Self-assembled hairy ball-like $\text{V}_2\text{O}_5$ nanostructures for lithium ion batteries

Dong Fang $^a$, Licheng Li $^a$, Weilin Xu $^{a*}$, Guangzhong Li $^b$, Jie xu $^a$, Zhiping Luo $^c$, Caowei Liang $^a$, Yongsheng Ji $^a$ and Chuanxi Xiong $^{a*}$

$^a$ Key Lab of Green Processing and Functional Textiles of New Textile Materials, Ministry of Education, College of Material Science and Engineering, Wuhan Textile University, Wuhan, P. R. China, Fax: (86)02759367580; Tel: (86)02759367580

$^b$ State Key Laboratory of Porous Metal Material, Northwest Institute for Non-ferrous Metal Research, Xi’an, P. R. China

$^c$ Department of Chemistry and Physics and Southeastern North Carolina Regional Microanalytical and Imaging Consortium, Fayetteville State University, Fayetteville, USA.

EXPERIMENTAL SECTION

Materials Synthesis. In a typical synthesis, $\text{NH}_4\text{VO}_3$ (1.2 g) and $\text{H}_2\text{C}_2\text{O}_4\cdot\text{H}_2\text{O}$ in a molar ratio of 1:2 were dissolved in 40 mL of deionized water under vigorous stirring for several hours until a blue clear solution is formed. The obtained blue solution was then added into a 50 mL Teflon container, followed by addition of hexamethylenetetramine under stirring. Then the container was sealed in an autoclave and transferred to an electrical oven and kept at 150 °C for different durations of 30 min, 40 min, 1 h, 2 h, 3 h and 5 h. After cooling down naturally, the precipitates were collected by centrifugation and washed with pure ethanol for three times, then dried at 60 °C overnight. Hairy ball-like $\text{V}_2\text{O}_5$ nanostructures were obtained by further calcining the obtained precursor in air at 360 °C for 5 h with a heating rate of 1 °C min$^{-1}$. In order to further study the temperature effect on the morphologies of the products, the precursors were annealed at 400 or 500°C without changing other parameters. The as-synthesized precursor with heating at 360 °C, 400 or 500 °C were designated as $\text{V}_2\text{O}_5$-360 °C, $\text{V}_2\text{O}_5$-400 °C, and $\text{V}_2\text{O}_5$-500 °C, respectively.

Materials Characterization. X-ray diffraction analyses of the samples were performed using an X-ray diffractometer (XRD, D/Max2500, Rigaku) with Cu $K_\alpha$ radiation ($\lambda = 1.5418$ Å). The morphology of the samples was characterized by scanning electron microscopy (SEM, JEOL JSM-6300, JSM-6360LV) and transmission electron microscopy (TEM, JEOL-2010). For SEM...
sample preparation, a thin Au layer (3 nm) was evaporated to form a conducting film for observation. Thermal analysis of the precursor powders were conducted using simultaneous thermal analyzer (STA, NETZSCH, Germany). A heating rate of 10°C/min was adopted. The chemical composition of the samples was analyzed by X-ray photoelectron spectroscopy (XPS, KAlpha 1063, Thermo Fisher Scientific, UK). The Brunauer-Emmett-Teller (BET) specific surface area was performed by ASAP 2020 physisorption analyzer (Micromeritics Instrument Corporation).

**Electrochemical Measurements.** The working electrode slurry was prepared by dispersing V_2O_5, acetylene black and poly-(vinylidene fluoride) (PVDF) binder in an N-methylpyrrolidone solution at a weight ratio of 80: 10: 10. The slurry was spread on aluminum foil disks and dried in a vacuum oven at 120 °C prior to coin-like cells assembly. Lithium foil was used as the counter and reference electrode, and 1.0 M LiPF_6 in ethyl carbonate/dimethyl carbonate (1:1 v/v ratio) was used as the electrolyte. Cyclic voltammetry measurements were performed on an AUTOLAB electrochemical workstation (PG302N). Galvanostatic charging/discharging was conducted on a battery tester (Land CT2001).

![Figure S1](attachment:image.png) Fig. S1 XRD pattern (a), XPS survey spectrum (b) and high-resolution V2p3/2 XPS spectra (c) of the as-prepared sample.
Fig. S2. SEM images of four hydrothermally prepared samples: (a) 30 min, (b) 1 h, (c) 3 h and (d) 5 h. Schematic illustration of the formation process of NH$_4$V$_4$O$_{10}$ microspheres from the side view (e).

Fig. S3 (a) TG-DSC curves of the NH$_4$V$_4$O$_{10}$ powders in air atmosphere with a heating rate of 10°C/min; (b) XRD pattern of hairy ball-like V$_2$O$_5$ spheres after annealing at 360 °C.
Fig. S4 low-magnification SEM images of hairy ball-like V$_2$O$_5$ spheres after annealing at different temperatures: 360 °C (a, b); 400 °C (c, d); and 500 °C (e, f).
Fig. S5 XRD patterns of V$_2$O$_5$ spheres after annealing at 400 and 500 °C.

Fig. S6 During the cycles, the coulombic efficiency of V$_2$O$_5$ spheres after annealing at different temperatures.