ml of Na₂TeO₃ (0.01 M), 1.28 g of MPA, and 536.5 mg of NaBH₄ were added successively under magnetic stirring. The molar ratio of Cd²⁺/MPA/Te was 1:7:0.25.

The pH of the final solution (stock solution) was then adjusted to 7–8 with 1 M of NaOH. After 32 h, 20 ml samples were extracted from the precursor solution and heated in an Anton Paar-Monowave 300 microwave system. The reaction conditions were as follows: temperatures of 139, 145, 156, and 172 °C, 300 W of power, 10 min heating time, and a 600 rpm stirring rate. After microwave irradiation, the prepared solutions were cooled to 50 °C. The CdTe-QDs were then washed in 2-propanol and precipitated via centrifugation. CdTe-QD dispersion was prepared by redissolving this colloid precipitate in 20 ml of deionized water.
2.3. Preparing PLA microfibers by forcespinning
The solution used to fabricate the PLA microfibers (PLA-MFs) was prepared by dissolving 3.0 mg of PLA in 30 ml of chloroform at 40 °C under magnetic stirring for 6 h to obtain a 10%w/w polymeric matrix solution. This concentration was selected based on a previous optimization study [28]. A Cyclone L-1000 forcespinning system (FiberRio Technology Corp.) was used to produce the PLA-MFs from the prepared solutions. Figure 1(a) shows that approximately 2 ml of the prepared solution was injected into a cylindrical spinneret that contained two 30-gauge bevel needles (Becton-Dickinson) at each end. The forcespinning system was set at a velocity of 9000 rpm that allowed the polymer solution to be extruded through the needles’ orifices 45 s after turning on the spinneret. A deep-dish fiber collector with equally distanced aluminum-coated vertical steel pillars was used to collect the fibers. The PLA-MFs were detached from the collector (figure 1(b)) and dried under a vacuum for 24 h at 80 °C to remove the solvent traces.

2.4. Preparing PLA microfibers impregnated with CdTe quantum dots
The precipitation method used in this study involved assembling the CdTe-QDs in aqueous phases over the PLA-NFs’ surfaces. The pure PLA-MFs were cut into small wafers 1.6 cm in diameter and immersed in 2 ml of different CdTe-QDs solutions at room temperature. Corning Costar 24-well cell culture plates were utilized, and plastic rings were used to hold the fibers at the bottom of the wells. Precipitation occurred after 24 h. The mats of the PLA microfibers impregnated with the CdTe quantum dots (CdTe-QDs/PLA-MFs) were then dried at 90 °C overnight.

2.5. Characterizing the CdTe quantum dots and the PLA microfibers impregnated with CdTe quantum dots
The surface morphology of the CdTe-QDs was examined using JEOL JEM 2010 transmission electron microscopy (TEM) with an acceleration voltage of 200 kV. Samples were prepared by dropping the CdTe-QD suspension on the surface of a gold-coated copper grid and drying it in air. UV/Vis measurement was performed using a Thermo Evolution 600 spectrophotometer with a wavelength of 400 to 700 nm. The morphology and diameters of the CdTe-QDs/PLA-MFs were evaluated by scanning electron microscopy (SEM) (JSM-6390LV, JEOL, Tokyo, Japan). The samples were sputter coated with a thin gold layer before imaging. The average diameters of every fiber sample were calculated by randomly selecting 20 fibers using ImageJ software (National Institutes of Health).

Fluorescence spectral scans (FL) were obtained using a Cytation Hybrid Multi-Mode Reader from BioTek Instruments (Winookski, VT, USA). For emission scans, the fixed excitation wavelength was set to 366/50 nm and the emission wavelengths were scanned from 430/9 nm to 700 nm. A xenon flash lamp was used as the light source, with the lamp energy set to high. Ten measurements were obtained for each data point. The images of the microfibers’ fluorescence properties were acquired using an LSM 510 META confocal laser scanning microscope (Carl Zeiss, Oberkochen, Germany). The molecular interactions between the PLA-MFs and CdTe-QDs were probed via Fourier transform infrared spectroscopy (FTIR) using a PerkinElmer Frontier spectrometer in a spectral range of 400–4000 cm⁻¹.
3. Results and discussion

In this study, a series of highly fluorescent CdTe-QDs stabilized with 3-mercaptopropionic acid (MPA) in aqueous phases under microwave irradiation were prepared. Figure 2 shows typical fluorescence images of different sized MPA-coated CdTe-QDs illuminated with an ultraviolet (UV) lamp. Figure 3 shows the UV–vis absorption spectra of four samples (S1-S4) prepared at 300 W with 10 min of heating and different temperatures: S1 green (139 °C), S2 yellow (145 °C), S3 orange (156 °C), and S4 red (172 °C). The excitation absorption peaks of the QDs were obvious, with reflux temperatures from 139 to 172 °C. The maximum absorption peaks appeared at 479, 509, 518, and 580 nm in samples S1 to S4, respectively. Compared with CdTe bulk material (827 nm) [29], the excitation absorption peaks of the CdTe-QDs had significant red shifts as the temperature increased from 139 to 172 °C, demonstrating obvious quantum confinement effects. These were demonstrated by the considerable displacement of the optical absorption band from 479 nm (S1) to 580 nm (S4) in the particles studied. The shift in the absorption and emission bands’ peak positions indicated that the CdTe-QD nanocrystals had different sizes. The larger the CdTe-QD, the redder (lower energy) is the fluorescence spectrum. Conversely, smaller dots emitted green (higher energy) light. The absorption peaks broadened due to larger size distributions with different nanocrystal sizes in the solution at higher temperatures; this effect is called Ostwald ‘ripening’ [30, 31]. The nanoparticles’ energy levels were discrete due to the quantum confinement of the charge carriers in the crystal. The Brus equation [32] was used to describe the quantum confinement’s effects.
on the nanoparticles’ radii to obtain their relative crystal sizes. Samples S1–S4 were 2.32, 2.51, 3.34, and 4.24 nm, respectively (see the supplementary material (available online at stacks.iop.org/MRX/8/096203/mmedia)).

The morphological characteristics of the CdTe-QDs in sample S4 (172 °C, red) were observed through transmission electron microscopy (TEM) at different magnifications (figure 4). The QDs were uniform and monodispersed and had spherical shapes, good crystallinity, and an average size of 4 nm. The particle size found via TEM was quite close to the size calculated using the Brus equation. Similar results between the experimental sizes and those calculated using the same relationship were previously reported by Rodríguez et al [33].

Images of the CdTe-QDs/PLA-MFs impregnated with the QDs in sample S1 (obtained at 139 °C, green) at room light conditions and excited under UV illumination are displayed in figures 5(a) and (b). The microfibers emitted fluorescence with high brightness and homogeneity due to the QDs’ stable structures. The color could
be tuned from green to orange and red by controlling the CdTe-QDs’ particle sizes. Figure 5(c) shows the entire set of solutions collected at 139 to 172 °C in the solutions in which the microfibers were submerged.

The PLA-MFs obtained by forcespinning had diameters between 0.7 μm and 11.0 μm, with an average diameter of 3.5 μm (figure 6(a)). The microfibers were randomly oriented, forming a three-dimensional mesh and exhibiting a rough, flexible, cylindrical, long, and continuous morphology. Their flexible properties provide easy handling and comfortable textures when used in medical applications. The diameters of the PLA-MFs obtained by forcespinning had similar values to those reported by Yoon et al [34]. They used electrospinning to obtain PLA/PEG fibers with diameters between 0.90 and 8.9 μm. Yin et al [35] used electrospinning to produce

![Figure 6. SEM micrographs of the PLA-MFs (a) and CdTe-QDs/PLA-MFs (b).](image)

![Figure 7. Confocal fluorescence microscopy images of the different CdTe-QDs/PLA-MF samples: (a) S1, green; (b) S2, yellow; (c) S3, orange; and (c) S4, red. The excitation wavelength was 370 nm. Scale bar: 100 mm.](image)

![Figure 8. Normalized PL spectra of the CdTe-QDs/PLA-MFs in the presence of differently sized CdTe-QDs: (a) λ_em = 535 nm, green; (b) λ_em = 545 nm, yellow; (c) λ_em = 575 nm, orange; and (d) λ_em = 605, red.](image)
PLA fibers with microcrystalline cellulose (MCC) between 0.1 and 3 μm in diameter, while PLA/collagen fibers with average diameters of 9 μm were reported in another study [36]. Since forcespinning uses centrifugal force rather than electrostatic force as in electrospinning and utilizes either solutions or solid materials that are in the solution or melt spun into nano fibers [27], fibers similar to those obtained with electrospinning were fabricated in this study. The only difference was that the process was much faster and enabled higher fiber production. After the CdTe-QD impregnation process, the diameter and morphology of the PLA-MFs were not affected because the CdTe-QDs' sizes were much smaller than those of the fibers (figure 6(b)). The formation of coarse fibers was attributed to the acceleration and deceleration phases during the forcespinning cycle [37]. Prior studies reported incorporating ZnS-QDs, CdSe-QDs, and other quantum dots into PLA nanocomposites [16–20], but incorporating CdTe into PLA microfibers using forcespinning was not previously described.

The presence of the CdTe-QDs on the surface of the PLA-MFs was confirmed using confocal fluorescence microscopy as shown in figures 7(a)–(d). Uniform and strong fluorescence emission was observed throughout all of the samples, which indicated that the CdTe-QDs were uniformly distributed in the nanofibers. The resulting hybrid fibers exhibited green, yellow, orange, and red emission fluorescence under ultraviolet excitation at 370 nm. All of the microfibers were clearly disordered and more than several millimeters in length, in accordance with the results of the SEM images.

To study the microfibers’ fluorescence properties, the fluorescence spectra of the CdTe-QDs/PLA-MFs were conducted at an excitation wavelength of 370 nm (figure 8). All of the samples exhibited the characteristic PL spectra of CdTe-QDs, the symmetrical PL spectra, and narrow full width at half maximum (FWHM) of the PL spectra (42–52 nm). The microfibers displayed size-dependent fluorescence emissions at 535, 545, 575, and 605 nm, corresponding to green, yellow, orange, and red light emissions, respectively. The PL spectra of the CdTe-loaded microfibers shifted to longer wavelengths due to the quantum confinement effect, which had a similar tendency with the UV–vis spectra. Before normalizing the spectra, the PL intensities did not increase linearly in the four samples (S1–S4) indicating an approximately similar concentration in each sample. Sharp and symmetrical emission peaks demonstrated that the quantum dots were stable with good monodispersity in...
had a characteristic absorption band at 1759 cm\(^{-1}\) for the polymer matrix and had a narrow particle size distribution \cite{38}. These facts confirmed that little aggregation or degradation occurred during the CdTe-QDs/PLA-MF fabrication process, indicating that the QDs’ crystallinity was not affected by the PLA matrix.

The chemical characterization of the CdTe-QDs/PLA-MFs’ surfaces was ascertained via the FTIR analysis. Figure 8 shows the FTIR spectra recorded between 500 and 4000 cm\(^{-1}\) for the (a) pure MPA (mercaptopropionic acid), (b) MPA-capped CdTe-QDs obtained with sample S1 (139 °C, green), (c) CdTe-QDs/PLA-MFs, and (d) pure PLA. In the pure MPA (figure 9(a)), the most pronounced IR absorption bands occurred at 3,500 to 3,000 cm\(^{-1}\) (νOH), 2,950 cm\(^{-1}\) (νCH\(_3\)), 2,574 cm\(^{-1}\) (νSH), 1,707 cm\(^{-1}\) (νC=O), 1,222 cm\(^{-1}\) (νC–O), and 680 cm\(^{-1}\) (νC–S). The MPA-capped CdTe-QDs (figure 9(b)) had absorption bands at 3,410 cm\(^{-1}\) (νOH, COOH), 1,185 cm\(^{-1}\) (C–O–C) symmetric stretching of the ester group, 1,391 cm\(^{-1}\) (C=O=O) asymmetric stretching vibrations of the ester group, 1,370 cm\(^{-1}\) (C=O=O), and 1,350 cm\(^{-1}\) (C=O=O). The missing S-H (2550–2650 cm\(^{-1}\)) vibration peak in the MPA-capped CdTe-QDs may have been due to the covalent bonding between thiols and Cd atoms on the QDs’ surfaces. The appearance of the bands at around 1570 cm\(^{-1}\) and 1400 cm\(^{-1}\) indicated that the MPA had chemisorbed on the Cd ions of the CdTe-QDs’ surfaces. The FTIR spectrum of the pure PLA fibers (figure 9(d)) had a characteristic absorption band at 1759 cm\(^{-1}\). In the pure PLA’s spectrum, a broad and strong absorption band at 1759 cm\(^{-1}\) was attributed to the –C=O stretching of the ester group \cite{39}. The bands at 2995 and 2945 cm\(^{-1}\) were due to the asymmetric and symmetric (C–H) stretching vibrations of the –CH\(_3\) group, respectively. The 1184 and 1212 cm\(^{-1}\) doublet observed was attributed to the symmetric (C–O–C) stretching of the ester groups. The strong absorption peak at 1130 cm\(^{-1}\) was due to the (–CH\(_3\)) rocking mode \cite{40}. The peaks are summarized in Table 1. All of these absorption bands were typical peak characteristics of PLA. All of the aforementioned absorption bands were characteristics that also appeared in the FTIR spectrum of the CdTe-QDs/PLA-MFs (figure 9(c)). The bending vibration peak at 1566 cm\(^{-1}\) indicated the successful conjugation of the PLA to the surface of the CdTe-QDs.

### Table 1. FTIR spectral data of MPA, MPA-capped CdTe QDs, pure PLA, CdTe QDs/PLA nanofibers.

| Peak position (cm\(^{-1}\)) | Band assignment |
|-----------------------------|-----------------|
| MPA                         |                 |
| 3,500 to 3,000              | O–H stretching vibration |
| 2,950                       | S–H stretching modes |
| 2,574                       | S–H stretching modes |
| 1,707                       | C=O stretching of COOH |
| 1,222                       | C–O stretching vibration |
| 680                         | C–S stretching vibration |
| MPA-capped CdTe QDs         |                 |
| 3,410                       | O–H stretching vibration |
| 1,570                       | (COO–) asymmetric stretch vibrations |
| 1,400                       | (COO–) symmetric stretch vibrations |
| 1,290                       | C–O stretching vibration |
| Pure PLA                    |                 |
| 1,759                       | C=O stretching vibration of the ester group |
| 1,185                       | (C–O–C) symmetric stretching of the ester group |
| 1,130                       | A-CH\(_3\) rocking mode |
| 1,089                       | (C–O–C) asymmetric stretching vibration |
| CdTe QDs/PLA nanofibers     |                 |
| 1,566                       | (COO–) asymmetric stretch vibrations |

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### 4. Conclusions

In conclusion, MPA-capped CdTe-QDs were synthesized in an aqueous solution through a convenient one-pot strategy using microwave synthesis. Their diameters varied between 2 and 4 nm. Manufacturing the PLA microfibers via forcespinning rapidly produced a larger amount of fibers. The fibers obtained had a wide diameter distribution, with an average diameter of 3.5 μm. The CdTe-QDs were impregnated in the PLA fibers, where they were stabilized, and the fluorescence images indicated that these were homogeneously distributed in

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all of the fibers. The incorporation of the QDs into the PLA fibers did not modify their morphology or size. The luminescent nanofibers with green, yellow, orange, and red fluorescence could easily be tuned by controlling the size of the CdTe-QDs and also demonstrated strong fluorescence properties over several months at room temperature. The CdTe-QDs/PLA-MFs were extremely flexible and had a high surface area to volume ratio due to their fine fiber diameters. Forcespinning will lead to the creation of a new family of advanced quantum dot and polymer products that can be used for biological labeling, optical/biological sensors, multifunctional textiles, photonic devices, and optical materials.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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