Electrospinning fabrication of $\text{SrTiO}_3\text{:Er}^{3+}$ nanofibers and their applications in drug delivery system

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Abstract: $\text{SrTiO}_3\text{:Er}^{3+}$ nanofibers were fabricated by the electrospinning process. The phase, microstructure, Doxorubicin loading and release properties, as well as upconversion emission of the fabricated $\text{SrTiO}_3\text{:Er}^{3+}$ nanofibers were investigated. The X-ray diffraction patterns indicate that the $\text{SrTiO}_3\text{:Er}^{3+}$ nanofibers have the pure cubic phase. The images of scanning electron microscopy and transmission electron microscopy demonstrate that nanofibers with a porous structure have been obtained. The $\text{SrTiO}_3\text{:Er}^{3+}$ nanofibers can be used in drug delivery system, which is confirmed by the Fourier transform infrared spectra, results of Barrett–Emmett–Teller measurements and thermogravimetry. Under 808 nm light irradiation, the release process of Doxorubicin in $\text{SrTiO}_3\text{:Er}^{3+}$ nanofiber shows close relation with the pH value. An acidic condition increases the solubility of the Doxorubicin for the protonated daunosamine group, which accelerates and improves the release of Doxorubicin in $\text{SrTiO}_3\text{:Er}^{3+}$ nanofibers. In addition, the degree of Doxorubicin release can be manifested by the changing intensity of upconversion emission.

Keywords: $\text{SrTiO}_3\text{:Er}^{3+}$ nanofibers; Drug delivery; Upconversion.

1. Introduction

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Lanthanide (Ln^{3+}) based upconversion nanomaterials have been paid extensive attention in past decade years due to their potential applications in fields of solar cells [1], biomedical imaging [2], optical temperature sensing [3], phosphors [4] and optical pH sensors [5]. Upconversion is a nonlinear antistokes process, which converts two or more photons with lower energy into an outcome photon with higher energy [6]. Recently, upconversion nanomaterials have been focused on the biomedical applications with the significant advances in the controllable fabrication, functional designing and optical performance [7]. Among the Ln^{3+} ions, erbium (Er^{3+}) possesses relatively high energy gap, which results in low probabilities of non-radiative transitions among different excited levels of the ion. Therefore, due to its merits of expedient structure of energy level and remarkable upconversion performance, Er^{3+} is widely used as an upconversion activator.

Perovskites with a general formula of ABO_3 , such as SrTiO_3 [8, 9], BiFeO_3 [10, 11] and $\text{KTa}_{(1-x)}\text{Nb}_x\text{O}_3$ [12-14], have wide applications in various fields. Among them, SrTiO_3 is kept a watchful eye because of its wide application as a standard substrate in the epitaxial growth of functional films, its use as a photocatalyst in the fields of photochemical water splitting and photocatalysis degradation of organic pollutants, as well as its biomedical applications [8, 9, 15-18]. Like Ca, Sr is an element that widely used in clinical treatments of osteoporosis [19]. In human body, the content of Sr in bone tissue is about 98% of the total amount in body. A lot of Sr-contained materials, such as Sr-Ti microarc oxidation [19], Sr doped hydroxyapatite [20], SrTiO_3 - TiO_2 [21] and Mg-Zn-Sr alloy [22], have shown wide applications in the biomedical fields.

In medical treatment, nanocarriers have been widely used in drug delivery in order to improve drug efficacy. Specifically, a stimuli-responsive drug delivery system using “smart” nanocarriers has become a promising modality for improving the drug efficacy due to its controlling drug release ability. In the control of drug release, near infrared light is one of widely used approaches because of its merits of ignorable injuries to body tissue and high-spatial resolution [23]. Nanofibers have turn into one of choices in the field of drug delivery system due to the hierarchically porous nanostructure [24]. Herein, we report on the electrospinning fabrication of $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers. Doxorubicin (DOX) has been loaded into the $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers as a model drug. The DOX releases in solutions with different pH values under irradiation of 808 nm have been measured.

2. Materials and methods

$\text{Ti}(\text{OC}_4\text{H}_9)_4$ (titanium butoxide, 99%), $\text{Sr}(\text{CH}_3\text{COO})_2$ (strontium acetate, 99%) and Er_2O_3 (erbium oxide, 99.99%) were used as raw materials. The mixture of CH_3OH (methanol, 99.8%) and CH_3COOH (acetic acid, 1.0M) was used as the solvent. The volume ratio of CH_3OH and CH_3COOH was 4:1. $(\text{C}_6\text{H}_9\text{NO})_n$ (Polyvinylpyrrolidone, PVP, $M_w=1300000$) was used as the addition. $\text{NH}_3\cdot\text{H}_2\text{O}$ (ammonia solution, 0.1M) was used to adjust the pH value of solution. All of chemicals were used directly without further purification. To obtain $\text{Er}(\text{NO}_3)_3$, appropriate Er_2O_3 was dissolved into HNO_3 .

In the fabrication of SrTiO_3 nanofibers, 5 mmol of $\text{Ti}(\text{OC}_4\text{H}_9)_4$, 4.95 mmol of $\text{Sr}(\text{CH}_3\text{COO})_2$, 0.05mol of $\text{Er}(\text{NO}_3)_3$ and 1 g of $(\text{C}_6\text{H}_9\text{NO})_n$ were dissolved into 15 mL

of solvent under continuous stirring at room temperature. Herein, the precursor solution was obtained. Then, the precursor solution was loaded into a plastic syringe with a stainless steel needle. A piece of aluminum foil was vertically placed 15 cm away from the needle to collect the fabricated nanofibers. The supplied voltage was 20 kV. The speed of the spinning was about 2 mL/h. The precursor nanofibers were heat treated at 600 °C for 6 h in a muffle furnace. The heating rate was controlled to be 5 °C/min and the cooling rate was controlled to be 3 °C/min.

The drug storage/release properties of $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers were evaluated by adding 20 mg of $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers to 5 mL of phosphate buffer solution (PBS, pH= 4.7, 5.8 and 7.4) of DOX with a concentration of 1.5 mg/mL at 37 °C. The mixture was gently shaken for 24 h at 37 °C in dark conditions to reach the equilibrium. The $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofibers were collected by centrifugation and washed with cyclohexane for several times. The in vitro delivery test was performed by immersing the $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofibers in 10 mL of PBS under gentle stirring in dark conditions, while the immersing temperature was kept at 37 °C. At selected time intervals, buffer solution was taken and replaced with fresh buffer solution.

The phase of the sample was confirmed by a D8 advance X-ray diffractometer (Bruker, German) using Cu K α radiation ($\lambda = 0.15418$ nm). The scanning electron microscopy (SEM) measurements were carried out by an FESEM-4800 field emission scanning electron microscope. The transmission electron microscopy (TEM) measurements were carried out by a Hitachi S-4800 instrument. The Fourier transform infrared (FTIR) spectra were measured by a Perkin-Elmer 580B infrared

spectrophotometer with the KBr pellet technique. The specific surface areas were determined by the Barrett–Emmett–Teller (BET) technique on a Micromeritics ASAP 2000 surface area and porosity analyzer. Thermogravimetry (TG) was implemented on Labsys evo STA Simultaneous thermal analyzer (France) in air with a heating rate of 10 °C/min. The emission spectra were acquired through a HitachiF-7000 spectrophotometer equipped with a 150W xenon lamp as the excitation source.

3. Results and discussion

Fig.1 presents the X-ray diffraction (XRD) patterns of the fabricated $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers and the standard data of JCPDS no. 35-0734. The diffraction peaks corresponding to (100), (110), (111), (200), (211) and (220) planes can be seen for the fabricated $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers, which are well according with the JCPDs no. 35-0734. This demonstrates that the $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers have a cubic crystal phase. The diffraction peaks of Er_2O_3 or $\text{Er}(\text{NO}_3)_3$ cannot be observed in the XRD pattern, which is due to the low concentration of Er^{3+} incorporated into the SrTiO_3 lattice. The average crystallite size can be calculated by the Scherrer's formula, $0.89\lambda/\beta \cos \theta$, where D is the average crystallite size, λ is the wavelength of X-ray, θ and β are the diffraction angle and full-width at half-maximum (in radians) of an observed peak [25]. The calculated crystallite size of SrTiO_3 nanofiber is 26.7 nm by using the full-width at half-maximum of (110) diffraction peak.

Fig.2 gives the SEM and TEM images of $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers. As shown in the SEM image (Fig.2A), a large number of $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers have been fabricated in current synthesis. The nanofibers have a diameter region of 125-170 nm

and a length region of several micrometers. The TEM image (Fig.2B) with a higher magnification shows that the nanofibers have a rough surface and lots of hollow caves. The formation of caves in these nanofibers is induced by the decomposition of $(C_6H_9NO)_n$ and the densification during $SrTiO_3:Er^{3+}$ crystallization. The porous structure of nanofibers shows functional applications in some fields, such as functioning as anchoring points for cells in tissue engineering, increasing the specific surface area in percolation and photocatalysis, modifying the wetting performance and altering the kinetics of drug release [26]. In drug delivery system, the main influence factor is the controlling drug traveling length or modulating the affinity between matrix materials and nanofiber with porous structure is one of remarkable candidates for drug delivery because that nanofiber provides a short diffusion passage length and an excellent mass transfer and drug release [27].

The vibrational spectrum is an effective way to verify the composition of the materials. Fig.3 shows the FTIR spectra of $SrTiO_3:Er^{3+}$ nanofibers, DOX and $SrTiO_3:Er^{3+}$ -DOX nanofibers. In the FTIR spectrum of $SrTiO_3:Er^{3+}$ nanofibers, the absorption band in the low frequency region with a peak at about 576 cm^{-1} corresponds to the Ti-O bond within the TiO_6 octahedron cluster [28]. The absorption bands with peaks at about 1632 cm^{-1} and 3428 cm^{-1} originate from the stretching and bending modes of the absorbed water [29, 30]. The absorption band peaking at about 1420 cm^{-1} is induced by the residual nitrate [29]. There are multiple peaks in the FTIR spectrum of DOX besides the stretching and bending modes of the absorbed H_2O . The absorption band at 2920 cm^{-1} corresponds to the asymmetrical stretching vibration of

C-H bands [31]. The absorption bands peaking at 796 cm^{-1} is due to the N-H deformation bonds. The absorption bands peaking at 992 cm^{-1} , 1088 cm^{-1} , 1268 cm^{-1} and 1708 cm^{-1} originate from the different quinine and ketone carbonyls of DOX and they are hard to trace due to the existence of carbonyl groups in them [32]. In the FTIR spectrum of $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofibers, there are absorption bands of SrTiO_3 and DOX, which suggests the successful loading of DOX to the SrTiO_3 nanofibers.

The successful loading of DOX to the SrTiO_3 nanofibers also can be confirmed by the BET and TG measurements. Fig.4 shows the nitrogen adsorption-desorption isotherms of $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers before and after the DOX loading. The representative iv-type isotherms suggest that the $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers before and after the DOX loading have the porous structure. Due to the loading of DOX, the value of specific surface area decreases from $22.6\text{ m}^2/\text{g}$ to $3.8\text{ m}^2/\text{g}$. Fig.5 shows the results of TG analysis. The weight loss of $\text{SrTiO}_3:\text{Er}^{3+}$ nanofiber is about 3.5wt%, which is induced by the evaporation of absorbed water. However, the weight loss of $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofiber is about 21.5wt%. A comparison of weight loss of $\text{SrTiO}_3:\text{Er}^{3+}$ and $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofibers indicates that about 18wt% DOX has been loaded to the $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers.

DOX is one of widely used antineoplastic agents in treatment of tumors. As shown in the FTIR spectra, BET and TG results, DOX has been successfully loaded to the $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers. The release properties of $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers to DOX have been examined under the irradiation of 808 nm light. 808 nm light has been regarded as a more appropriate light for medical purpose than 980 nm light because of

the less than 5% water absorption in comparison with 980 nm, which induces the super penetrability and low thermal injury to cells and tissues [18]. The PBS solutions with pH values of 4.7, 5.8 and 7.4 are requisitioned. As shown in Fig.6, the release percentages of DOX are about 35.5%, 68.9% and 87.8% in the initial 4 h when the pH values of PBS solutions are 7.4, 5.8 and 4.7 respectively. After the drug release for 12 h, the release percentages of DOX are about 39.3%, 74.5% and 91.1% when the pH values of PBS solutions are 7.4, 5.8 and 4.7 respectively. The results indicate that the decreasing pH value accelerates the DOX release, which is induced by the increase of solubility of the DOX for the protonated daunosamine group under acidic conditions [23].

The degree of DOX release can be manifested by the changing intensity of upconversion emission. The upconversion emissions of $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofibers have been characterized at different stages of DOX release. Fig.7 shows the upconversion emission spectra of $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofibers under the excitation of 980 nm laser and pump power of 320 mW at different release times. Three emission bands in the range of 500-700 nm can be seen in the emission spectra. The two strong emission bands in the range of 500-600 nm correspond to the $^2\text{H}_{11/2} \rightarrow ^4\text{I}_{15/2}$ and $^4\text{S}_{3/2} \rightarrow ^4\text{I}_{15/2}$ transitions of Er^{3+} and the emission band in the range of 650-700 nm results from the $^4\text{F}_{7/2} \rightarrow ^4\text{I}_{15/2}$ transition of Er^{3+} ion [3]. The intensities of emission bands in the range of 500-600 nm increase with the progress of drug release, but the intensity of emission band in the range of 650-700 nm is near to be a constant. The relative intensities of the emission bands of $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofibers at different release

times are shown in Fig.8. The figure clearly shows the intensity-dependence of emission bands in the range of 500-600 nm on release time and intensity-independence of emission bands in the range of 650-700 nm. It is known that DOX has the strong absorbance in the wavelength range of 400-580 nm [32], which overlaps with the upconversion emission corresponding to the $^2H_{11/2} \rightarrow ^4I_{15/2}$ and $^4S_{3/2} \rightarrow ^4I_{15/2}$ transitions of Er^{3+} . The emission quenching induced by DOX loading is weakened because of the release of DOX in $SrTiO_3:Er^{3+}$ -DOX nanofiber, which induces the increases of emission intensity in the drug release process.

4. Conclusion

We fabricated $SrTiO_3:Er^{3+}$ nanofibers by the electrospinning process. The XRD patterns indicate that the $SrTiO_3:Er^{3+}$ nanofibers have the pure cubic phase. The images of SEM and TEM demonstrate that the nanofibers have the porous structure. $SrTiO_3:Er^{3+}$ nanofibers show excellent drug loading and release properties, which is confirmed by the FTIR spectra, results of BET and TG. Under 808 nm light irradiation, the release process of DOX in $SrTiO_3:Er^{3+}$ nanofiber shows close relation with the pH value. An acidic condition increases the solubility of the DOX for the protonated daunosamine group, which accelerates and improves the release of DOX in $SrTiO_3:Er^{3+}$ nanofibers. Under the 980 nm excitation, $SrTiO_3:Er^{3+}$ nanofibers show upconversion emission bands corresponding to the $^2H_{11/2} \rightarrow ^4I_{15/2}$, $^4S_{3/2} \rightarrow ^4I_{15/2}$ and $^4F_{7/2} \rightarrow ^4I_{15/2}$ transitions of Er^{3+} ion. In addition, the degree of DOX release can be manifested by the changing intensity of upconversion emission because of the quenching effect of DOX to emissions originating from the $^2H_{11/2} \rightarrow ^4I_{15/2}$ and $^4S_{3/2} \rightarrow$

$^4I_{15/2}$ transitions.

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Figure captions

Figure 1 XRD patterns of $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers and standard data of JCPDs no. 35-0734

Figure 2 SEM and TEM images of $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers

Figure 3 FTIR spectra of $\text{SrTiO}_3:\text{Er}^{3+}$ nanofibers, DOX and $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofibers

Figure 4 N_2 adsorption/desorption isotherms of $\text{SrTiO}_3:\text{Er}^{3+}$ and $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofibers

Figure 5 TG curves of $\text{SrTiO}_3:\text{Er}^{3+}$ and $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofibers

Figure 6 Cumulative DOX release from $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofibers in PBS solutions with pH values of 4.7, 5.8 and 7.4

Figure 7 Upconversion emission spectra of $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofibers in PBS solutions with a pH value of 4.7 at different release stages

Figure 8 Relative intensities of upconversion emission spectra for $\text{SrTiO}_3:\text{Er}^{3+}$ -DOX nanofibers in PBS solutions with a pH value of 4.7 at different release stages

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8