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Non-hydrothermal synthesis of $(NH_4)_2V_3O_8$ hierarchical flowers and their conversion into V_2O_5 for lithium ion battery



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ABSTRACT

 $(NH_4)_2V_3O_8$ microflowers were synthesized through a non-hydrothermal/solvothermal method. And 3D hierarchical $(NH_4)_2V_3O_8$ as precursor could be easily transformed into V_2O_5 with preserved microstructure *via* subsequent calcination. The thickness of nanosheets (petals) can be controlled by varying the ratio of ethanol/water in the deposition system. 3D hierarchical V_2O_5 flowers demonstrate a very high capacity (289 mA h g⁻¹) and good cyclic stability.

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1. Introduction

The emerging two-dimensional (2D) ultrathin nanosheets have received tremendous attention for their applications in electronics, energy devices and sensors [1–6]. Due to the aggregation of nanosheets resulted from their high surface energy, substantial efforts have been made on the facile and large-scale synthesis of 3D hierarchical structures assembled from nanosheets to conquer this problem. Hydrothermal/solvothermal method has been employed as a common synthetic strategy for the fabrication of flower-like structures [7–11]. However, these methods have become well-known bottlenecks for large-scale production because of rigorous reaction conditions and high cost. Therefore, it is highly desirable to develop non-hydrothermal/solvothermal approaches [12] for the controllable fabrication of nanosheets assembled flower-like hierarchical structures.

Vanadium oxides are a family of promising materials with exceptional energy storage and photoelectronic [13–17]. Especially, several nanostructured vanadium oxides have been extensively studied as high capacity cathode materials for lithium ion batteries (LIBs) in the past decades [18–21]. However, controllable fabrication of flower-like hierarchical vanadium oxides through non-hydrothermal/solvothermal process remains a great challenge. Herein, we report the controllable synthesis of flower-like hierarchical (NH₄)₂V₃O₈ and V₂O₅ through a non-hydrothermal/solvothermal method, that is, (NH₄)₂V₃O₈ microflowers were firstly

prepared by direct deposition in water/ethanol system, then transformed into V_2O_5 *via* heat treatment. The thickness of petals can be simply tuned by adjusting the ratio of ethanol/water. The yielding V_2O_5 microflowers demonstrate a very high capacity and good cyclic stability when evaluated as cathode materials for LIBs.

2. Experimental section

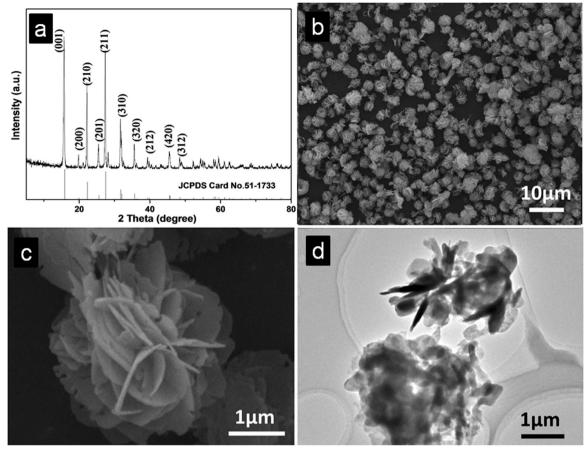
2.1. Synthesis of flower-like $(NH_4)_2V_3O_8$ and V_2O_5

In a typical synthesis, 0.1 mol ammonium vanadate $((NH_4)_2V_3O_8)$ was dissolved in 70 mL DI water with vigorous stirring for half an hour at ambient temperature. Subsequently, ethanol (30 mL) was dropped with continuous stirring. And then ammonium hydroxide was carefully added to adjust the pH value of reaction system to 10. The obtained slurry was heated in 40 °C water bath for 8 h. The finally prepared flower-like $(NH_4)_2V_3O_8$ was filtered, washed thoroughly with absolute ethanol, dried at 60 °C for 12 h in a vacuum oven. The V_2O_5 product can be obtained by calcination of precursor at 400 °C (heating rate: 5 °C/min) under air for one hour.

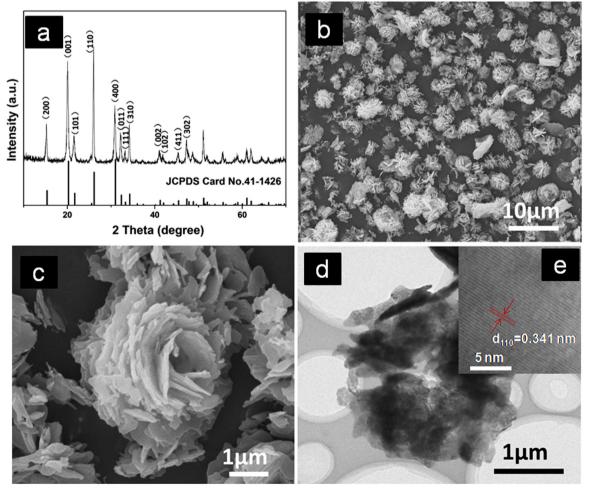
2.2. Characterization

The morphology of the as-prepared products was tested by field-emission scanning electron microscope (FE-SEM, JSM-6700 F) and transmission electron microscope (TEM, Tecnai G^2 20 instrument). The XRD pattern was conducted using Rigaku D/max 2400 X-ray diffractometer equipped with graphite monochromatized Cu K α radiation

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 $\textbf{Fig. 1.} \ \, \textbf{(a)} \ \, \textbf{XRD} \ \, \textbf{pattern, (b)} \ \, \textbf{a} \ \, \textbf{typical overview} \ \, \textbf{FESEM image, (c)} \ \, \textbf{an enlarged FESEM image and (d)} \ \, \textbf{TEM image of the flower-like (NH_4)}_2 \textbf{V}_3 \textbf{O}_8.$



 $\textbf{Fig. 2.} \ \ (a) \ \ \textbf{XRD pattern, (b) an overview FESEM image, (c) an enlarged FESEM image, (d) TEM image and (e) HRTEM image of the flower-like V_2O_5. \\$

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