

Supporting Information for

Superior Cathode of Sodium-Ion Batteries:

Orthorhombic V₂O₅ Nanoparticles Generated

in Nanoporous Carbon by Ambient

Hydrolysis Deposition

*Vadivukarasi Raju,[†] Jordan Rains,[†] Cooper Gates,[†] Wei Luo,[†] Xingfeng Wang,[†]
William F. Stickle,[‡] Galen D. Stucky[§] and Xiulei Ji^{*†}*

[†]Department of Chemistry, Oregon State University, Corvallis, Oregon, 97331-4003,
USA

[§]Department of Chemistry and Biochemistry, University of California, Santa Barbara,
California, 93117-9510, USA

[‡]Hewlett-Packard Co., 1000 NE Circle Blvd., Corvallis, Oregon, 97330, USA

*E-Mail: David.Ji@oregonstate.edu

Experimental

Porous carbon (RFC) was prepared according to the procedure reported previously in the literature using resorcinol and formaldehyde as precursors.¹

AHD method: the nanoporous carbon was coated with V₂O₅ using our previously reported AHD method.^{2,3}

Step I: Water loading. Degassed RF carbon, 50 mg, was allowed to equilibrate in an ambient water vapor / air mixture with 100% relative pressure at 80 °C for one hour. Later, the sample was poured in to a Petridish and kept at stagnant atmosphere for one more hour.

Step II: Hydrolysis deposition of V₂O₅. Water-loaded RF carbon was dispersed and soaked for one hour in a dilute solution of vanadium triisopropoxide solvated in cyclohexane (10 vol%). The solid product was collected by filtration in a glovebox. Samples were heated at 225 °C under air for 2 hrs.

Characterization methods. X-Ray diffraction (XRD) patterns were collected using a Rigaku Ultima IV Diffractometer with Cu K α irradiation ($\lambda = 1.5406 \text{ \AA}$). The morphology was examined by field emission scanning electron microscopy (FESEM) using an FEI NOVA 230 high resolution SEM with an energy-dispersive X-ray (EDX) attachment. Transmission electron microscopy (TEM) images were recorded by FEI Titan 80-200 TEM. High-angle annular dark field scanning TEM (HAADF-STEM) measurements were carried out on an FEI Titan 80-200 microscope coupled with a HAADF detector and an EDX spectrometer. Nitrogen sorption measurements were performed on Micromeritics

TriStar II 3020 analyzer. Thermogravimetric analysis (TGA) was performed using Shimadzu TGA-50 instrument under air from room temperature to 800 °C with a ramping rate of 10 °C min⁻¹.

Electrochemical measurements. Electrode slurries were prepared by mixing 70 wt% active material (V₂O₅/C composite), 20 wt% carbon black (Super-P) and 10 wt% polyvinylidene fluoride (PVDF). The mixture was suspended in N-methyl-2-pyrrolidinone (NMP) before casting onto an Al foil current collector by the doctor blade method. The typical active-mass loading is around 1-1.3 mg/cm². Sodium foil anodes were polished in an Ar environment before using as the counter/reference electrode. The electrodes were assembled in a coin cell in glovebox. Glass-fiber membrane was used as the separator, and 1.0 M NaClO₄ in propylene carbonate (PC) as the electrolyte. Galvanostatic cycling was conducted on an Arbin BT2000 system, and cyclic voltammograms (CVs) were collected on a VMP-3 multi-channel workstation at a scanning rate of 0.5 mVs⁻¹ at room temperature.

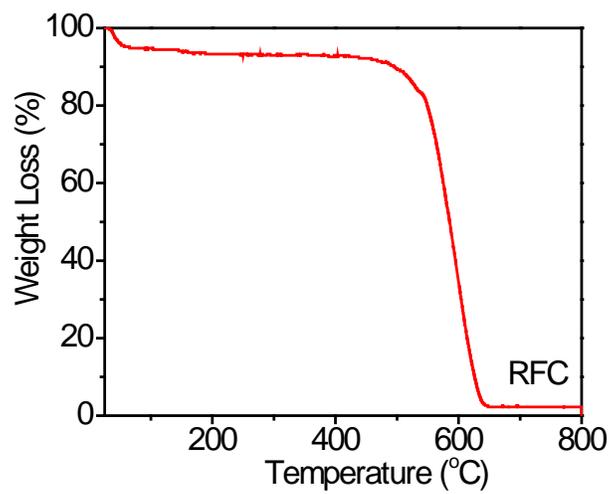


Figure S1. TGA profile for RFC in air.

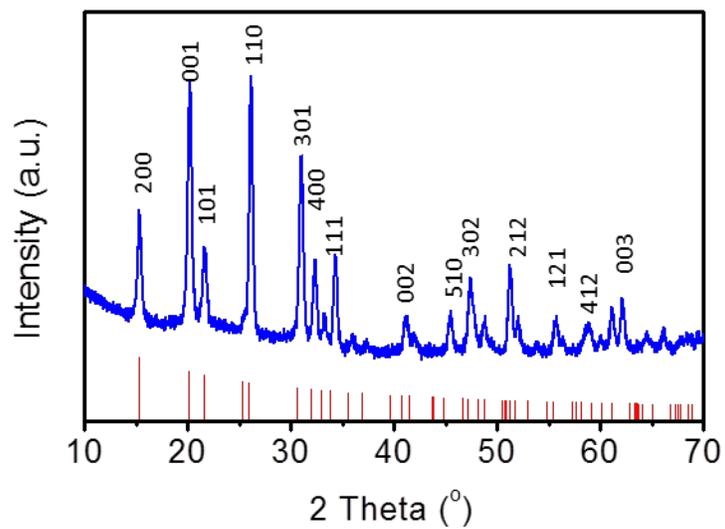


Figure S2. Wide angle XRD pattern of bulk V₂O₅.

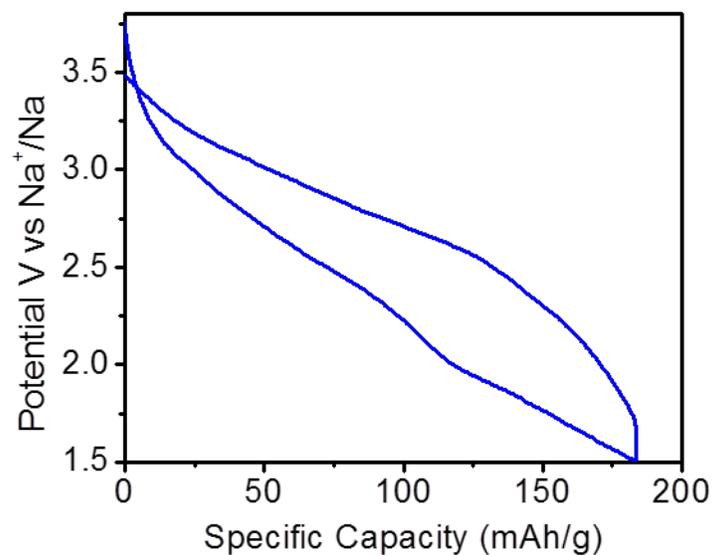


Figure S3. Galvanostatic charge-discharge profiles for 55-V₂O₅-RFC in the third cycle at a current density of 40 mA/g in a potential window between 1.5 V to 3.8 V.

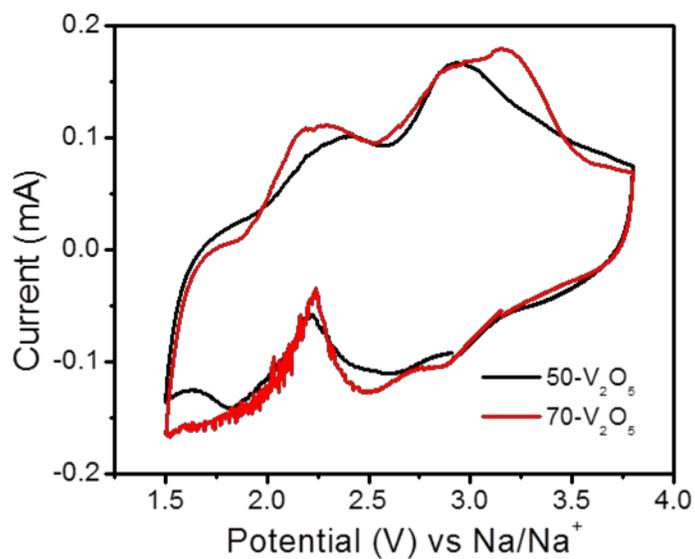


Figure S4. Comparison of CV curves for 55-V₂O₅-RFC and 70-V₂O₅-RFC at a sweeping rate of 0.5 mV/s between 1.5 V to 3.8 V.

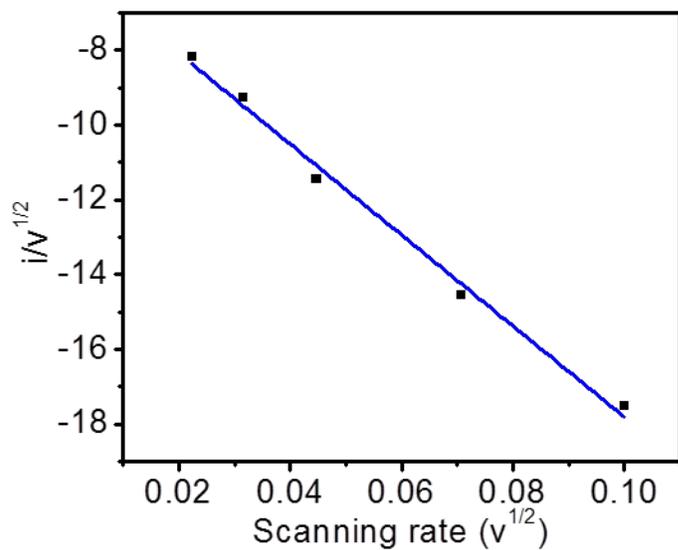


Figure S5. Linear behavior of $i/v^{1/2}$ as a function of $v^{1/2}$. This is used to determine the slope k_1 and the intercept k_2 of the function of

$$i(V) = k_1 v + k_2 v^{1/2}$$

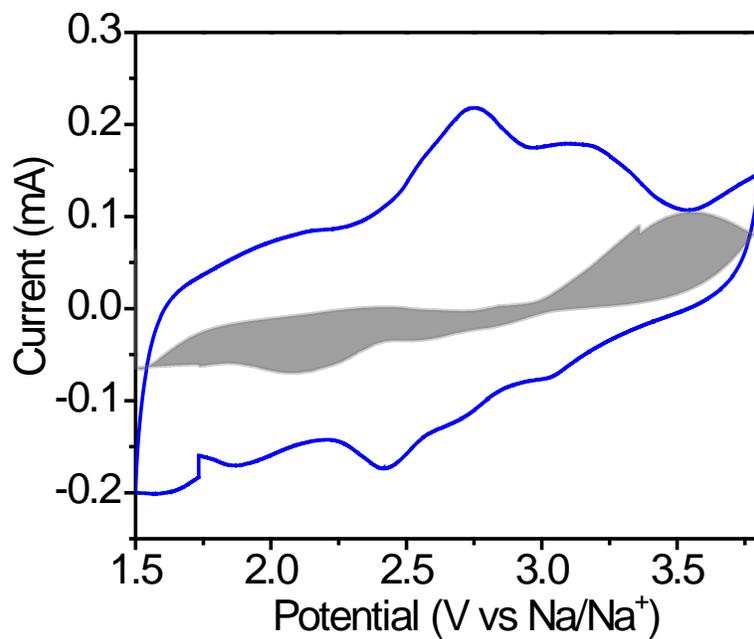


Figure S6. Cyclic voltammetry curves for 55-V₂O₅-RFC at a sweeping rate of 0.5 mV/s.

The capacitive contribution to the total current is shown in the shaded region.

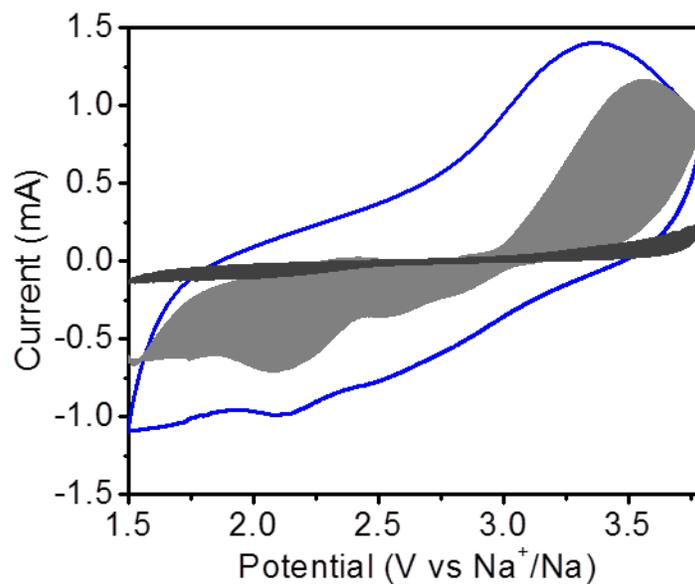


Figure S7. Comparison of CV curves of 55-V₂O₅-RFC with 1.0 M TEABF₄ in PC and 1.0 M NaClO₄ in PC at 5 mV/s. The light grey shaded area shows the total capacitive contribution to the total current and the dark grey shaded area shows the contribution from the double-layer capacitance.

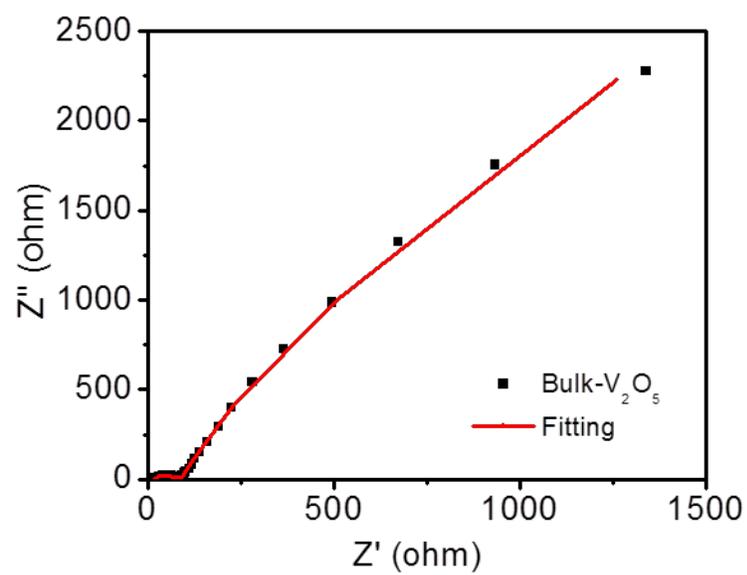


Figure S8. Nyquist plot and fitting data for bulk V_2O_5 .

Table S1. Physical characteristics of the samples

Sample	BET surface area (m ² /g)	Pore volume (cm ³ /g)	Pore diameter (nm)
RFC	1081	5.3	39
55-V ₂ O ₅ -RFC	641	2.9	36
70-V ₂ O ₅ -RFC	463	2.6	35
V ₂ O ₅	40	-	-

Table S2. Electrode resistance obtained from the equivalent circuit fitting of EIS results

Sample	R _s	R _f	R _{ct}
55-V ₂ O ₅ -RFC	7.7	1.5	51
Bulk V ₂ O ₅	12.3	24.7	66

References

-
- [1] Han, S.; Hyeon, T. *Chem. Commun.* **1999**, 1955-1956.
- [2] Wang, X.; Raju, V.; Luo, W.; Wang, B.; Stickle, W. F.; Ji, X. *J. Mater. Chem. A* **2014**, 2, 2901-2905.
- [3] Raju, V.; Wang, X.; Luo, W.; Ji, X. *Chem. Eur. J.* **2014**, 20, 1-7.