Electronic supplementary information (ESI)

Self-assembled hairy ball-like V₂O₅ 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, NH_4VO_3 (1.2 g) and $H_2C_2O_4 \cdot 2H_2O$ 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 V₂O₅ 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, respectively.

Materials Characterization. X-ray diffraction analyses of the samples were performed using an X-ray diffractometer (XRD, D/MAX2500, Rigaka) with Cu K_{α} radiation (λ = 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 LiPF₆ 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).



Fig. S1 XRD pattern (a), XPS survey spectrum (b) and high-resolution V2p^{3/2} 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 NH₄V₄O₁₀ microspheres from the side view (e).



Fig. S3 (a) TG-DSC curves of the $NH_4V_4O_{10}$ powders in air atmosphere with a heating rate of 10°C/min; (b) XRD pattern of hairy ball-like V₂O₅ spheres after annealing at 360 °C.



Fig. S4 low-magnification SEM images of hairy ball-like V_2O_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_2O_5 spheres after annealing at 400 and 500 $^\circ\text{C}.$



Fig. S6 During the cycles, the coulombic efficiency of V_2O_5 spheres after annealing at different temperatures.