



## Short communication

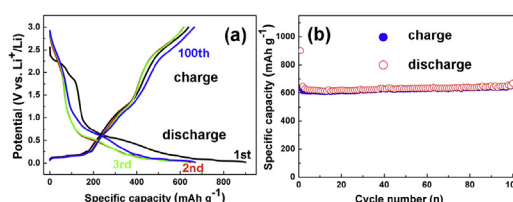
## Electrochemical performance of cobalt vanadium oxide/natural graphite as anode for lithium ion batteries

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## HIGHLIGHTS

- A new and facile way was developed to prepare CoV<sub>2</sub>O<sub>6</sub>/NG electrode.
- Systemic study on the electrochemical characteristics of CoV<sub>2</sub>O<sub>6</sub>/NG electrode.
- Clear clarification of the charge/discharge mechanism of CoV<sub>2</sub>O<sub>6</sub>.
- The CoV<sub>2</sub>O<sub>6</sub>/NG anode exhibits high capacity and excellent cycle performance.

## GRAPHICAL ABSTRACT



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## ABSTRACT

CoV<sub>2</sub>O<sub>6</sub>/natural graphite electrode with sodium alginate binder is prepared, which shows excellent electrochemical performance as anode for Li-ion batteries. It exhibits initial discharge and charge capacity of 902 and 638 mAh g<sup>-1</sup> at a specific current of 110 mA g<sup>-1</sup>. After 100 cycles, the discharge and charge capacity maintain of 669 and 665 mAh g<sup>-1</sup>, respectively. The charge/discharge mechanism of CoV<sub>2</sub>O<sub>6</sub> is also studied, suggesting a structure variation in discharging, which involves the initial formation of LiV<sub>2</sub>O<sub>5</sub> and Co<sub>3</sub>V<sub>2</sub>O<sub>8</sub>, the subsequent transition from Co<sub>3</sub>V<sub>2</sub>O<sub>8</sub> to Li<sub>x</sub>V<sub>2</sub>O<sub>5</sub> and CoO, and the later reduction of CoO into Co. The structure variation of Co<sub>3</sub>V<sub>2</sub>O<sub>8</sub> accompanies by an amorphization process, which maintains in the subsequent discharging and charging process.

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## 1. Introduction

High performance electrode materials have played crucial roles in the development of Li-ion batteries. By now, many sorts of cathodes such as LiNi<sub>x</sub>Mn<sub>y</sub>Co<sub>1-x-y</sub>O<sub>2</sub> and LiFePO<sub>4</sub> have been successfully commercialized since the first application of LiCoO<sub>2</sub> as cathode for Li-ion batteries [1–3]. However, the dominant anode in commercial Li-ion batteries has always been graphite material,

which is determined by both the stable electrochemical performance and the mature fabrication technology of graphite. Searching for new advanced anode materials with higher capacity and better cycle stability is crucial for the development of high performance Li-ion batteries. Further more, good compatibility with graphite material may be beneficial for the practical application of new anode materials.

As known, natural graphite (NG) shows advantages over other carbon materials in terms of low cost, abundance in nature, good electronic conductivity and fine electrochemical performance, which has become the uppermost graphite anode for commercial Li-ion batteries. Certainly, it can be used as an ideal carbon component in composite material for further constructing high

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performance anode [4,5]. As a new sort of anode,  $\text{CoV}_2\text{O}_6$  shows obvious plateau in discharge curve [6], endowing it with potential application in Li-ion batteries. However, systemically evaluation on the electrochemical performance of  $\text{CoV}_2\text{O}_6$  has not been reported by now, which is still under way. Here in this paper, we report the preparation of  $\text{CoV}_2\text{O}_6$  via a facile way, and the electrochemical performance of  $\text{CoV}_2\text{O}_6/\text{NG}$  composite as a new sort of anode for Li-ion batteries was systemically studied. High specific capacity and excellent cycle stability of the  $\text{CoV}_2\text{O}_6/\text{NG}$  electrode demonstrate great potential of  $\text{CoV}_2\text{O}_6$  as anode for Li-ion batteries.

## 2. Experimental section

### 2.1. Fabrication procedure

The chemicals were analytical grade and purchased from Shanghai Chemical Reagents. NG was obtained from Yichang Hengda graphite company (99.9%). In a typical procedure, 2 mmol  $\text{C}_4\text{H}_6\text{CoO}_4 \cdot 4\text{H}_2\text{O}$ , 2–3 mmol  $\text{V}_2\text{O}_5$  and 2 mmol glucose were dissolved in 30 ml distilled water. After stirring for 20 min, the homogeneous yellowy suspension was transferred into a 50 ml teflonlined autoclave, distilled water was subsequently added to 80% of its capacity. The autoclave was at last sealed and placed in an oven, heated at 160 °C for 24 h. The final products were washed by distilled water and ethanol both four times and dried in an oven, then sintered in air atmosphere at 600 °C for 5 h (the products obtained with 2 and 3 mmol  $\text{V}_2\text{O}_5$  were defined as sample a and b, respectively). For preparing  $\text{CoV}_2\text{O}_6/\text{NG}$  composite, NG and  $\text{CoV}_2\text{O}_6$  with weight ratio of 1:1 were manually rubbed for 30 min.

### 2.2. Structure and morphology characterization

The structure and morphology of the resulting products were characterized by X-Ray powder diffraction (XRD, Rigaku Ultima IV, Cu K $\alpha$  radiation,  $\lambda = 1.5406 \text{ \AA}$ ), field-emission scanning electron microscopy (FE-SEM JSM 7500F, JEOL) and transmission electron microscopy (JEOL, JEM-2100F) equipped with selected area electron diffraction (SAED). For the morphology and structure characterization of the electrode after charge and discharge testing, the cells were disassembled in glove box and the electrode was washed with dimethyl carbonate.

### 2.3. Electrochemical characterization

Before the assembly of Li-ion battery, a mixture of  $\text{CoV}_2\text{O}_6/\text{NG}$  (80 wt%), acetylene black (10 wt%), and sodium alginate (SA, dissolved in deionized water, 0.02 g ml<sup>-1</sup>, 10 wt%) were coated on copper foil and cut into disc electrodes with a diameter of 14 mm using a punch. Coin-type cells (2025) of Li/1 M LiPF<sub>6</sub> in ethylene carbonate, dimethyl carbonate and diethyl carbonate (EC/DMC/DEC, 1:1:1 v/v/v)/ $\text{CoV}_2\text{O}_6/\text{NG}$  disc electrode were assembled in an argon-filled dry box (MIKROUNA, Super 1220/750, H<sub>2</sub>O < 1.0 ppm, O<sub>2</sub> < 1.0 ppm). A Celgard 2400 microporous polypropylene was used as separator membrane. The cells were tested in the voltage region between 0.02 and 3 V with a multichannel battery test system (LAND CT2001A). When calculating the specific capacity of the electrode, the weight of both  $\text{CoV}_2\text{O}_6$  and NG was considered as the total weight of active materials. The cyclic voltammetry (CV) measurement of the electrodes was carried out on a CHI660C electrochemical workstation at a scan rate of 0.2 mV s<sup>-1</sup> between 0 and 3 V.

## 3. Results and discussion

XRD patterns of sample a and b are shown in Fig. 1. For sample a, diffraction peaks located at 20.4°, 27.5°, 28.8°, 29.4°, 32.9°, 38.9°,

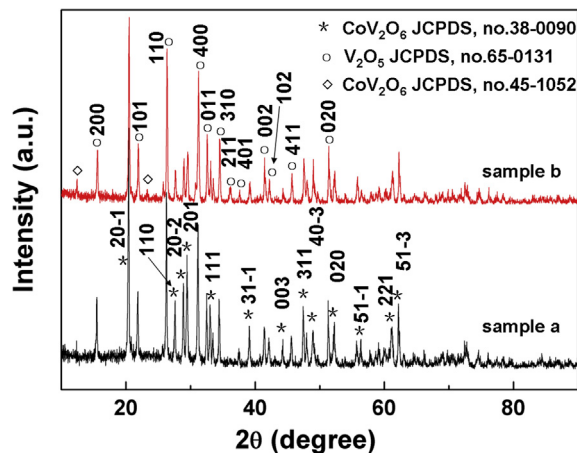


Fig. 1. XRD patterns of sample a and sample b.

41.1°, 47.3°, 48.7°, 52.2°, 56.4°, 61.0°, and 62.1° can be attributed to the (20-1), (110), (20-2), (201), (111), (31-1), (003), (311), (40-3), (020), (51-1), (221) and (51-3) faces of anorthic  $\text{CoV}_2\text{O}_6$  (JCPDS, no. 38-0090). Meanwhile, diffraction peaks at 15.4°, 21.7°, 26.2°, 31.1°, 32.4°, 34.3°, 36.1°, 37.4°, 41.3°, 42.1°, 45.5°, and 51.2° correspond to the (200), (101), (110), (400), (011), (310), (210), (401), (002), (102), (411) and (020) faces of orthorhombic  $\text{V}_2\text{O}_5$  (JCPDS, no. 65-0131). Tuning the mole ratio between  $\text{C}_4\text{H}_6\text{CoO}_4 \cdot 4\text{H}_2\text{O}$  and  $\text{V}_2\text{O}_5$  precursors is not a guarantee to improve the purity of the anorthic  $\text{CoV}_2\text{O}_6$ . As seen, excepting the typical diffraction peaks of  $\text{CoV}_2\text{O}_6$  and  $\text{V}_2\text{O}_5$ , sample b shows enhanced diffraction peaks of other phase of  $\text{CoV}_2\text{O}_6$  (JCPDS, no. 45-1052).

Fig. 2(a) is a low magnification SEM image of sample a, which exhibits a large number of micro-particles and micro-rods. The mean size of these micro-particles is about 200 nm, and the length of these micro-rods ranges from 500 nm to 2.5  $\mu\text{m}$ . A high magnification SEM image of sample a is shown in Fig. 2(b). As seen, these micro-particles exhibit spherical morphology with smooth surface. These micro-rods exhibit smooth surface with mean diameter about 300 nm. For further clarifying the morphology and microstructure of the  $\text{CoV}_2\text{O}_6$ , TEM and HRTEM images were provided. As shown in a TEM image in Fig. 2(c), the  $\text{CoV}_2\text{O}_6$  shows clear sphere-like and rod-like morphologies, which are in accordance with those in SEM images. Fig. 2(d) is a high-resolution TEM (HRTEM) image of a single micro-rod, which shows clear lattice fringes. The interplanar spacing is about 0.272 nm, which corresponds to the (111) face of monoclinic  $\text{CoV}_2\text{O}_6$ . A SAED pattern of a single micro-rod is shown in Fig. 2(e), which exhibit regular diffraction spots, suggesting the as-synthesized  $\text{CoV}_2\text{O}_6$  is well crystallized.

Charge/discharge test of the  $\text{CoV}_2\text{O}_6/\text{NG}$  (sample a) electrode was carried out in the potential window of 0.02–3.0 V versus Li. Fig. 3(a) is the initial three and the 100th charge and discharge curves at a specific current of 110 mA g<sup>-1</sup>. As seen, the initial discharge curve differs much from the subsequent two curves, showing a short sloping potential region from 2.1 to 1.7 V, a sharp voltage decrease from 1.7 to 0.7 V, and a long sloping potential region from 0.7 to 0.02 V for multistep electrochemical reactions for the generation of  $\text{LiV}_2\text{O}_5$  and  $\text{Co}_3\text{V}_2\text{O}_8$ , the subsequent transition from  $\text{Co}_3\text{V}_2\text{O}_8$  to  $\text{Li}_x\text{V}_2\text{O}_5$  and CoO, the reduction of CoO into Co, as well as the insertion of lithium ions into NG [4,5,7,8]. The short potential region disappears in the subsequent discharge curves, suggesting irreversible phase transition in the initial cycle, which is similar to that of  $\text{CuV}_2\text{O}_6$  [9]. The subsequent discharge curves exhibit two sloping potential regions from 2.5 to 0.7 V and 0.7 to 0.02 V, which correspond to the formation of  $\text{Li}_x\text{V}_2\text{O}_5$  and Co as well

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