



Facile synthesis of V_6O_{13} micro-flowers for Li-ion and Na-ion battery cathodes with good cycling performance



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ARTICLE INFO

Article history:

Received 26 January 2014

Accepted 13 March 2014

Available online 21 March 2014

Keywords:

Vanadium oxide

Lithium-ion battery

Sodium-ion battery

Hydrothermal

Oxalic acid

Nitrate

Ammonium metavanadate

ABSTRACT

A simple and versatile method for the preparation of V_6O_{13} microflowers is developed via a simple hydrothermal route with the aid of an alkali metal nitrate salt, which has important effects on the formation of V_6O_{13} . It is found that V_6O_{13} microflowers display good cycling stability as cathode materials for lithium-ion battery. In addition, they show high capacities for sodium-ion battery. We have V_6O_{13} microflowers discharge capacities up to $225.7 \text{ mA h g}^{-1}$ for sodium-ion battery. The reason may be the fewer phase transitions occurring upon lithium and sodium insertion for phase-unpure V_6O_{13} microflowers.

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1. Introduction

Nowadays, there is a remarkable demand for rechargeable batteries with reversible and efficient electrochemical energy storage and conversion in the field of portable electronic consumer devices, electric vehicles, and large-scale electricity storage in smart or intelligent grids [1]. The most fascinating and promising rechargeable batteries are both lithium-ion and sodium-ion batteries for the following reasons: Rechargeable lithium-ion batteries are of interest for large capacity, high power, and long cycling life, whereas sodium-ion battery has been brought into focus in large-scale electric energy storage applications for renewable energy and smart grid because of its huge abundant sodium resources and low cost [2].

Vanadium oxides offer advantages for being cheap, easy to synthesize, plenty in the earth and high-energy density, and thus they have attracted much interest in energy conversion and storage [3]. Amongst various vanadium oxides, V_6O_{13} has been considered as an excellent candidate for the cathode material owing to high electrochemical capacity and electronic conductivity [4]. As a result, V_6O_{13} has been extensively investigated as a cathode material for rechargeable lithium-ion battery since 1979 [5,6]. Unfortunately, it is difficult to achieve high capacities with good cyclability for V_6O_{13} cells because of the four phase transitions,

which decrease in electronic conductivity upon lithium insertion, and the loss of electrode integrity [4]. Though Munshi et al. reported that V_6O_{13} had showed high stability and good reversibility for the sodium-polymer electrolyte system with a limited number of cycles as sodium-ion battery cathode material, indicating that cell feasibility and good cycling efficiency [7]. Few people paid full attention to V_6O_{13} cathode material for sodium-ion battery. Recently, great interest has been attracted to develop various methods for the preparation of V_6O_{13} with an application to lithium-ion battery and electrochemical supercapacitor, for example, annealing the hydrated vanadium pentoxide aerogels under pure Ar [8], thermal decomposition of NH_4VO_3 [9] in a stream of high purity argon [10] or a flushing nitrogen atmosphere [11], a novel electrodeposition-thermal process [12], and a sol-hydrothermal approach with V_2O_5 and hydrogen peroxide (H_2O_2) as reagents [13].

Herein, we report the fabrication of V_6O_{13} micro-flowers involving the use of the simple hydrothermal method required low cost manufacturing and ultra-low power consumption. The synthesis was performed in a water solvent using ammonium metavanadate, oxalic acid and nitrate as reactants. V_6O_{13} micro-flowers were prepared with certain alkali metal nitrate ($LiNO_3$). When used as cathode materials for lithium-ion batteries, V_6O_{13} micro-flowers show high capacities and good cycling stability as well as sodium ammonium vanadium bronze [14]. It is also a promising cathode material for sodium-ion battery with higher discharge capacity than ammonium vanadium bronze [15].

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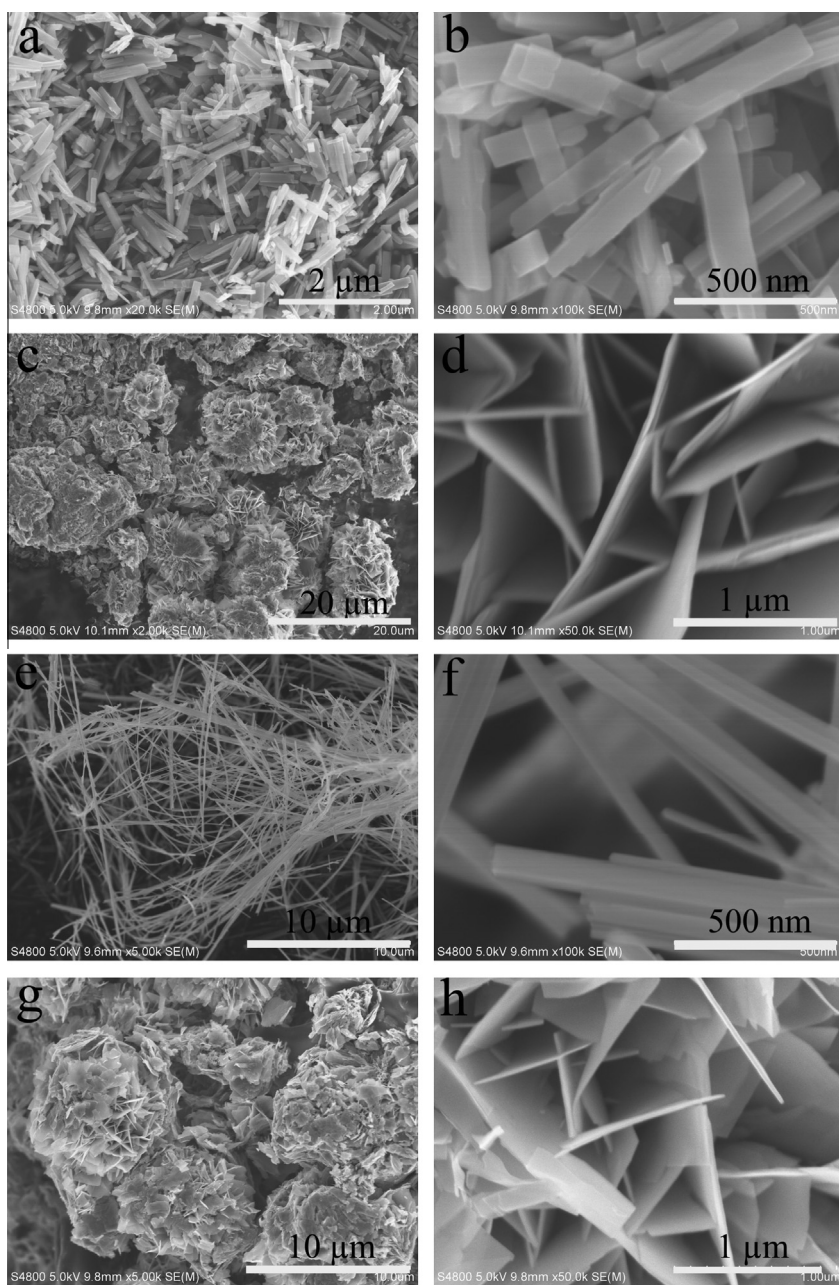


Fig. 1. SEM images of samples prepared without any nitrates (a, b) and with LiNO_3 (c, d), NaNO_3 (e, f) and KNO_3 (c, d), respectively.

2. Materials and methods

V_6O_{13} micro-flowers were prepared according to the following procedure. 1.44 g oxalic acid and 0.1 g LiNO_3 were dissolved in 30 ml deionized water, and then 0.8 g ammonium metavanadate (NH_4VO_3) was added into the solution under stirring at room temperature for 2 h. After that, the mixture was transferred into a 50-ml Teflon-lined stainless autoclave, sealed, kept at 200 °C for 24 h and cooled to room temperature. To investigate the effect of different alkali metal nitrate salts, equimolar NaNO_3 and KNO_3 to LiNO_3 were used to replace LiNO_3 , and two different samples were prepared under the identical condition. One sample was also prepared without any alkali metal nitrates under the identical circumstance.

The morphology of all the samples was observed by Hitachi S-4800 field emission scanning electron microscope (SEM). X-ray diffraction (XRD) patterns were recorded on a diffractometer

(Co K α , PANalytical, X'Pert). A Land CT2001A battery tester was used to measure the electrode activities at room temperature.

V_6O_{13} micro-flowers were tested as a cathode material for lithium-ion battery. The composite positive electrodes were consisted of the active material, conductive material (acetylene black) and binder (PVDF) in a weight ratio of 7/2/1. The Li metal was used as the counter electrode. The electrolyte was 1 M LiPF_6 in a 1/1/1 (volume ratio) mixture of ethylene carbonate (EC), propylene carbonate (PC) and dimethyl carbonate (DMC). The cells were charged and discharged between a 2.0 and 3.4 V voltage limit at various discharge currents.

V_6O_{13} micro-flowers were further investigated as a cathode material for sodium-ion battery. The composite positive electrodes were consisted of the active material, conductive material (acetylene black) and binder (PVDF) in a weight ratio of 7/2/1. The Na metal was used as the counter electrode. The electrolyte was 1 M

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