Controllable preparation of trifold nanorods and nanospheres of cadmium telluride via solvothermal process

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Abstract
In this study, trifold CdTe nanorods and CdTe nanospheres were prepared by the environmentally benign solvothermal process where K₂TeO₃ and CdCl₂ acting as the raw materials, L-ascorbic acid as the reducing agent, ethylene glycol as the solvent and polyvinylpyrrolidone as the surfactant, respectively. The morphology of the products can be regulated by the amount of KOH. Specifically, trifold CdTe nanorods could be prepared with 0.1 g of KOH before the reaction; while CdTe nanospheres were obtained with 0.5 g of KOH. The higher the amount of KOH added, the stronger the reducibility of L-ascorbic acid. For higher reducibility, CdTe crystal nuclei which was continuously generated will first nucleate and grow in the gaps between the rods of the trifold nanorods because of the special morphology and structure of the trifold nanorods. Therefore, with the increasing of the number of CdTe crystal nuclei caused by the accelerated reaction rate, the trifold nanorods will gradually evolve into nanospheres.

1. Introduction
Nanomaterials usually refer to the materials with at least one-dimension between 1 nm and 100 nm [1]. In recent years, semiconductor nanomaterials are widely preferred for their applications in nanodevices [2, 3]. For example, they are widely employed in solar cells [4], photodetectors [5], field effect transistors [5, 6], biosensors [7–11] and other fields, and also have the high energy conversion efficiency [12], which have aroused widespread concern in today’s society [13]. As one of the most widely used semiconductors, cadmium telluride (CdTe) has a direct energy band gap of 1.45 eV under room temperature [14] and exhibit excellent semiconductor properties such as ideal band gap, high light absorption coefficient, good stability, and high carrier mobility [15]. The optical absorption coefficient of CdTe is also higher so as to reduce the quantity of semiconductors needed [16]. Therefore, it has a broad application value in the field of optoelectronics [17].

Recently, CdTe nanomaterials with a variety of morphologies have been explored, such as nanorods [18], nanowires [19], nanotubes [20] and so on. As new semiconductor materials, these CdTe nanomaterials show excellent performance in energy and information fields. However, the successful exploration of a reliable and simple preparation method is the premise for the further application of CdTe nanomaterials. Over the past decade, chemical vapor deposition [21], electrochemical route [22], self-assembly technology of colloidal nanocrystals [13, 23], hydrothermal [24] and solvothermal [25] are commonly employed to prepare these CdTe nanomaterials. For example, X Zhang et al [21] fabricated Zn-doped CdTe nanowires with an average diameter of about 40 nm and lengths up to several tens of micrometers through chemical vapor deposition under 850 °C D Choi et al [16] prepared CdTe nanowires with the diameter of about 50 nm on the Parylene substrates in virtue of an anodic aluminum oxide (AAO) template. Dhanuskodi et al [18] prepared CdTe nanorods with the length of 176 nm and diameter of 43 nm by hydrothermal process using ascorbic acid as a reducing agent. Tang et al [25] synthesized the zinc blende CdTe nanowires via solvothermal process with the aid of polyvinyl alcohol and ethylenediamine. Of various known methods, solvothermal method is a synthetic method in which the original mixture react in a closed system under optimized conditions of temperature and pressure [26]. This method can
be used to synthesize some oriented nanomaterials and regulate the morphology of the products by adjusting the reaction conditions [25, 27].

In this paper, trifold CdTe nanorods (NRs) and CdTe nanospheres (NSs) with different morphologies were prepared by the environmentally benign solvothermal process with CdCl₂ and K₂TeO₃ as the raw materials, L-ascorbic acid as the reducing agent, ethylene glycol as the solvent and PVP as the surfactant. A trifold CdTe NR is made up of three needle-like components which bonded together at the middle part. All the trifold CdTe NRs and CdTe NSs obtained possess face-centered cubic phase and defect-free. The prepared trifold CdTe NRs have an outer diameter of about 400 ∼ 500 nm and a length of about 3 ∼ 4 μm. Each trifold NRs is made up of three needle-like components which bonded together at the middle part. The angle of the link is 120°. The size of the CdTe NSs is relatively smaller with a diameter of about 150 nm. The surface of NSs is extremely rough. In order to obtain the morphology evolution from trifold NRs to NSs, the effect of KOH contents on the morphology of trifold CdTe NRs and CdTe NSs was also analyzed. It shows that with the increasing of the amount of KOH, the reducibility of L-ascorbic acid will be improved, which will make the reaction more completely.

2. Experimental procedure

2.1. Reagents
In the process of experiment, potassium tellurite (≥98%, K₂TeO₃, M_W: 253.79), L-ascorbic acid (≥99%, C₆H₈O₆, M_W: 176.12), polyvinylpyrrolidone (PVP K-30), cadmium chloride (≥99%, CdCl₂·10.5H₂O, M_W: 228.35), potassium hydroxide (≥85%, KOH, M_W: 56.11), ethylene glycol (≥99%, (CH₂OH)₂, M_W: 62.07) and anhydrous ethanol were used. The raw materials used in the experiment were purchased from Sinopharm Chemical Reagent Co., Ltd, and no purification treatment was done before using them. The water used in the experiment was primary distilled water.

2.2. Synthesis
In a typical synthesis procedure of CdTe nanomaterials, 0.1523 g of K₂TeO₃ (0.6 mmol), 0.4 g of PVP, and 30 ml of ethylene glycol were put into a beaker under vigorous magnetic stirring at 80 °C to prepare homogeneous stock tellurium solution. Then 2.0 ml of CdCl₂ aqueous solution (300 mM) and 5.0 ml of L-ascorbic acid solution (1.2 M) was added to the stock tellurium solution as prepared above, and the mixture was continuously stirred for 2 min. It should be noted that the atomic ratio of Te and Cd added before the reaction is 1:1. Finally, the mixture was transferred to a stainless-steel autoclave (KH-50ML) with the volume of 50 ml. The inner lining material of the autoclave was polytetrafluoroethylene (PTFE), which can withstand high temperature of about 200 °C. These samples were fully reacted in an electric blast drying oven (BGZ-76) for 36 h. In this study, KOH was employed to adjust the reducibility of L-ascorbic acid. The specific experimental conditions are shown in table 1.

After reaction, the mixtures of the products were cooled to room temperature naturally and took liquid-solid separation through centrifugation. The precipitate was rinsed with the anhydrous ethanol and distilled water and centrifuged for 3 ~ 4 times. Finally, the products were dispersed into 30 ml absolute ethanol and stored away from light.

2.3. Characterization
The phase composition of the products was performed by the D/max 2500 pc type x-ray diffraction (XRD, Rigaku), with a scanning rate of 8° min⁻¹ in the 2θ range from 20° to 80° under the acceleration voltage of 40 kV. The morphology of products was observed by using the JSM-7900F type scanning electron microscope (SEM, JEOL), which the acceleration voltage was set at 15 kV. In the SEM test, the samples were prepared by dropping CdTe ethanol dispersion on the silicon substrate. And the structural characteristics of the products was analyzed...
by the JEM-2100F type transmission electron microscope (TEM, JEOL). At the same time, the speciation analysis of surface elements of the prepared products was analyzed by the equipment of Nicolet 6700 type Fourier infrared spectrometer (FT-IR, Thermo Fisher Scientific) and Thermo Scientific ESCALAB 250Xi type x-ray photoelectron spectrometer (XPS, Thermo Fisher Scientific) between the wave number of 500 and 3900 cm$^{-1}$.

3. Results and discussion

3.1. XRD phase composition

To illustrate the phase composition of prepared trifold CdTe NRs, X-ray diffraction data set was collected. Figure 1 shows the XRD pattern of the trifold CdTe NRs synthesized with 0.1 g of KOH. All the diffraction peaks can be indexed to the face-centered cubic phase of CdTe, corresponding to the standard card number (JCPDS: 03-065-0880, F-43m (216)). There are no impurity peaks in the pattern beside CdTe, indicating pure CdTe were prepared. The lattice constant of the cell is 0.6483 nm, which are also in agreement with the standard literature [28]. The diffraction peaks at 23.753°, 27.494°, 39.275° and 46.417° correspond to the diffraction planes (111), (200), (220) and (311), respectively.

3.2. SEM morphology analysis

The SEM images of the trifold CdTe NRs and CdTe NSs synthesized in this study are shown in figure 2. In the case of trifold CdTe NRs, 0.1 g of KOH was employed (figure 2(a)). It can be seen the prepared products have good dispersion ability and relatively uniform distributions of length and diameter. The prepared trifold CdTe NRs have an outer diameter of about 400 ∼ 500 nm and a length of about 3 ∼ 4 μm. Each trifold NRs is made up of three needle-like components which bonded together at the middle part. The angle of the link is 120°. However, the three NRs are separated from each other at the ends. The products with this morphology are very similar to the trifold Te NRs we prepared before [29].

To illustrate the element composition of the trifold CdTe NRs, the Energy-dispersive spectroscopy (EDS) analysis is also performed, as shown in figure 3. According to the mapping results of the products shown in figures 3(b)–(e), the products are composed of Cd and Te. The atomic ratio of Cd and Te is approximately 1:1. Combined with EDS analysis and XRD pattern of the trifold CdTe NRs, it is further proved that the products are composed of pure CdTe. It should be noted that the element Si mainly comes from the silicon substrate loaded with the products.

3.3. TEM analysis of trifold CdTe NRs

The TEM images of trifold CdTe NRs synthesized with 0.1 g of KOH are performed in figure 4. It can be seen the size of products is uniform. The prepared products have an inner diameter of 400 ∼ 500 nm with the length of ∼4 μm. The trifold CdTe NR is made up of three needle-like components which bonded together at the middle part. The average diameter of each component nanorod is about 60 nm. The fragment marked by red arrow in figure 4(a) shows a fragment of an individual trifold CdTe NR. The formation of the fragments can be attributed to the high-speed centrifugal separation carried out after the reaction, and the large centrifugal force promoted...
Figure 2. SEM images of the products synthesized with 0.1 g (a) and 0.5 g (b) of KOH respectively.

Figure 3. EDS spectrum of trifold CdTe NRs synthesized with 0.1 g KOH: (a) the original image of EDS analysis; (b) the spectrum of EDS; (c)–(e) EDS mapping results of the products shown in (a).
the violent collision between the trifold CdTe NRs, resulting in the fracture of a small number of trifold NRs from the middle part.

The images of selected-area electron diffraction (SAED) and high-resolution transmission electron microscopy (HRTEM) of trifold CdTe NRs synthesized with 0.1 g of KOH are shown in figure 5. It is found that the outer region of the trifold NRs is coated with some amorphous sheaths, as shown in figure 5(a).

Figure 5(b) shows the HRTEM image taken from the area marked with a rectangular box in figure 5(a). It can be seen that the as-prepared NRs are highly crystallized and defect-free [29]. The lattice spacing of the crystal is ∼0.374 nm, corresponding to the crystal plane of (111) in the face-centered cubic CdTe. The inset in figure 5(b) shows the selected-area electron diffraction (SAED) pattern of a single trifold CdTe NR. The diffraction rings show that the trifold CdTe NRs are polycrystalline.

3.4. X-ray photoelectron spectrometer (XPS analysis)
The XPS spectrum is usually used to analysis the chemical states of the products. The extended scan of Cd 3d region is shown in figure 6(a), and the extended scan of Te 3d region is shown in figure 6(b). It can be seen from figure 6 that Cd and Te were existed in the products, and the chemical composition of the products was also determined. The results are consistent with the XPS analysis of CdTe nanowires by Nicholas A Kotov et al [30].

The two peaks at 411.0 eV and 404.3 eV in figure 6(a) correspond to Cd 3d_{5/2} and Cd 3d_{3/2} [30]. The two peaks at 582.6 eV and 572.2 eV in figure 6(b) correspond to Te 3d_{5/2} and Te 3d_{3/2} respectively, which also confirm the existence of Cd-Te bonding [31, 32]. It can be seen from the quantization results in figures 6(a) and (b) that the atomic ratio of Cd and Te is approximately 1:1, which is consistent with the stoichiometric ratio of CdTe [33]. It can be seen from figure 6(b) that the peaks at 572.2 eV and 582.6 eV were all derived from Te(0) 3d,
and there was no Te(IV) 3d characteristic peaks, which indicated that there was no obvious oxidation during the process of storing and testing.

3.5. FT-IR analysis of trifold CdTe NRs
The FT-IR spectroscopy of prepared products with different amount of KOH are shown in figure 7. The absorption peak at 1422 cm\(^{-1}\) was the characteristic absorption peak of pyrrolidinyl group, and the absorption peaks at 1677 cm\(^{-1}\) and 1017 cm\(^{-1}\) were mainly attributed to the vibration of C=O double bond and C–N bond in PVP. The absorption peaks at 2953 cm\(^{-1}\) and 1422 cm\(^{-1}\) were mainly attributed to the stretching vibration of C–H bond, and the absorption peak at 1286 cm\(^{-1}\) was arose by the vibration of C–O single bond respectively\[34–36\]. The FT-IR spectroscopy of the prepared products and pure PVP sample are shown in figures 7(a) and (b), respectively. It can be seen from figure 7(a) that the characteristic absorption peaks of PVP on the products were obviously weakened, but there were still some typical peaks, indicating that there were some PVP molecules adsorbed on the surface of the products. This results also indicate that the PVP molecules on the surface of the products cannot be washed away thoroughly with the absolute ethanol and distilled water\[34\]. Furthermore, it can be seen from figure 7 that the absorption peaks at 3522 cm\(^{-1}\), 2953 cm\(^{-1}\), and 1617 cm\(^{-1}\) of the pure PVP sample were red-shifted to 3431 cm\(^{-1}\), 2924 cm\(^{-1}\), 1610 cm\(^{-1}\) respectively. The decreasing in wavenumber for the C=O double bond absorption of the products might result from the bond weakening via partial donation of oxygen to the cadmium\[34\].

3.6. Effect of the amount of KOH
According to our study, alkalinity of solution is a key factor affecting the morphology of the product. The alkalinity of the solution can be easily adjusted by adjusting the amount of KOH added. In the study of the effect of KOH, we fixed other reaction conditions to be the same as those detailed in the Experimental section: 2.0 ml
Figure 6. X-ray photoelectron spectroscopy of trifold CdTe NRs, and the extended scan in Cd 3d (a) and Te 3d region (b).

Figure 7. FT-IR spectrometer of the prepared products (a) and pure PVP sample (b).
of CdCl₂ aqueous solution and 5.0 ml of L-ascorbic acid solution at 150 °C for 36 h. The synthesized trifold CdTe NRs and CdTe NSs are shown in figure 8. When KOH is not added, the product is the trifold CdTe NRs with rough surface and disordered size (figure 8(a)). However, when 0.1 g KOH is added, the trifold CdTe NRs has relatively smooth surface and uniform size (figure 8(b)). Further increasing the amount of KOH, such as 0.5 g (figure 8(c)) and 3.36 g (figure 8(d)), only spherical substances with rough surface can be obtained. The size of CdTe NSs has become relatively smaller with a diameter of about 150 nm (figure 8(c)) and 500 nm (figure 8(d)). The morphology evolution of the obtained products synthesized with different amount of KOH will be briefly analyzed in the following.

In the solvothermal process with alkaline condition, there may be three reduction reactions under the action of reducing agent:

1. \( \text{Cd}^{2+} + 2e^- \rightarrow \text{Cd} \quad E^0_1 = -0.403 \text{ V} \)  
2. \( \text{TeO}_3^{2-} + 3\text{H}_2\text{O} + 4e^- \rightarrow \text{Te} + 6\text{OH}^- \quad E^0_2 = -0.57 \text{ V} \)  
3. \( \text{Te} + 2e^- \rightarrow \text{Te}^{2-} \quad E^0_3 = -1.143 \text{ V} \)

It can be seen the reduction potential has the following sequence: \( E^0_1 > E^0_2 > E^0_3 \). As we all know, the higher the reduction potential, the easier the reduction reaction will take place under standard conditions. As a result, the Cd(II) in the solution will be reduced to Cd(0) according to reaction (1) firstly. Then, the Te(IV) in the solution will be reduced to Te(0) via reaction (2). Finally, the Te(0) may be reduced to Te(II-) through reaction (3). If all three reactions are completed under the current reduction conditions, the products should be pure Cd or Cd-rich CdTe. But in this study, according to EDS analysis (figure 3), the molar ratio of Cd and Te in the product is 1:1. Therefore, it can be inferred from the reduction potential that the reaction (1) and (2) were complete, the reaction (3) did not occur. Hence, the Te(0) and Cd(0) that reduced by L-ascorbic acid will form the final products CdTe through the Te-Cd alloy reaction rather than the combination reaction between Cd²⁺ and Te²⁻ ions, as shown in reaction equation (4). It is similar to the reaction of Ag and Te described in [37].

\[ \text{Te}(0) + \text{Cd}(0) \rightarrow \text{CdTe} \quad (4) \]

As we all know, L-ascorbic acid will lose H⁺ during the solvothermal process, resulting in the generation of dehydroascorbic acid, as shown in reaction equation (5).

\[ \text{C}_6\text{H}_8\text{O}_6 - 4e^- \rightarrow \text{C}_6\text{H}_6\text{O}_6 + 4\text{H}^+ \quad (5) \]

During the solvothermal process, KOH was added and dissolved to the mixed solution before adding L-ascorbic acid. In this case, the reducibility of L-ascorbic acid was significantly enhanced due to the high basicity of the solution at the initial stage of the reaction [29]. A large number of Te(0) and Cd(0) were formed in the solution at the initial stage of the reaction and lots of CdTe crystal nuclei were obtained through reaction (4). Then, these CdTe nuclei grew up competitively and continuously. Furthermore, as the reaction proceeding, the reduction rate decreased because the alkalinity of the solution as well as the concentration of L-ascorbic acid decreased. Consequently, there are few Te(0) and Cd(0) formed in the later stage of reaction in the solution,
resulting in the products with smaller size [29, 38]. Therefore, the addition of more KOH will promote the formation of large number of nuclei in the solution at the initial stage of the reaction, and the aggregation and growth of these nuclei will make the product spherical structure. This is consistent with the experimental results presented in figure 8.

When the amount of KOH is low, the reduction ability of L-ascorbic acid is mild, and the nucleation will not be generated in the reaction process, so the growth rate of the product is slow, and finally the trifold NRs is obtained. However, at a higher amount of KOH, the nucleation in the solution at the initial stage of the reaction is explosive. During the growth process of trifold CdTe NRs at this time, the newly formed nuclei were preferentially adsorbed in the depressions at both ends of the trifold NRs and completed the crystallization process. In this growth mode, the energy change of the system was the smallest. The trifold NRs structure will gradually evolve into NSs structure as a result. The reaction mechanism is shown as figure 9.

Figure 9 shows the reaction mechanism diagrams of the trifold CdTe NRs and CdTe NSs synthesized in this study. For trifold CdTe NRs, corresponding to the 0.1 g of KOH, due to the low basicity of the solution, the reducibility of L-ascorbic acid was weak, and the crystal growth rate was slow, resulting in the obvious anisotropic products, as shown in figure 9(a). Low reduction rate and growth rate are beneficial to obtain the trifold CdTe NRs structure with smooth surface and uniform size. However, with the increasing of the amount of KOH, the reducibility of L-ascorbic acid increased significantly. There were large number of CdTe crystal nuclei in the solution when using 0.5 g KOH. At this point, a lot of CdTe crystal nuclei flew into the depression area of the trifold NRs, and finally formed nanospheres, as shown in figure 9(b).

4. Conclusion

In this paper, trifold CdTe NRs and CdTe NSs with different morphologies were prepared by the solvothermal process with K₂TeO₃ and CdCl₂ as the raw materials, L-ascorbic acid and ethylene glycol as the reducing agents and the solvents, and PVP as the surfactants, respectively. The morphology of the products was regulated by adjusting the reducibility of L-ascorbic acid. The evolution of the morphology of products is also analyzed, which provides important reference value for research in related fields.

Large number of experimental studies have shown that the reducibility of L-ascorbic acid can be adjusted by the amount of KOH. By fully reacting in an oven for 36 h, trifold CdTe NRs could be prepared with 0.1 g of KOH before the reaction; while CdTe NSs were obtained with 0.5 g of KOH. During the solvothermal process, the reducibility of L-ascorbic acid was enhanced by the increasing of the amount of KOH, the number of CdTe crystal nuclei increased as a result. For higher reducibility, the newly formed nuclei were preferentially adsorbed in the depressions at both ends of the trifold NRs and completed the crystallization process. The trifold NRs structure will gradually evolve into NSs structure as a result.

Acknowledgments

This work was financed by the Natural Science Foundation of Qinghai Province, Grant No. 2018-ZJ-957Q. The authors also thank the group members for valuable discussions.
Data availability statement

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

Conflicts of interest

There are no conflicts to declare.

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