

The preparation of CdO nanowires from solid-state transformation of a layered metal-organic framework

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Abstract

A new coordination polymer formulated as $[C_8H_{10}CdO_7]_n \cdot 4H_2O$ has been prepared via a hydrothermal procedure by using 1,4-benzenedicarboxylic (p-BDC) and Cd^{II} salt as starting materials. The structure was determined by single-crystal X-ray diffraction and the result shows that the complex crystallizes in orthorhombic system, space group $Pcca$, with $M_r = 402.62$, $a = 7.293(2) \text{ \AA}$, $b = 9.980(3) \text{ \AA}$, $c = 19.889(6) \text{ \AA}$, $V = 1447.6(8) \text{ \AA}^3$, $Z = 4$, $D_c = 1.847 \text{ g/cm}^3$, $F(000) = 808$, $\mu(\text{MoK}\alpha) = 1.559 \text{ mm}^{-1}$, $R = 0.0478$, $wR = 0.1150$, $GOF = 1.199$. It displays a neutral layered framework along ab plane constructed by hydrogen-bonding interaction through infinite zigzag chains. Its thermal decomposition and solid-state transformation course between 30 and 550 °C was recorded by TG curve and XRD pattern, respectively. Interestingly, it is found that at higher temperature the crystal material was converted to uniform CdO nanowires, suggesting an effective and reasonable complex-precursor procedure for preparing one-dimensional crystalline nanomaterials.

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1. Introduction

During the past decade, one-dimensional (1D) nano-sized structure materials have sparked a worldwide interest owing to their unique electronic, magnetic, optical and mechanical properties [1–4]. Nanowires, which represent one kind of 1D structured material, have attracted particular attention because of their potential applications in nanodevices and functional materials [5–10]. Many methods have been used to prepare nanowires, such as electrochemistry [5], template (mesoporous silica, carbon nanotubes, etc.) [6], emulsion or polymeric system [7], solution [8], vapor transport [9] and organometallic method [10].

Recently, it has been found that some complexes which have chain-like or tube-like metal-organic framework architecture can be used as precursors to grow 1D nano-

sized structure material. For example, Yi Xie et al. prepared Cu_2O monocrystalline nanowires via a novel complex-precursor surfactant-assisted (CPSA) route, in which linear alignment of copper cations in complex as precursors provided orientation for the growth of Cu_2O nanowires [11]. Loye and co-workers [12] reported the preparation of ZnO and $ZnCN_2$ nanorod from a coordination polymer precursor (an exceptionally stable, hollow tubular metal-organic framework) by heating the precursor in the presence of O_2 and N_2 at higher temperature. These works have drawn our attention because they provided a novel strategy for the preparation of 1D structured materials. Most remarkably, the relationship between molecular structure and morphology of as-desired materials was well examined.

In particular, the design and synthesis of coordination polymer with 1D helical or zigzag chain structures are of great interest on account of its diverse geometry of chains, packing styles and potential applications in the areas of fluorescent and magnetic materials [13–26]. Many single,

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double helical or zigzag chains have been generated by self-assembly processes. Although rational design strategy for preparing 1D chain structures has been reported [20,21], controlling the way of molecular components assembled in the crystalline phase remains a great challenge since the self-assembly process is heavily influenced by many factors, such as coordination preference of metal ion, solubility of the organic link and metal salt, solvent polarity, crystallization conditions, the metal/ligand ration and so on [27–30].

On the other hand, 1D semiconductor nano-sized structure materials are known to have many interesting physical properties and great applications in opto-electronic devices. As a II–IV n-type semiconductor materials, CdO, which exhibits a large direct band gap of 2.4 eV, is a promising candidate for opto-electronic applications such as solar cells, phototransistors, photodiodes, transparent electrodes, and gas sensors [31–33].

In this work, we synthesized a new coordination polymer with layered metal-organic framework assembled by 1D zigzag chains via a hydrothermal procedure by executing 1:1 stoichiometry [34] of 1,4-benzenedicarboxylic (p-BDC) to Cd^{II} salt. Using the coordination polymer as precursor, its solid-state transformation course at higher temperature has been studied. It is interesting to note that at high temperature the crystal material was converted into uniform CdO nanowires, confirmed by transmission electron microscopy (TEM) image and energy-dispersive X-ray (EDX) spectrum, suggesting an effective and reasonable procedure to grow 1D nano-sized structure materials from coordination polymer precursor.

2. Experimental

2.1. General procedures

All chemicals were of reagent grade and were used as received. Elemental analysis was performed on a Pekin-Elmer 1400C analyzer. FT-IR spectra was recorded from KBr pellets in the range of 400–4000 cm⁻¹ on a Nicolet 5DX spectrometer. Thermogravimetric analysis was carried out on a BOIF-WCT-2A instrument in flowing air with a heating rate of 20 °C/min. X-ray powder diffraction (XRD) patterns were acquired on a Bruker D8 powder X-ray diffractometer with graphite monochromatized CuK α radiation ($\lambda = 1.5406 \text{ \AA}$). Microscopic morphology of CdO nanowires was visualized by TEM (JEM 2100) operated at an acceleration voltage of 200 kV.

2.2. Synthesis of $[\text{C}_8\text{H}_{10}\text{CdO}_7]_n \cdot 4\text{H}_2\text{O}$

Cd(CH₃COO)₂ · 2H₂O (1 mmol), p-BDC (1 mmol), H₂O (10 ml) and DMF (5 ml) were mixed in a 50 ml beaker, then sealed in Teflon-lined stainless steel autoclave reactor at 90 °C for 3 days. Subsequently, the reactor was cooled down to room temperature at a cooling rate of 2 °C/h; colorless needle crystals of complex were collected by

filtration and washed by distilled water. Yield: 38%, based on Cd. Anal. Calcd. For C₈H₁₈CdO₁₁ (%): C, 23.86; H, 4.51; O, 43.71; Found: C, 23.79; H, 4.58; O, 43.77. IR spectrum (cm⁻¹), (1) Complex: 3552(m), 3420(m), 1696(w), 1566(s), 1504(m), 1393(s), 1303(w), 1101(w), 1011(m), 889(w), 838(s), 789(w), 748(s), 526(s). (2) Solid-state transformed products: 3420(w), 1386(m), 852(w).

2.3. Solid-state transformation

The freshly prepared crystals were loaded into an alumina boat which was placed in the center of a high-temperature tube furnace. The sample was heated in air at a rate of 5 °C/min in a step-wise manner. At each step, the temperature was maintained for 2 h prior to any experiment. The X-ray diffraction pattern of heated sample was recorded at each temperature.

2.4. Crystal structure determination

A colorless needle crystal of complex (0.50 × 0.09 × 0.05 mm³) was subjected to single-crystal X-ray diffraction using a Bruker SMART 1000 CCD diffractometer instrument equipped with graphite monochromated MoK α radiation ($\lambda = 0.71073 \text{ \AA}$) at 293(2) K. Data collection and reduction were performed using the SMART and SAINT software. An empirical absorption correction (SADABS) was applied to the raw intensities. The structures were solved by direct methods and refined by full-matrix least-squares based on F^2 using the SHELXTL program package. All of non-hydrogen atoms were refined anisotropically. Hydrogen atoms were located from difference Fourier maps. The crystal data and structure refinement parameters are listed in Table 1.

3. Results and discussion

3.1. Description of crystal structure

X-ray crystallography analysis shows that the complex adopts a 1D structure, where Cd^{II} ions are chelated by bridging linkers (p-BDC) in a bis-bidentate mode [35] to give a linear zigzag chain, as shown in Fig. 1a. The coordination environment of Cd^{II} is shown in Fig. 1b, it is seven-coordinated by oxygen atoms, three of which are (O3, O4, O4A) contributed from water and the rests (O1, O2, O1A, O2A) are from carboxylic group of two p-BDC ligands. After careful structure analysis, it can be found that each repeated $[\text{Cd}(\text{p-BDC})_2]^{2-}$ unit in the chain lies on one plane. Additionally, O3 from H₂O is on the plane as well. The mode of O4 and O4A from H₂O bound to Cd^{II} is symmetric (Cd–O4 2.264(5) Å, Cd–O4A 2.264(5) Å) and nearly perpendicular to the plane as evidenced by the angles (O4–Cd–O1 88.69(16)°, O4–Cd–O2 88.14(16)°, O4A–Cd–O1 90.53(15) Å, O4A–Cd–O2 92.00(16) Å). The distances of Cd–O1 and Cd–O2 are quite similar to that reported results of Cd–OCO structure of p-BDC complexes

[36–38]. The related bond lengths and angles are listed in Table 2.

It is well known that hydrogen-bonding plays an important role in crystal engineering [39]. The interesting

Table 1

Crystal data and structure refinement of complex

Complex	
Empirical formula	C ₈ H ₁₈ CdO ₁₁
Formula weight	402.62
Temperature (K)	293(2)
Wavelength (Å)	0.71073
Crystal size (mm ³)	0.50 × 0.09 × 0.05
Crystal system	Orthorhombic
Space group	<i>Pcca</i>
<i>a</i> (Å)	7.293(2)
<i>b</i> (Å)	9.980(3)
<i>c</i> (Å)	19.889(6)
α (deg)	90
β (deg)	90
γ (deg)	90
<i>V</i> (Å ³)	1447.6(8)
<i>Z</i>	4
Calculated density (Mg/m ³)	1.847
Absorption coefficient (mm ⁻¹)	1.559
<i>F</i> (000)	808
θ range for data collection (deg)	2.04–26.08
Limiting indices	$-9 \leq h \leq 8, -12 \leq k \leq 12, -24 \leq l \leq 21$
Reflections collected/unique	7204/1407 [<i>R</i> (int) = 0.0330]
Max. and min. transmission	0.9233–0.5090
Refinement method	Full-matrix least-squares on <i>F</i> ²
Data/restraints/parameters	1407/0/92
Goodness-of-fit	1.199
Final <i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> ₁ = 0.0478, <i>wR</i> ₂ = 0.1150
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0526, <i>wR</i> ₂ = 0.1176
Largest diff. peak and hole (e Å ⁻³)	1.243 and -0.493

structure feature of the complex is the formation of layered metal-organic framework architecture constructed by the connections between chains through complete hydrogen-bonding interaction. As shown in Fig. 2, O4 of one chain as H-donors, interact with carboxyl O1 of neighboring chain (O...O 2.710(6) Å, \angle O–H...O 169°) and O5 of solvent H₂O molecules in the layer (O...O 2.845(6) Å, \angle O–H...O 138°), respectively. In addition, O3 of ligand H₂O is hydrogen-bonded with O5 of solvent H₂O as well (O...O 2.774(6) Å, \angle O–H...O 144°) (Table 3). Considering O4–H4A...O1 has the shortest distances (2.710(6) Å) and largest bond angle (169°), it is safe for us to drive that the interaction between O4 and O1 is the strongest one. It is worthwhile to note that uncoordinated solvent H₂O molecules act as layer filling particles and contribute to the buildup of layered framework by O4–H4B...O5 and O3–H3A...O5 interactions. These hydrogen-bondings make the parallelly oriented coordination polymer chains connected in solid state to construct a 2D layered structure.

Table 2

Selected bond lengths (Å) and bond angles (deg) of complex

Cd–O(1)	2.355(4)	O(3)–Cd–O(4)	90.54(11)
Cd–O(3)	2.244(6)	O(4)#1–Cd–O(4)	178.9(2)
Cd–O(2)	2.500(4)	O(3)–Cd–O(1)	136.01(9)
Cd–O(4)	2.264(5)	O(4)–Cd–O(1)	88.69(16)
Cd–O(1)#1	2.355(4)	O(3)–Cd–O(2)	82.47(10)
Cd–O(2)#1	2.500(4)	O(4)–Cd–O(2)	88.14(16)
Cd–O(4)#1	2.264(5)	O(1)–Cd–O(2)	53.54(13)
O(1)–C(1)	1.247(6)	O(1)#1–Cd–O(2)	141.51(13)
O(2)–C(1)	1.269(7)	O(2)#1–Cd–O(2)	164.95(19)

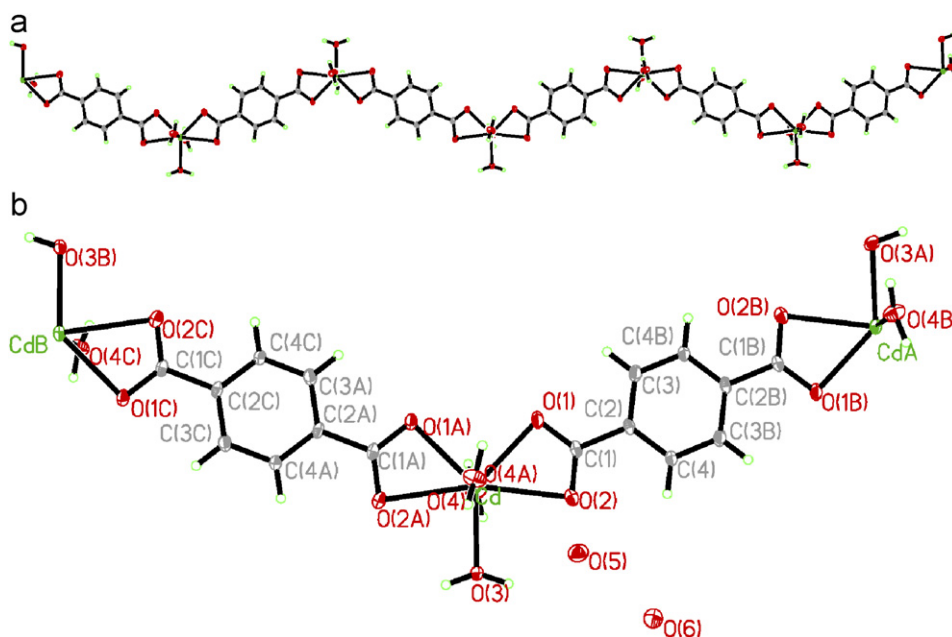
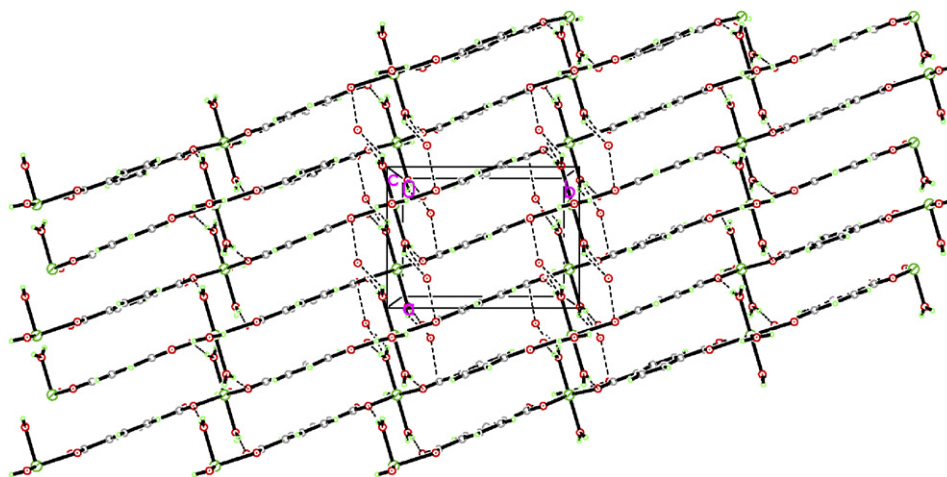
Symmetry codes: #1 $-x + \frac{1}{2}, -y, z$.

Fig. 1. (a) ORTEP diagram of one-dimensional zigzag chain with solvent molecules omitted for clarity and (b) ORTEP diagram of coordination environment of cadmium ion (hydrogen atoms of solvent H₂O were not added).

Fig. 2. ORTEP diagram of perspective view of packing diagram along *c*-axis.Table 3
Hydrogen bonding geometry (Å, deg) of complex

Donor–H⋯acceptor	<i>d</i> (D–H)	<i>d</i> (H⋯A)	<i>d</i> (D–H⋯A)	<i>a</i> (D–H⋯A)
O4–H4A⋯O1	0.85	1.87	2.710(6)	169
O3–H3A⋯O5 ^a	0.85	2.04	2.774(6)	144
O4–H4B⋯O5 ^b	0.85	2.15	2.845(6)	138

Symmetry codes: O1 = $-x, -y, -z$; O5^a = $\frac{1}{2}+x, -y, \frac{1}{2}-z$; O5^b = $-\frac{1}{2}-x, -y, z$.

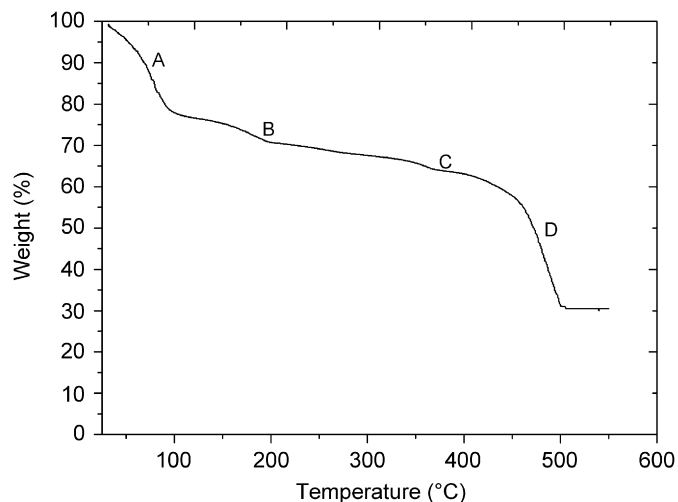


Fig. 3. TG curve of crystal sample measured in flowing air.

3.2. Thermogravimetric analysis (TGA) and solid-state transformation

TGA was carried out on the freshly prepared crystal samples. As shown in Fig. 3, a total of four weight losses were observed. A large weight loss (18.8%, designated as A) occurred immediately upon heating and completed before the temperature reached 100 °C, and this weight loss is indicative of the loss of four solvent water molecule per formula unit (calc. 17.9%). Subsequently, two intermittent, gradual weight loss (15.8%, designated as B and C) were

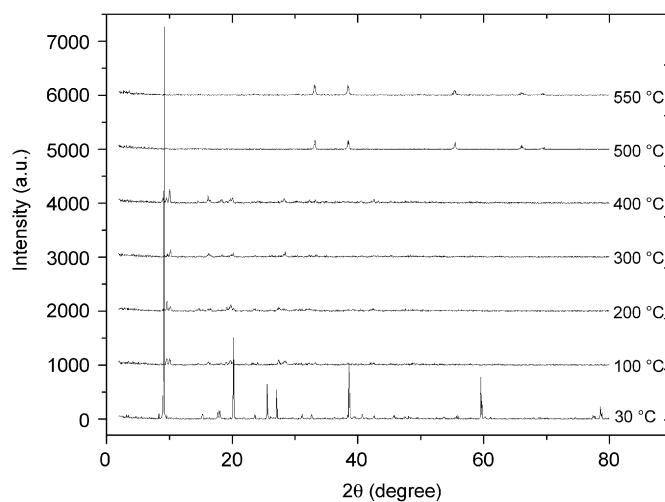


Fig. 4. X-ray powder diffraction pattern of crystal sample after heat treatment in flowing air between 30 and 550 °C.

observed between 100 and 400 °C corresponding to the liberation of three coordinated H₂O molecule (calc. 16.3%); and is followed by complete framework decomposition as indicated by abrupt weight loss at about 450 °C (designated D). To clarify the changes of crystallinity in each case, powder X-ray diffraction was performed to monitor this solid-state transformation as a function of temperature. The result shows that the crystal only maintain its crystallinity at low temperature, as shown in Fig. 4, when heated it to 100 °C, an amorphous solid was formed as illustrated by the disappearance of diffraction peaks in the XRD pattern. It is notable from the XRD pattern that the sample remains amorphous at the temperature range of 100–400 °C. Optical microscopy was also used to demonstrate that crystals lost transparency and crystallinity between 100 and 400 °C. After heated to 500 °C, the amorphous solid begins to recrystallize as shown by the appearance of new diffraction peaks in the XRD pattern. Meanwhile, the TG curve displayed a

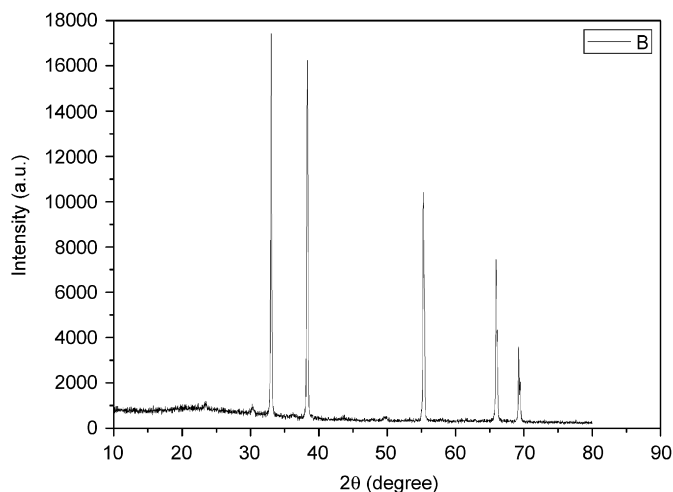


Fig. 5. X-ray powder diffraction pattern of crystal sample when heated to 550 °C.

continuous weight drop until the temperature reached about 520 °C. These findings suggest that a new crystalline phase forms while the framework falls apart. The phase characterization was performed by XRD pattern which identified the product as CdO with cubic structure (PDF 75-0591) as illustrated in Fig. 5.

The solid-state transformed sample was further investigated with TEM. Our experiment result indicates that the crystal sample was converted to uniform nanowires when heated to 55 °C, confirmed by TEM image. Fig. 6A is a typical TEM image showing the general view of morphology of wires. It is visible that the entangled wires are of high purity and their diameters range from several to a few ten nanometers, typically around 20 nm (Fig. 6D). The composition of obtained nanowires was then analyzed by EDX spectrum. As shown in Fig. 6B, only Cd and O are present in the sample, furthermore at % confirms the right stoichiometry for CdO nanowires. It is noteworthy from the morphology of nanowires showing in Fig. 6C and D

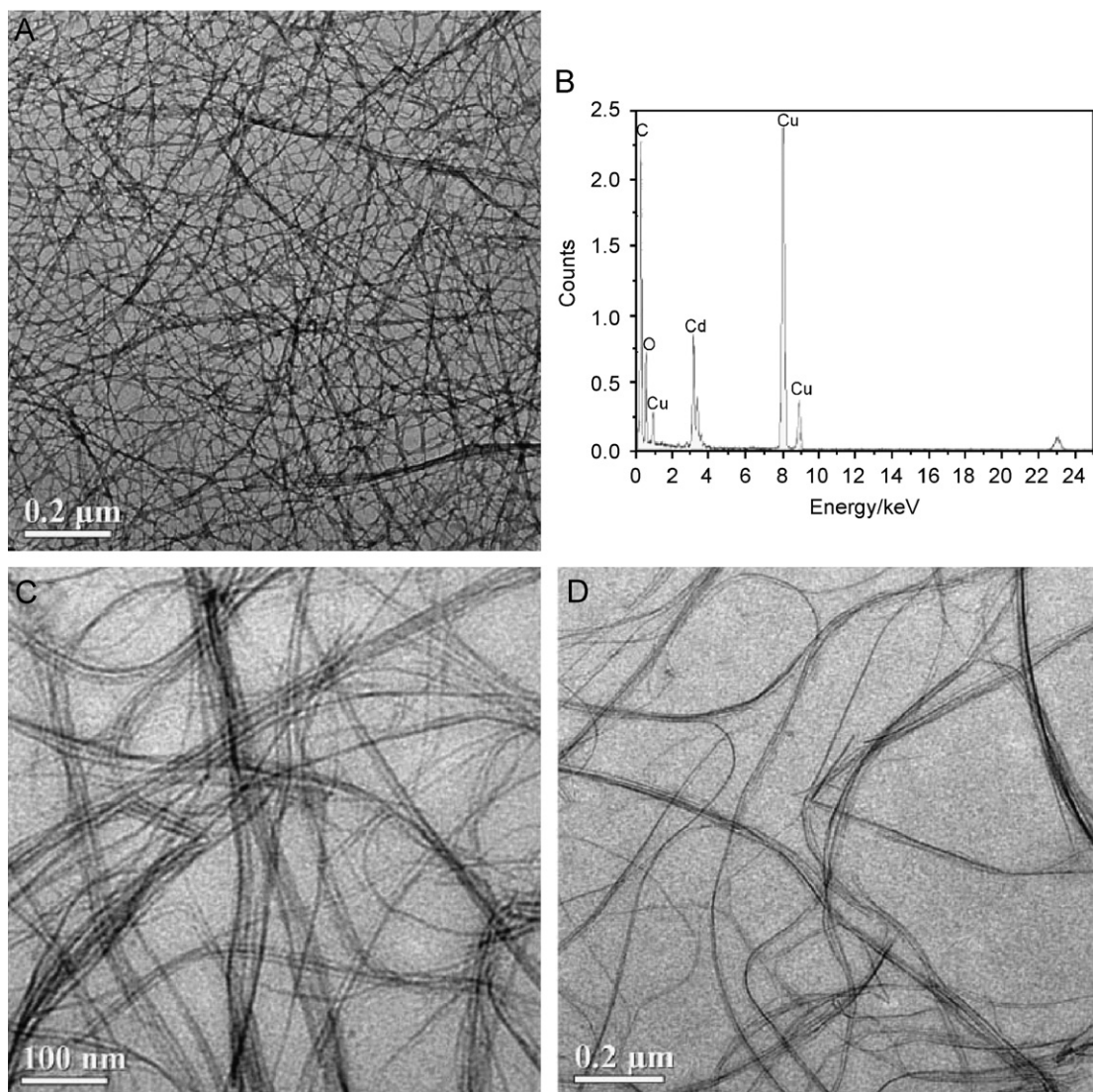


Fig. 6. TEM images of (A, C, D) and EDX spectrum (B) obtained from nanowires.

that some wires are in cluster shape assembled by several pieces of single wire.

Despite the exact transformation mechanism from complex to CdO nanowires is still not completely understood, a plausible one is that the appearance of nanowires was originated from 1D zigzag nanochains of crystal precursor. As a result, although the metal-organic framework decomposed and the chemical composition changed, the intrinsic microscopic morphology of as-synthesized crystal was “inherited” during the solid-state transformation course. In addition, because the zigzag chain morphology of coordination polymer precursor is inherited during the solid-state transformation, the resultant sizes of the CdO nanowires are fixed by the sizes of zigzag chains of the layered framework crystal precursor. To our knowledge, this is the only successful example to fabricate 1D metal oxide nanomaterials from solid-state transformation of coordination polymer precursor after the report by Loye et al. Furthermore, it clearly indicates that the procedure is effective and reasonable.

4. Conclusions

In summary, another 1D metal oxide nano-sized structure material was obtained via the complex-precursor strategy. As an example, CdO nanowires were prepared from solid-state transformation of the complex, in which a layered metal-organic framework assembled by 1D infinite zigzag chains was heated in the presence of oxygen at higher temperature. During the solid-state transformation course, the 1D chain morphology of molecular dimension was inherited and confined the diameter of nanowires, confirmed by TEM and EDX of solid-state transformed products, suggesting an effective and reasonable procedure for fabricating 1D inorganic nanomaterials. We expect that it could be of great importance to prepare other 1D nano-sized structure oxides.

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Appendix A. Supplementary material

Crystallographic data for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 275216. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1223 336 033; E-mail address: deposit@ccdc.cam.ac.uk).

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