

Electronic supplementary information (ESI)

Fabrication of Nanostructured V₂O₅ via Urea Combustion for High-performance Li-ion Battery Cathode

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EXPERIMENTAL METHODS

Materials Synthesis.

10.0 g urea (99.0% purity) was heated at 140 °C until it completely melted away. Then commercial V₂O₅ (99.0% purity) with different mass ratios of V₂O₅/urea (1: 3/4/5) were added into melted urea under active stirring for 30 min at 140 °C. The color of the molten mixture gradually changed from yellow to mazarine, which indicated that the V₂O₅ have been reduced by molten urea and the black precursor was formed as it cooled. And then the black precursor was deposited into a 50 ml crucible. The crucible was put into the preheated furnace in air at 450°C for 1h. Yellow foam products (Figure S1b) in the crucible were obtained as the urea burned completely. And the yellow foam products were isothermally heated at 630 °C for different times from 5 to 30 min. The crucibles with yellow foam products were putted into the muffle preheated furnace and pulled out quickly after the desired time. The yellow foam products can be broken into smaller particles and individual nanosized particles by grind simply. If required, a fraction with required sizes can be separated by sieving. Rapid foaming and combustion of the precursor in muffle furnace took place to form bread like structure. And its weak agglomerates structure can be broken to smaller particles and individual nanosized particles by grind simply.

Characterization

FT-IR spectra were recorded on a Bruker IFS 66V interferometer. Thermogravimetric analysis (TGA) was performed on a Perkin-Elmer TGA 7 unit with a heating rate of 10 °C·min⁻¹. Crystalline phase identification was characterized by powder X-ray diffraction (XRD) on a Rigaku D/max 2400 X-ray equipped with graphite monochromatized Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$) with samples mounted using a grooved glass sample holder. A scan rate of 0.02° s⁻¹ was applied to record the pattern in the 2 θ range from 10° to 70°. The shapes and structures of as-prepared samples were observed with scanning electron microscope (SEM) and transmission electron microscopy (TEM) at Electron Microscope Lab, Dalian University of Technology. The SEM (HITACHI, S-4800) and TEM (Tecnai G20 instrument) were operated at an accelerating voltage of 5 kV and 200 kV, respectively

Electrochemical measurements

Vanadium oxides electrodes were fabricated by coating an NMP based slurry with a mixture of 80.0 wt% of active materials, 10 wt% of polyvinylidene fluoride (PVDF) binder and 10 wt% of carbon black on an aluminum current collector. After dried at 120 °C in vacuum oven for 12 h, then it was cut into a 14 mm diameter discs as cathode. A solution of LiPF₆ in EC/DMC/DEC (1: 1: 1 in weight) (Guotaihuarong LTD., Zhangjiagang, China) was used as electrolyte. A CR2032 coin-type cell was assembled with lithium metal as the counter and reference electrode and polypropylene film as a separator. The cells were constructed and handled in an argon-filled glovebox. The charge-discharge measurements were carried out using the Land battery system (CT2001A) at a constant current density in a voltage range of 2.0-4.0 V versus Li/Li⁺.

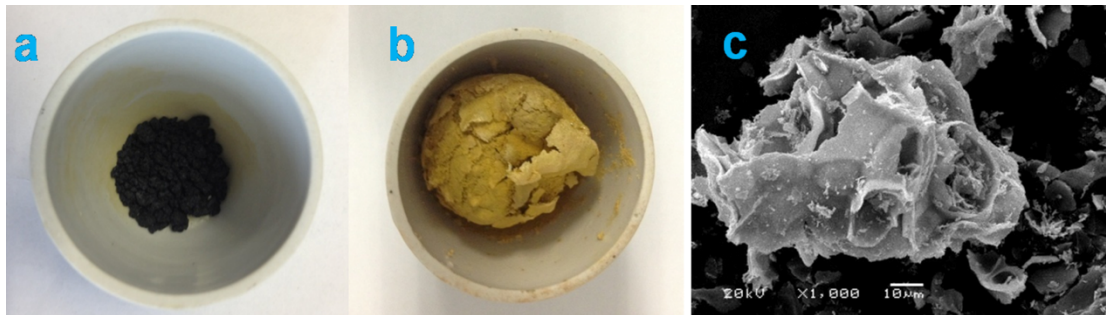


Figure S1 (a) photograph of the black precursor with mass ratio V_2O_5 /urea 1 : 4; (b) photograph of the sample synthesized by thermal decomposition of the black precursor (the starting mass ratio of V_2O_5 to urea is 1 : 4) at 450 °C for 1h; (c) SEM image of the sample at low magnification

The photograph of the precursor via the reaction of V_2O_5 and molten urea is shown in Figure S1a. The color of the melt mass gradually changed from yellow to black, which indicated that the V_2O_5 have been reacted with molten urea.

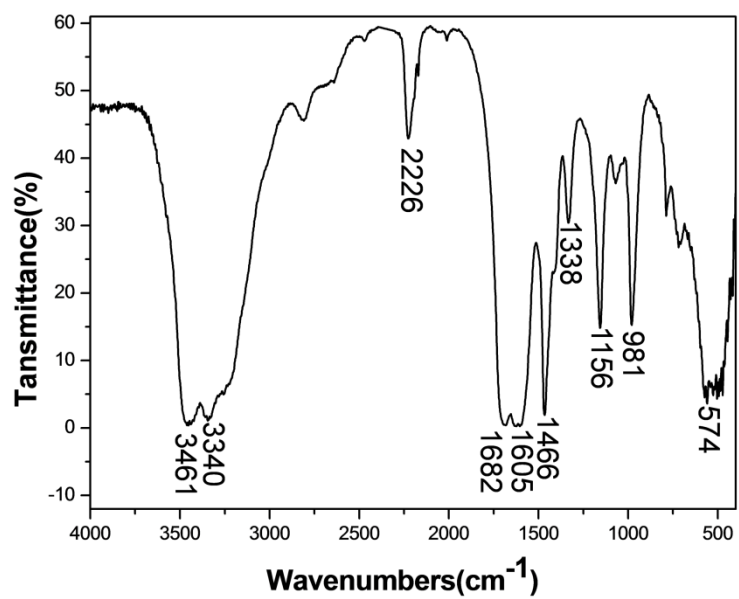


Figure S2 Fourier transform infrared spectrum of the black precursor (the starting mass ratio of V₂O₅ to urea is 1 : 4)

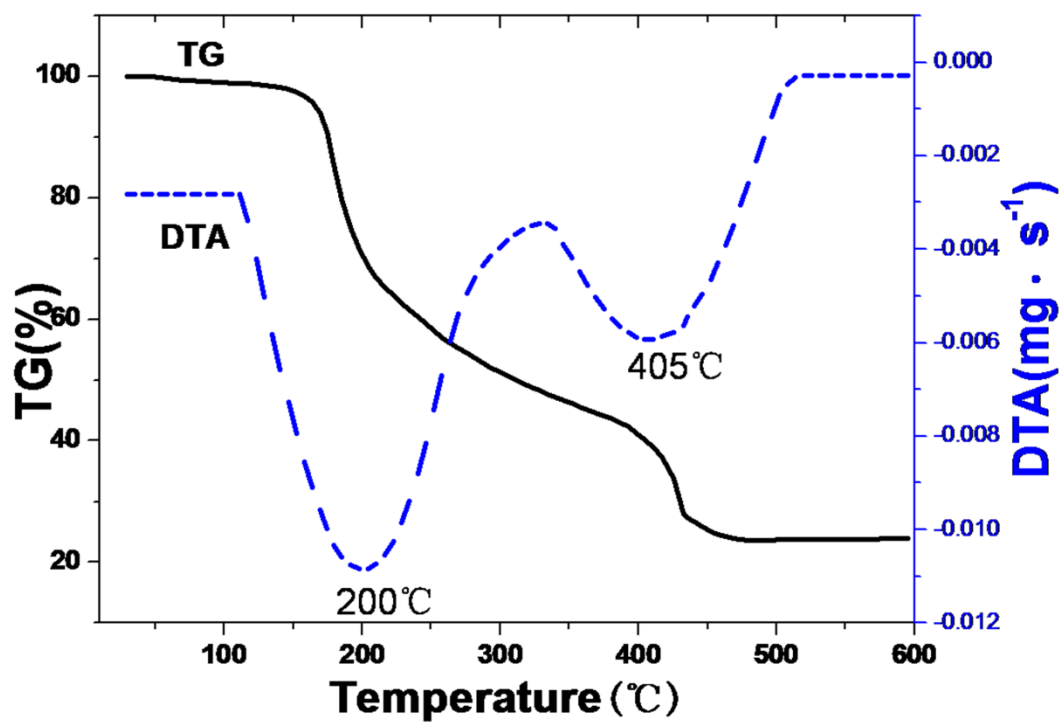


Figure S3 Thermal gravimetric (TG) analysis pattern of the black precursor (the starting mass ratio of V_2O_5 to urea is 1 : 4) the heating rate was $10\text{ }^\circ\text{C min}^{-1}$

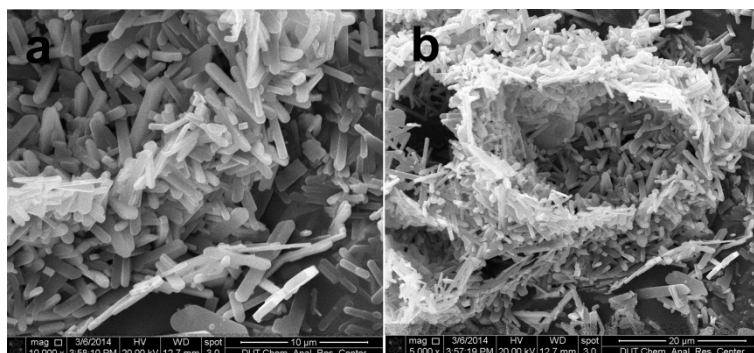


Figure S4 SEM images at low magnification of the V₂O₅ nanorods after annealing in air at 630 °C for 30 min

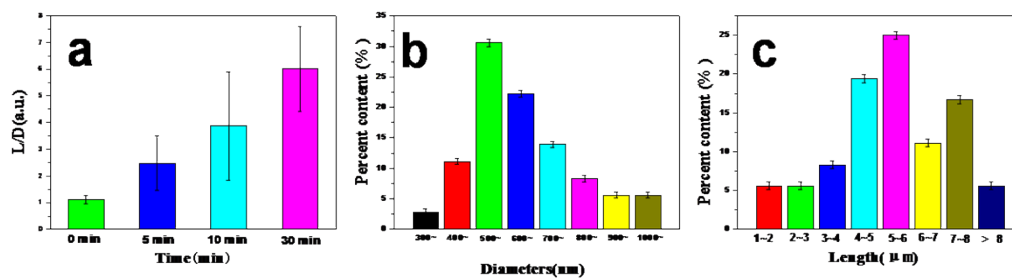


Figure S5 (a) Histogram of the L/D ratio for the nanocrusts V_2O_5 after subsequent annealing in air at 630 °C for 0, 5, 10 and 30 min Histogram of the diameter (b) and length (c) distribution of the V_2O_5 nanorods after annealing in air at 630 °C for 30 min.

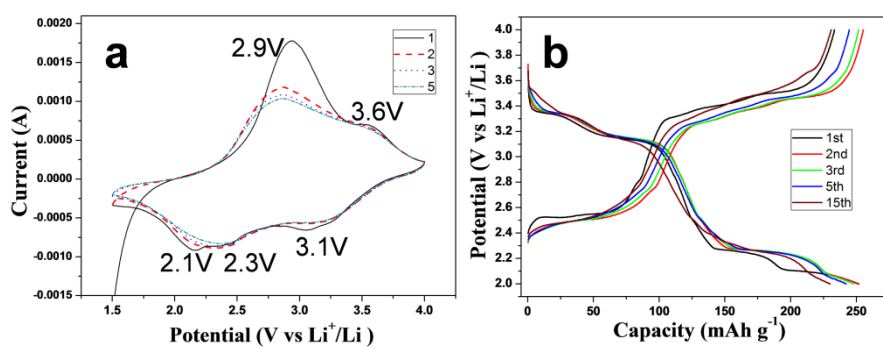


Figure S6 (a) CV curves of the V_2O_5 nanocrusts for the initial five cycles scanned at $1\ mV\ s^{-1}$ in the voltage range of 1.5 – 4.0V; (b) Charge and discharge voltage profiles of the V_2O_5 nanocrusts for the first, second, 3rd, 5th and 15th cycles at a current density of $300\ mA\ g^{-1}$.

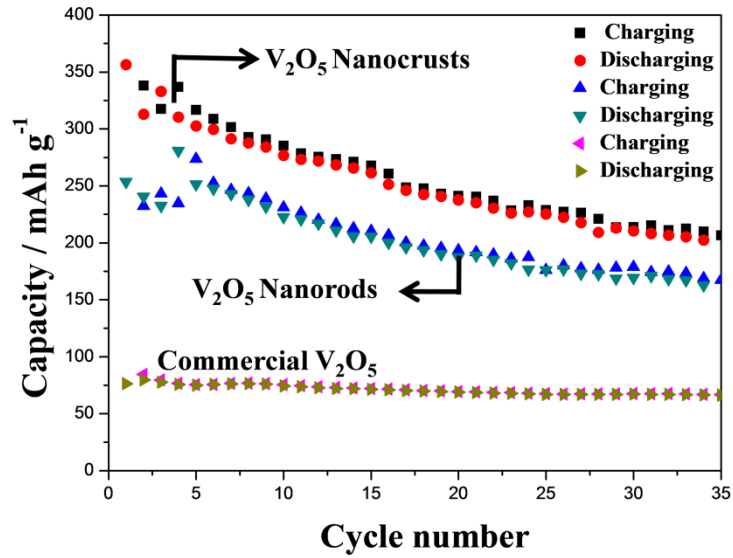


Figure S7 Cycling performances of Commercial V_2O_5 , the V_2O_5 nanocrusts synthesized via thermal decomposition of black precursor (the starting mass ratio of V_2O_5 to urea is 1:4) at 450 °C for 1h and the V_2O_5 nanorods obtained by the V_2O_5 nanocrusts after subsequent annealing in air at 630 °C for a hour in the voltage range of 1.5–4.0 V at a rate of 30 mA g^{-1}