



Novel radial vanadium pentoxide nanobelt clusters for Li-ion batteries



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ABSTRACT

This paper reports the synthesis, characterization and Li-ion intercalation properties of moundlily-like radial vanadium pentoxide (V_2O_5) nanobelt clusters. The V_2O_5 nanobelt clusters was successfully synthesized by a novel soft template assisted hydrothermal process followed by thermal annealing. The as-prepared products were characterized by X-ray diffraction, thermogravimetric analysis, FT-IR spectrometry, scanning electron microscopy and high resolution transmission electron microscopy. The obtained V_2O_5 possesses a single-crystalline structure with a preferred orientation along the [010] crystal plane. Electrochemical analysis shows that the specific discharge capacity of the V_2O_5 nanobelt clusters reaches 134 mA h/g at a current density of 2 A/g coupled with good cycle stability.

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1. Introduction

As is well known, inorganic metal oxide semiconductors have been considered as important functional materials due to their outstanding physical and chemical properties [1–17]. Vanadium pentoxide (V_2O_5) has drawn great interest for the potential applications of super capacitors [18–20], light photocatalysis [21,22], lithium ion batteries [23–27], sensors [28,29] and catalysts [30], due to its low-cost, abundance, easy synthesis, high energy density and good stability [31–33]. V_2O_5 is also a typical intercalation compound because of its layered crystal structure, which plays an important role in Li-ion intercalation applications. It is known that most advanced functional inorganic materials greatly depend on their crystalline structure, morphology, dimensionality and size [34,35], etc. Moreover, nanostructured materials display high surface-to-volume ratio and short ions diffusion distance, resulting in high charge/discharge rates [36]. Thus, the development of controllable syntheses of V_2O_5 nanostructures with excellent performance is of paramount importance. Until now, a variety of approaches, such as hydrothermal synthesis [28,29,37], electrodeposition [38,39], atomic layer deposition technique [40], sol–gel route [41], have already been carried out in the design and preparation of various nanostructures of V_2O_5 for electrochemical energy storage. Among them, the hydrothermal synthesis as a means of preparing V_2O_5 is considered to be a convenient and versatile method, which possesses potential advantages of considerable

well-controlled morphology, high crystallinity and purity, as well as relatively low cost [42,43]. It is particularly worth noting the uncomplicated hydrothermal treatment in combination with soft template generatings could result in nanostructures with outstanding reliability, flexibility, selectivity, and diversity. For example, Chou et al. used an ultrasonic assisted hydrothermal method and combined with a post-annealing process to obtain various V_2O_5 nanomaterials, including nanoribbons, nanowires, and microflakes by using different surfactants, which exhibited enhanced electrochemical performance [44]. The preparation of single-crystalline vanadium oxide nanobelts by a facile hydrothermal treatment of bulky V_2O_5 powder in aqueous solution with the help of the surfactant sodium dodecylsulfate (SDS) has been reported by Cao et al. [45]. They found that the morphology and the structure of the as-prepared product had an obvious effect on the Li-ion intercalation properties. Mohan and co-workers [46] synthesized V_2O_5 nanotubes by hydrothermal condensation using 1-hexadecylamine (HDA) and PEO as a template and surface reactant, respectively. The battery performance of the V_2O_5 nanotubes was evaluated by the cyclic voltammograms and discharge curves. Up to now, the sizes of the majority of the reported V_2O_5 nanobelts are up to several hundreds of micrometers in length and the morphology-controllable synthesis of moundlily-like radial V_2O_5 nanobelts clusters was rarely reported. Han et al. has successfully synthesized the similar biomimic moundlily-like β - $AgVO_3$ nanowires which can reduce the self-aggregation and effectively improve the electrochemical performance [47].

Herein, a facile and efficient block copolymers assisted-hydrothermal route was designed to synthesize the $NH_4V_4O_{10}$ precursor. The moundlily-like V_2O_5 nanobelt clusters were

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obtained via the thermal decomposition of $\text{NH}_4\text{V}_4\text{O}_{10}$ precursor. The dimension, morphology and phase structure of the resulting products were selectively controlled through adjusting the experimental parameters such as the time and temperature of the hydrothermal reaction, and the calcination temperature. Furthermore, the Li-ion intercalation behaviors of the V_2O_5 were also investigated.

2. Experimental

All the reagents were of analytical grade and were used without further purification. In a typical procedure, 0.5 g of poly (ethylene oxide)-block-poly (propylene oxide)-block-poly (ethylene oxide) copolymer (P123 molecular weight: 5800) and 0.2 g of NH_4VO_3 were mixed in 40 mL of deionized water and magnetically stirred at 40 °C to form a clear yellow solution. Then, 4 mL of isopropyl alcohol was dropped into the above solution and stirred for 10 min. The final solution was transferred into a 50 mL Teflon-lined autoclave with a stainless shell. The autoclave was heated at 160 °C for 24 h and then allowed to cool down to room temperature. The dark green precipitates were collected, washed with deionized water and ethanol for several times, and dried at 60 °C for 24 h. Subsequently, the product was calcined at 300, 400 and 500 °C for 2 h in air, and the corresponding products were denoted as S1, S2 and S3, respectively.

The phase structures of the products were analyzed by a MSAL-XD2 X-ray diffractometer (Cu K α , 36 kV, 20 mA, $\lambda = 1.5406 \text{ \AA}$). Thermogravimetric analysis (TGA) was carried out on a Perkin–Elmer TGA 7 thermal analyzer under N_2 atmosphere from room temperature to 600 °C at a heating rate of 10 °C/min. The functional groups of the samples were detected through a Nicolet 6700 FT-IR spectrometer. The morphologies were examined by scanning electron microscope (SEM, Philips SEM-XL30S), transmission electron microscopy (TEM, Philips TECNAI-10) and high resolution transmission electron microscope (HRTEM, JEOL JEM-2100F) with an accelerating voltage of 200 kV.

Electrochemical measurements were performed on a CHI 660B electrochemical workstation. A working electrode was obtained via pressing the mixture of active materials, carbon black and 5%-PTFE (80:15:5 wt%) into a $12 \times 16 \text{ mm}$ nickel foam electrode. The experiments were carried out in a standard three electrodes cell at room temperature, with a platinum foil as counter electrode, a double salt bridge saturated SCE electrode as reference electrode and the above-mentioned electrode as working electrode. The working electrode was characterized by cyclic voltammetry (CV) and galvanostatic charge/discharge in 1 M LiClO_4 /ethylene carbonate (EC)/dimethyl carbonate (DMC) (1:1 v/v).

3. Results and discussion

Fig. 1a shows the XRD patterns of the precursors with different hydrothermal reaction times at 160 °C. All the sharp and strong diffraction peaks of the products are consistent with those of the monoclinic $\text{NH}_4\text{V}_4\text{O}_{10}$ (JCPDS 31-0075). With the increase in the hydrothermal time, it is obvious that the intensity of peaks gradually increased, indicating the enhancement of crystallinity. Fig. 1b shows the XRD patterns of the samples with different calcination temperatures. The XRD spectrum of S1 presents diffraction

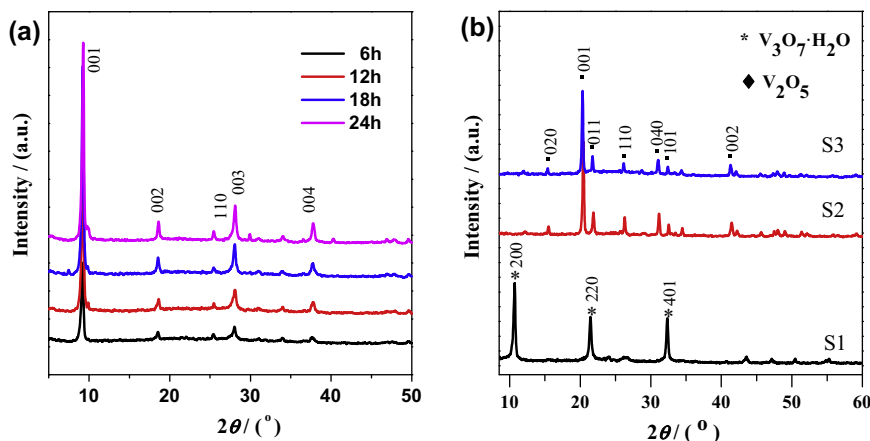


Fig. 1. XRD patterns of (a) the precursors prepared at different hydrothermal times at 160 °C; (b) the samples calcined at different temperatures: S1: 300 °C, S2: 400 °C and S3: 500 °C.

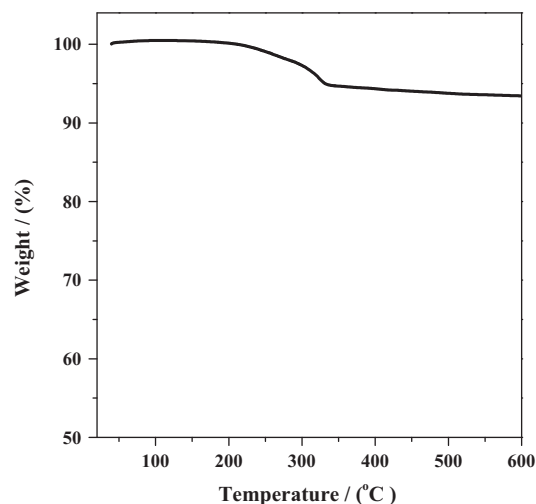


Fig. 2. TGA curve of the $\text{NH}_4\text{V}_4\text{O}_{10}$ precursor.

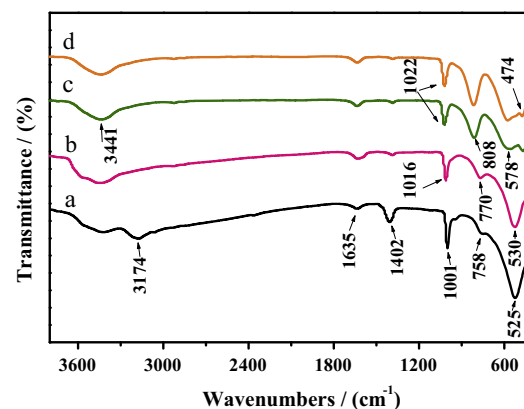


Fig. 3. FT-IR spectra of (a) $\text{NH}_4\text{V}_4\text{O}_{10}$ precursor, (b) S1: $\text{V}_3\text{O}_7 \cdot \text{H}_2\text{O}$, (c) S2: V_2O_5 and (d) S3: V_2O_5 .

peaks at $2\theta = 10.5^\circ$, 21.4° and 32.3° corresponding to the orthorhombic $\text{V}_3\text{O}_7 \cdot \text{H}_2\text{O}$ (JCPDS 85-2401). When the calcination temperature was increased to 400 and 500 °C, the characteristic peaks of S2 and S3 are well matched with the those of the orthorhombic phase V_2O_5 (JCPDS 85-0601). It can be seen from these results that pure V_2O_5 can be obtained via the thermal decomposition of $\text{NH}_4\text{V}_4\text{O}_{10}$ precursor at 400 °C or above.

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