Electronic Supplementary Information

for

Controllable synthesis of cypress-derived hard carbon for high-rate sodium ion storage

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Experimental Section

Materials. Cypress wood was purchased from Mulinsen Timber Co., Ltd (Guangxi, China). Sodium hydroxide (NaOH), sodium sulfite (Na_2SO_3) and hydrogen peroxide (30% H_2O_2) were purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China).

Synthesis of the NOCNRs. 10 g sample of clean cypress wood was finely pulverized, subsequently immersed in water for softening and eliminating soluble contaminants. Following this pretreatment, the powdered cypress was soaked in a solution meticulously blended with 2.5 M NaOH (25 mL) and 0.4 M Na_2SO_3 (50 mL) for 12 h. Subsequent to the soaking process, the sample was bleached in 30% H_2O_2 (30 mL) for 24 h and then washed several times with deionized water until pH 7, ensuring the complete removal of bleaching agents. The rinsed sample was then dried in an oven for 12 h to remove moisture. The dried material was then subjected to carbonization in a tube furnace, operated under an inert argon (Ar) atmosphere with a controlled gas flow rate of 80 sccm. Carbonization was performed in a tube furnace under an argon atmosphere (Ar) with a gas flow rate of 80 sccm. The dried sample was cooked at 900, 1100, and 1300 °C, respectively, for 3h at each temperature. Post carbonization, the samples were immersed and agitated in 1 mol/L HCl solutions for 3 h, followed by filtration and repeated washing with distilled water until neutral pH. The final step involved transferring the thoroughly cleaned samples to a vacuum oven and dried at 60 °C for 12 h to ensure complete removal of any residual moisture. The end products prepared following this rigorous synthesis protocol are referred to as WC-900, WC-1100, and WC-1300 in the paper.

Characterizations. Morphological analyses were conducted using a Field Emission Scanning Electron Microscope (FE-SEM, JEOL JSM-7800F), Transmission Electron Microscope (TEM, FEI Talos F200S), and High-Resolution Transmission Electron Microscope (HRTEM, equipped with super-X technology). These advanced instrumentations enabled a detailed examination of the surface morphology and internal structures of the samples. Nitrogen adsorption-desorption measurements were performed at 77 K, employing a state-of-the-art instrument from Micromeritics (ASAP-2460-BET), to accurately determine the porosity and surface areas of the samples. The phase composition and purity of the samples were verified through rigorous analysis using a powder X-ray diffractometer (XRD, Shimadzu XRD-6100 Lab) equipped with Cu Kα radiation (λ=1.5418 Å) and operated at a scan rate of 7° min^{-1}. This ensured a precise identification of the crystalline phases present in the samples. The Raman spectra of the samples were recorded with high accuracy on a Zolix RTS2 Confocal Laser Raman microscope, utilizing an excitation wavelength of 532 nm. This spectroscopic technique provided valuable insights into the vibrational modes and molecular structure of the samples. Furthermore, Fourier Transform Infrared Spectroscopy (FTIR, Thermo Scientific Nicolet IN10) was employed to characterize the chemical structure of the product in depth. This method enabled the identification of functional groups and chemical bonds present within the samples, contributing to a comprehensive understanding of their chemical composition.

Fabrication of Working Electrodes and Na metal half-cells. The fabrication initiates with mixing active material (70 wt.%), superconducting carbon black (SCCB, 15 wt.%), and polyvinylidene fluoride (PVDF, 15 wt.%) binder in an 8:1:1 mass ratio. N-methylpyridine (NMP) was used as a solvent to create a homogeneous slurry. Thorough blending and fine grinding ensured uniform dispersion and good interfacial contact, resulting in a smooth, consistent slurry ready for coating. Subsequently, the resultant slurry was uniformly coated onto the surface of pre-cut carbon-coated copper foil. The coating procedure was executed with precision to maintain a consistent layer thickness, ensuring even distribution of the electrode material. Upon completion of the coating step, the copper foil was placed into a vacuum oven and subjected to a thermal treatment at 80 °C for 12 h. This step is crucial for ensuring thorough drying of the electrode material and structural consolidation, thereby enhancing its mechanical integrity and electrochemical stability. Finally, the dried electrode material was precisely trimmed to a dimension of 1×1 cm². This meticulous process ensured rigorous control over the composition ratio and coating uniformity of the electrode material, ultimately guaranteeing the consistency and reproducibility of the electrode sheets.

CR2032-type coin cells were assembled in an Ar-filled glove box (H_2O and O_2 contents < 0.1 ppm). The electrochemical performance was tested utilizing CR2032 type coin cells with a Na metal chip as counter and reference electrodes and 1 mol L^{-1} $NaPF_6$ in diglyme as an electrolyte.

Electrochemical measurements. Galvanostatic intermittent titration technique (GITT) and galvanostatic charge–discharge (GCD) tests were evaluated using a LAND battery tester (CT2001A) at a controlled temperature of 25 °C. GITT was performed using current pulses (100 mA g^{-1}) for 10 min and a relaxation process over 1.0 h. GCD tests were conducted to assess the cycling stability and capacity retention of the electrodes. Cyclic voltammetry (CV) was recorded on the Gamry electrochemical workstation (Interface 1000E), providing insights into the redox reactions occurring at the electrode surfaces. Electrochemical impedance spectroscopy (EIS) was conducted over a frequency range of 100 kHz to 0.01 Hz, enabling the characterization of charge transfer resistances and ion diffusion processes within the electrolyte-electrode interfaces.

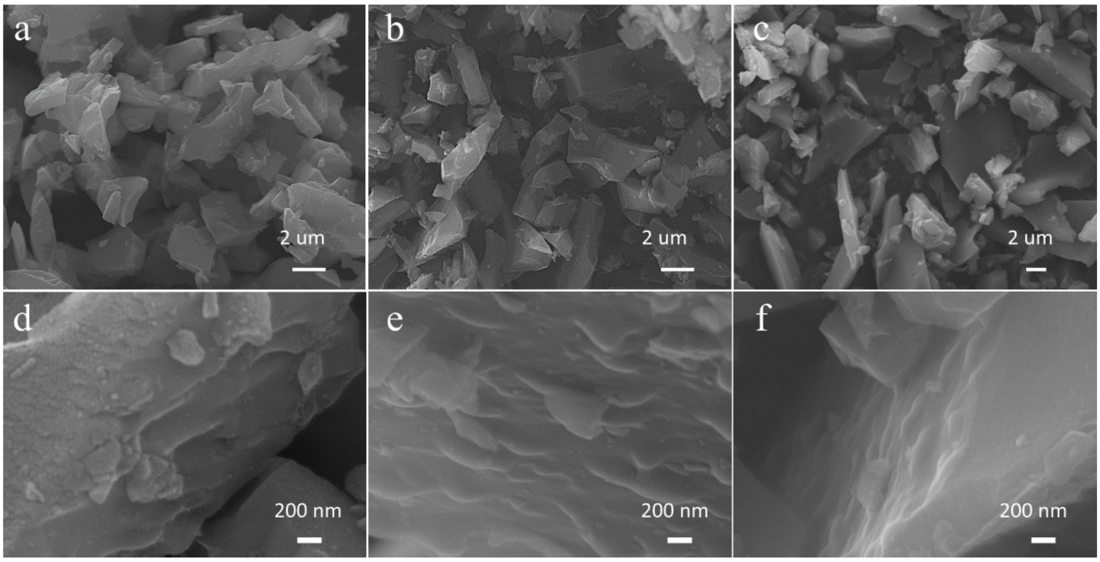

Fig. S1 SEM images of (a, d) WC-900, (b, e) WC-1100, and (c, f) WC-1300 samples.

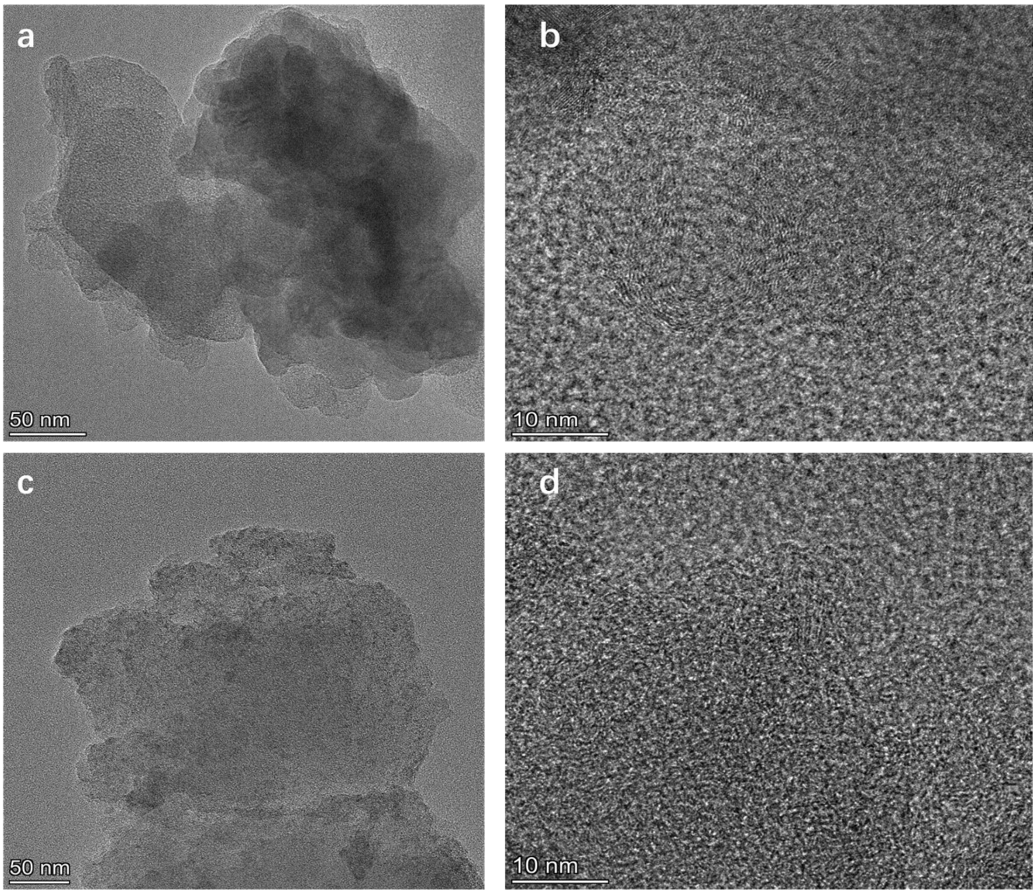

Fig. S2 (a) TEM and (b) HRTEM images of the WC-900. (c) TEM and (d) HRTEM images of the WC-1300.

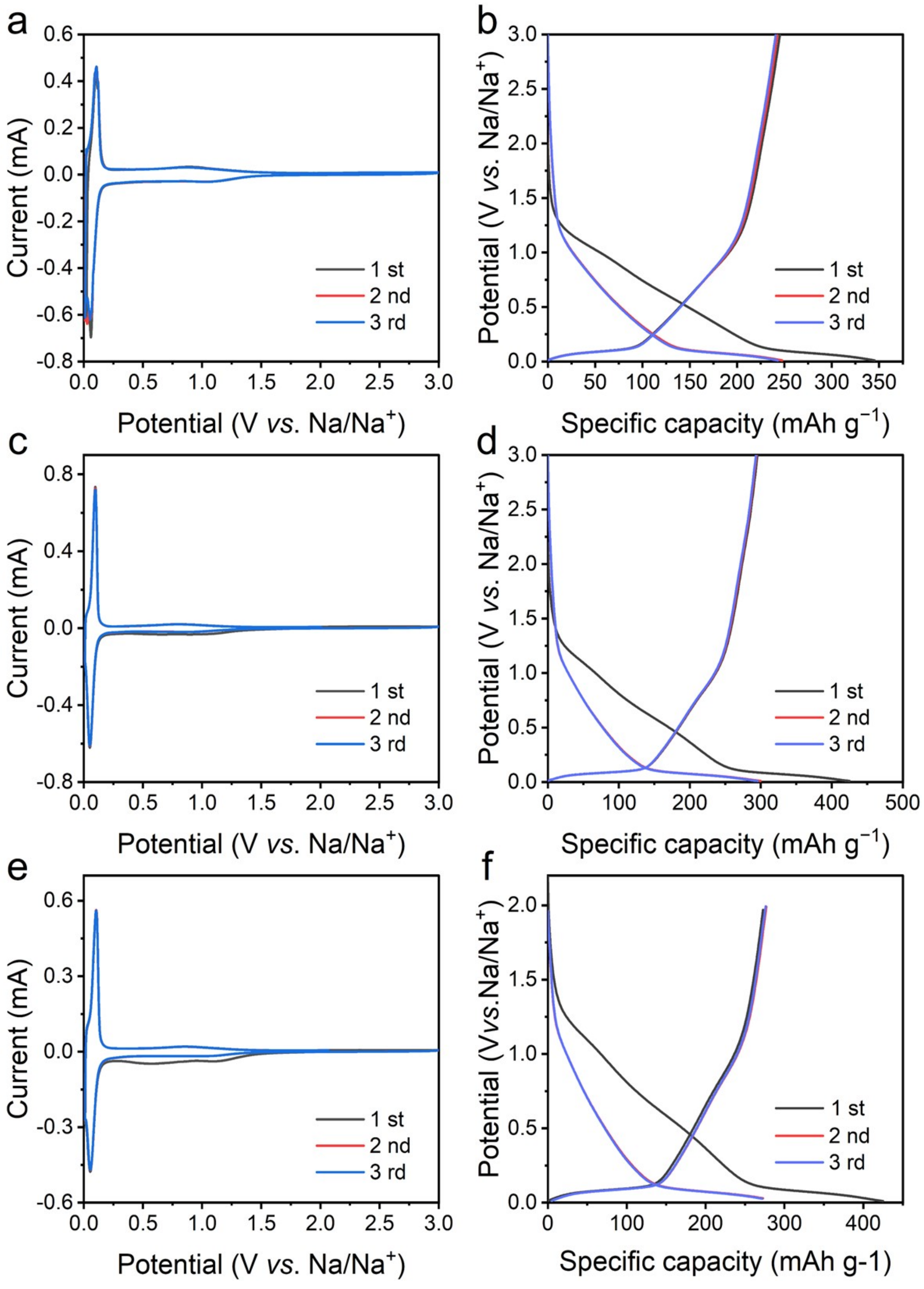

Fig. S3 Initial three CV curves, at scan rate of 0.1 mV s^{-1} and GCD curves at current density 0.1 A g^{-1} for the (a, b) WC-900, (c, d) WC-1100, and (e, f) WC-1300 electrodes.

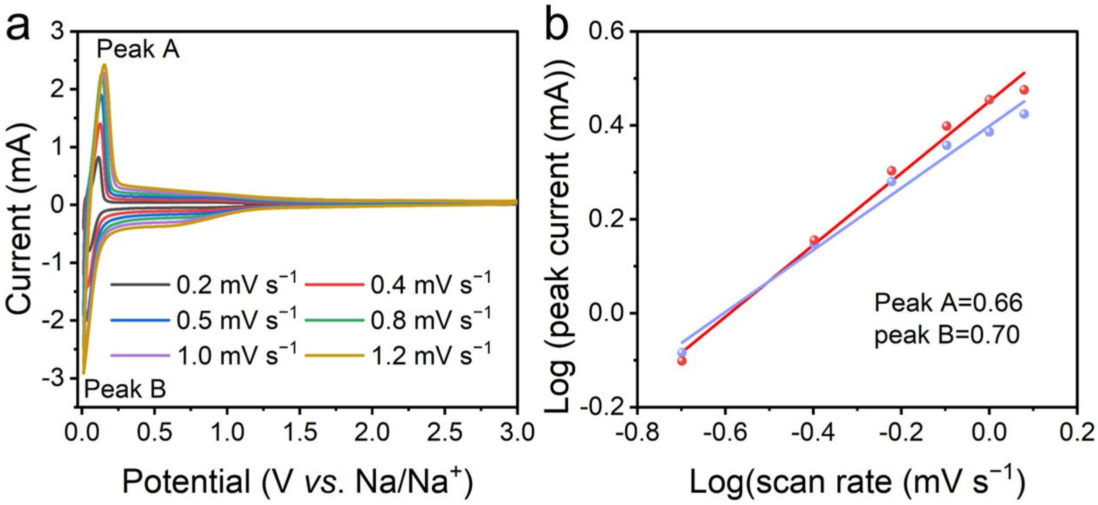

Fig. S4 (a) CV curves at the scan rates of 0.1–1.2 mV s^{-1} and (b) *b*-value of the WC-1100 electrodes.

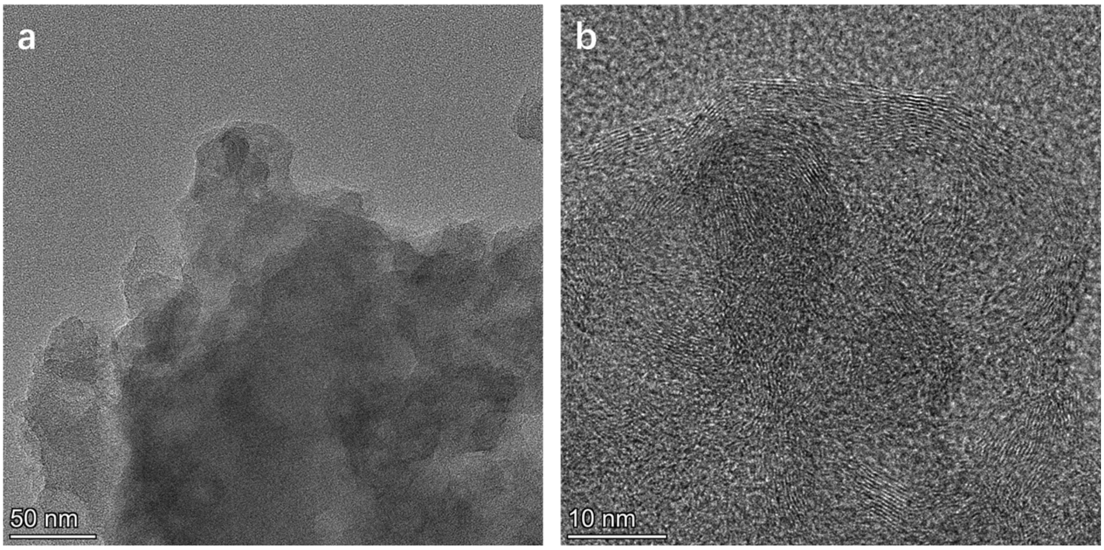

Fig. S5 (a) TEM and (b) HRTEM images of the WC-1300 after cycling.