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Chemical Oxidation Synergistically Regulates Surface Chemistry and Pore Structure of Cotton Stalk-Based Hard Carbon for Enhanced Sodium Storage Performance

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Abstract

Biomass-derived hard carbon (HC) represents a promising anode candidate for sodium-ion batteries, owing to its disordered structure and abundant micropores. This study systematically investigates three chemical oxidation strategies (NaClO, H₂SO₄ and H₂O₂+NaOH) applied to cotton stalk-derived HC carbonized at 1300 °C. The NaClO-treated sample delivers the optimal overall electrochemical performance, achieving a high discharge capacity of 330.2 mAh g⁻¹ at 0.1 C, a high initial Coulombic efficiency (ICE) of 81.7%, and a capacity retention of 81.9% after 1000 cycles at 2 C (from 226.0 to 185.1 mAh g⁻¹). This superiority is attributed to the formation of a three-dimensional hierarchical pore network and optimal oxygen functional groups. The H₂SO₄ treatment yields a discharge capacity of 323.2 mAh g⁻¹, an ICE of 75.3%, and a capacity retention of 77.1% after 1000 cycles (from 183.5 to 141.5 mAh g⁻¹), benefiting from structural densification. The H₂O₂+NaOH treatment delivers a capacity of 269.8 mAh g⁻¹ and an ICE of 74.2%, exhibiting a distinct activation behavior likely due to its thin pore walls and abundant open mesopores. Overall, all treated samples significantly outperform the pristine HC, which exhibits a discharge capacity of 320.3 mAh g⁻¹, an ICE of 68.0%, and a retained capacity of 90.4 mAh g⁻¹ after 1000 cycles at 2 C.

Keywords: sodium-ion batteries; hard carbon; cotton stalk; chemical oxidation; surface chemistry; pore structure

1. Introduction

Sodium-ion batteries (SIBs) represent one of the most promising next-generation technologies for large-scale energy storage, owing to the abundance of sodium and a mechanism analogous to that of Li-ion batteries [1–3]. Hard carbon (HC), characterized by its disordered layers, abundant micropores, and large interlayer spacing (0.37–0.42 nm), enables reversible Na⁺ intercalation/deintercalation, positioning it as one of the most practical anode materials for SIBs [4–8]. Biomass waste has emerged as a research focus for HC due to its renewability, hierarchical porosity, and low environmental footprint [9–12]. Cotton stalks, a massive agricultural by-product, have been successfully utilized as SIB anodes with considerable potential. However, previous studies have focused mainly on carbonization temperature and precursor design, while systematic chemical post-treatments for surface and pore engineering need further exploration [13–20]. In addition, residual impurities, oxygen-containing functional groups, and insufficient graphitization often result in low initial Coulombic efficiency (ICE, <80%) and poor cycling stability [21]. Therefore,



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rational chemical modification strategies are urgently required to enhance the sodium storage performance.

Common modification strategies for HC encompass heteroatom doping (e.g., N, P and S), surface coating, high-temperature chlorination, acid washing, and chemical oxidation [22]. Chemical oxidation enables the straightforward and controllable regulation of surface functional groups, selective etching of amorphous carbon to optimize pores, and the introduction of defects that enhance Na^+ adsorption and pseudocapacitance [23–26]. Representative oxidants include NaClO , H_2SO_4 , H_2O_2 , NaOH , HNO_3 , KMnO_4 , and persulfates [27–34]. Among them, H_2SO_4 introduces sulfur-containing groups via sulfonation and exfoliates disordered carbon layers, and has been employed for sucrose dehydration and biomass pretreatment. The H_2O_2 + NaOH combination exhibits synergistic effects. NaOH alkaline etching removes unstable carbon components and thins pore walls, while H_2O_2 generates hydroxyl/peroxy radicals that mildly oxidize carbon edges, thereby increasing open porosity and the mesopore proportion. In contrast, NaClO acts as a mild oxidant that effectively exfoliates surface-graphitized fragments and introduces oxygen functional groups onto carbon fibers and activated carbon. Given that carbon materials differ substantially in graphitization degree, pore structure, and impurity content, the same oxidation treatment can affect different systems in markedly different ways. Therefore, a systematic investigation of these treatments on cotton stalk-derived HC is highly desirable.

Herein, cotton stalk-derived precursors were pretreated with NaClO , H_2SO_4 and H_2O_2 + NaOH as oxidants at 60°C for 5 h and subsequently annealed at 1300°C for 2 h, yielding HC_{NaClO} , $\text{HC}_{\text{H}_2\text{SO}_4}$ and $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ alongside untreated HC (Figure 1). The three oxidation strategies induce markedly different structural modifications. NaClO treatment generates a three-dimensional hierarchical pore network with expanded interlayer spacing, high defect density, and abundant closed pores. This architecture delivers the best overall performance, with a discharge capacity of 330.2 mAh g^{-1} at 0.1 C (assume $1\text{ C} = 350\text{ mA g}^{-1}$), a high ICE of 81.7%, and a capacity retention of 81.9% after 1000 cycles at 2 C (from 226.0 to 185.1 mAh g^{-1}). In contrast, untreated HC shows a discharge capacity of 320.3 mAh g^{-1} at 0.1 C , a low ICE of 68.0%, and a limited capacity of 90.4 mAh g^{-1} after 1000 cycles at 2 C . In addition, the H_2SO_4 and H_2O_2 + NaOH treated samples also outperform the pristine HC.

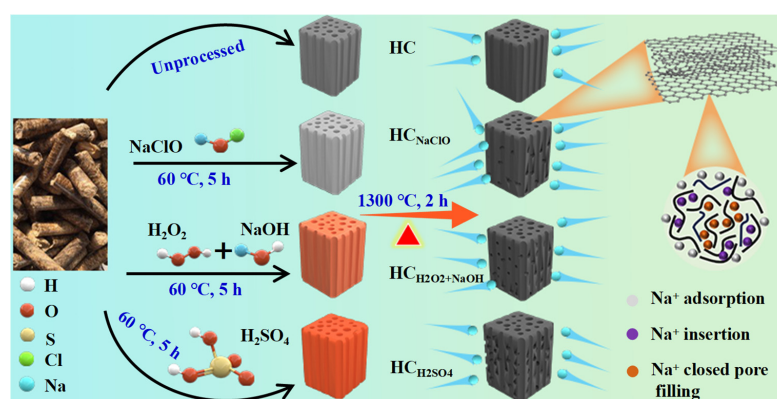


Figure 1. Scheme of the synthesis routes of HC and HC_x ($x = \text{NaClO}$, H_2SO_4 and H_2O_2 + NaOH) and their Na^+ storage mechanism.

2. Experimental Section

2.1. Material Synthesis

Cotton stalks were chemically activated using three different pre-treatment solution systems. In the H_2O_2 + NaOH system, 6 g of NaOH (AR, >96%, Nanjing Chemical Reagent Co., Ltd., Nanjing, Jiangsu, China) and 10 mL of H_2O_2 (AR, 30%, Chengdu Kelong Chemical

Co., Ltd., Chengdu, Sichuan, China) were dissolved in 290 mL of deionized (DI) water. In the H_2SO_4 system, 2.2 mL of concentrated sulfuric acid (AR, 95–98%, Chengdu Kelong Chemical Co., Ltd.) was diluted with DI water to a final volume of 400 mL. In the NaClO system, 10 mL of a 10 wt% NaClO (AR, 6–14% available chlorine, Macklin, Shanghai, China) solution was mixed with 290 mL of DI water. The stalks were individually immersed in these three systems and reacted at 60 °C under constant stirring for 5 h.

Following the reaction, surface impurities were eliminated, and the stalks were thoroughly rinsed with DI water until the supernatant became completely colorless. The purified products were subsequently dried in an oven at 60 °C for 12 h. The dried precursors were then placed in a tube furnace, heated to 1300 °C at a ramp rate of 2 °C min^{−1} under an Ar atmosphere, and held for 2 h. The resulting samples were designated as HC_x ($x = \text{NaClO}$, H_2SO_4 and $\text{H}_2\text{O}_2 + \text{NaOH}$). As a control, untreated raw stalks were directly subjected to the same annealing procedure and designated as HC.

2.2. Material Characterization

The morphological features and microstructural characteristics of the specimens were examined by scanning electron microscopy (SEM, Zeiss Merlin, Carl Zeiss, Oberkochen, Germany) equipped with an energy-dispersive spectrometer (EDS) and transmission electron microscopy (TEM, JEOL JEM-F200, 200 kV, Akishima, Tokyo, Japan). X-ray diffraction (XRD) patterns were collected on a Rigaku SmartLab SE (Tokyo, Japan) using Cu K α radiation ($\lambda = 0.15406$ nm). Raman spectra were acquired with a confocal micro-Raman spectrometer (Renishaw plc, Wotton-under-Edge, UK) under 532 nm excitation. X-ray photoelectron spectroscopy (XPS) was carried out on an Thermo Scientific K-Alpha (Thermo Fisher Scientific, East Grinstead, UK) system employing monochromatic Al K α radiation (1486.6 eV). Nitrogen adsorption–desorption isotherms were measured at 77 K on a Micromeritics ASAP 2460 instrument (Norcross, Georgia, USA). The specific surface area was calculated using the Brunauer–Emmett–Teller (BET) method, and the pore size distribution was determined by the Barrett–Joyner–Halenda (BJH) method.

2.3. Electrochemical Tests

The electrochemical properties were evaluated using CR2032 coin cells (Canrd Technology Co., Ltd., Dongguan, China) assembled with sodium metal foil as the counter electrode, 1 M NaPF₆ (battery-grade, DoDoChem, Suzhou Dodochem Co., Ltd., Suzhou, Jiangsu, China) in diglyme as the electrolyte, and a glass fiber membrane (Whatman, Cytiva, Maidstone, Kent, UK) as the separator. The working electrode was prepared by mixing the active material (HC_x), carbon black (Kejing Star, Shenzhen Kejing Star Technology Co., Ltd., Shenzhen, Guangdong, China), and sodium carboxymethyl cellulose (CMC, Kejing Star) at a mass ratio of 8:1:1 in DI water, and then casting the resultant slurry onto copper foil (Hubei Zhongyi Technology Inc., Xiaogan, Hubei, China). All electrode sheets were vacuum-dried at 80 °C for 12 h. The active material mass loading on the electrode was approximately 1.2 mg cm^{−2}.

Galvanostatic charge–discharge (GCD) measurements were performed at ambient temperature using a LAND battery testing system. Each material in our assembled coin cell was tested three times or more. Cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) were carried out on a CHI 760E electrochemical workstation (CHI, Shanghai, China) at 25 °C, with CV sweep rates spanning 0.1 to 1 mV s^{−1} and an EIS frequency range from 10⁵ to 0.1 Hz. The sodium-ion diffusion coefficient (D_{Na^+}) was evaluated via the galvanostatic intermittent titration technique (GITT) by applying a 0.1 C

pulse current (20 min discharge followed by 120 min relaxation) and calculated according to the following equation [35]:

$$D_{\text{Na}^+} = \frac{4}{\tau\pi} \left(\frac{m_B V_M}{M_B S} \right)^2 \left(\frac{\Delta E_S}{\Delta E_\tau} \right)^2$$

where τ denotes the constant current discharge time. m_B represents the mass of the active material. V_M is the molar volume. M_B corresponds to the molar mass of the active material. S indicates the electrode surface area. ΔE_S refers to the steady-state voltage change. ΔE_τ signifies the total voltage change during the pulse.

3. Results and Discussion

To elucidate the regulatory effects of distinct pretreatment strategies on the macroscopic morphology and microstructure of the resulting carbon materials, the architectures of HC and HC_{*x*} (where *x* = NaClO, H₂SO₄ and H₂O₂+NaOH) were systematically characterized by SEM and TEM. As shown in the SEM image (Figure 2a), HC retains the fibrous architecture of cotton stalks with diameters ranging from 10 to 20 μm. This structure is formed through gas evolution during pyrolysis, which induces wall shrinkage, wrinkling, and surface densification. In contrast, HC_{NaClO} (Figure 2b) develops a well-defined, three-dimensionally interconnected porous network with thin, honeycomb-like walls (1–2 μm, red arrow in the inset of Figure 2b). HC_{H₂O₂+NaOH} (Figure 2c) maintains the fibrous morphology but features thinner walls and more open pores compared to HC_{NaClO}, albeit with less uniform porosity. While all samples undergo pyrolysis-induced collapse, the severity varies significantly. HC_{H₂SO₄} exhibits the most pronounced collapse, forming dense micro-block aggregates with disordered carbon layers and indistinct nanopores (Figure 2d). In contrast, other samples show only mild collapse characterized by wall shrinkage or local pore deformation, without aggregation.

EDS mapping (Figure 2e–h) reveals a uniform O/C distribution in HC, with an oxygen content of 11.9 wt%. HC_{H₂SO₄} exhibits the highest O content (14.6 wt%), but displays clustered heterogeneity due to structural collapse. Both HC_{H₂O₂+NaOH} (12.3 wt% O) and HC_{NaClO} (5.1 wt% O) show uniform elemental distributions. The former benefits from a synergistic oxidation/etching mechanism that grafts oxygen functional groups while thinning the pore walls, whereas the lower O content of the latter underscores the pivotal role of NaClO in exfoliation and pore generation. TEM analysis (Figure 2i) further resolves the microstructure of HC_{NaClO}, revealing a carbon interlayer spacing of ~0.373 nm (inset), which is larger than that of graphite (0.335 nm) and thus facilitates reversible Na⁺ intercalation. Notably, numerous closed micropores (2–5 nm, yellow arrows) are also observed, serving as key sites for low-potential pore-filling reactions that contribute to the plateau capacity.

Figure 3 presents a systematic analysis of the microcrystalline structure, pore characteristics, and surface chemical states of HC and HC_{*x*}. The XRD patterns (Figure 3a) reveal that all samples display two broad diffraction peaks centered near 23.5° and 43°, which correspond to the (002) and (100) planes of graphite, respectively. The interlayer spacings were determined to be 0.393 nm (HC), 0.368 nm (HC_{H₂O₂+NaOH}), 0.380 nm (HC_{NaClO}), and 0.373 nm (HC_{H₂SO₄}) [36]. All values are well within the established range for hard carbon (0.36–0.42 nm), in good agreement with the TEM observations (Figure 2i).

Figure 3b,c presents the Raman spectra (Figure 3b) alongside the corresponding peak deconvolution (Figure 3c) for all samples. As can be seen, all samples exhibit two characteristic bands centered near 1350 and 1580 cm^{−1}, which are attributed to the D band (associated with disordered and defective domains) and the G band (associated with ordered graphitic structures), respectively [37]. The defect ratio (A_{D1}/A_G), derived from the

integrated peak areas, serves as a quantitative metric for the structural ordering of carbon, where a higher value signifies an elevated defect density and increased disorder within the carbon lattice. The defect ratios follow the sequence of HC_{NaClO} (1.44) > HC (1.25) > $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ (1.21) > $\text{HC}_{\text{H}_2\text{SO}_4}$ (1.18). It indicates that stronger oxidative treatments reduce the overall defect density by selectively removing disordered carbon domains and annealing defects at 1300 °C, whereas mild NaClO treatment preserves more defect sites. This heightened defect density provides abundant active sites for Na^+ adsorption, thereby bolstering the sodium storage capacity.

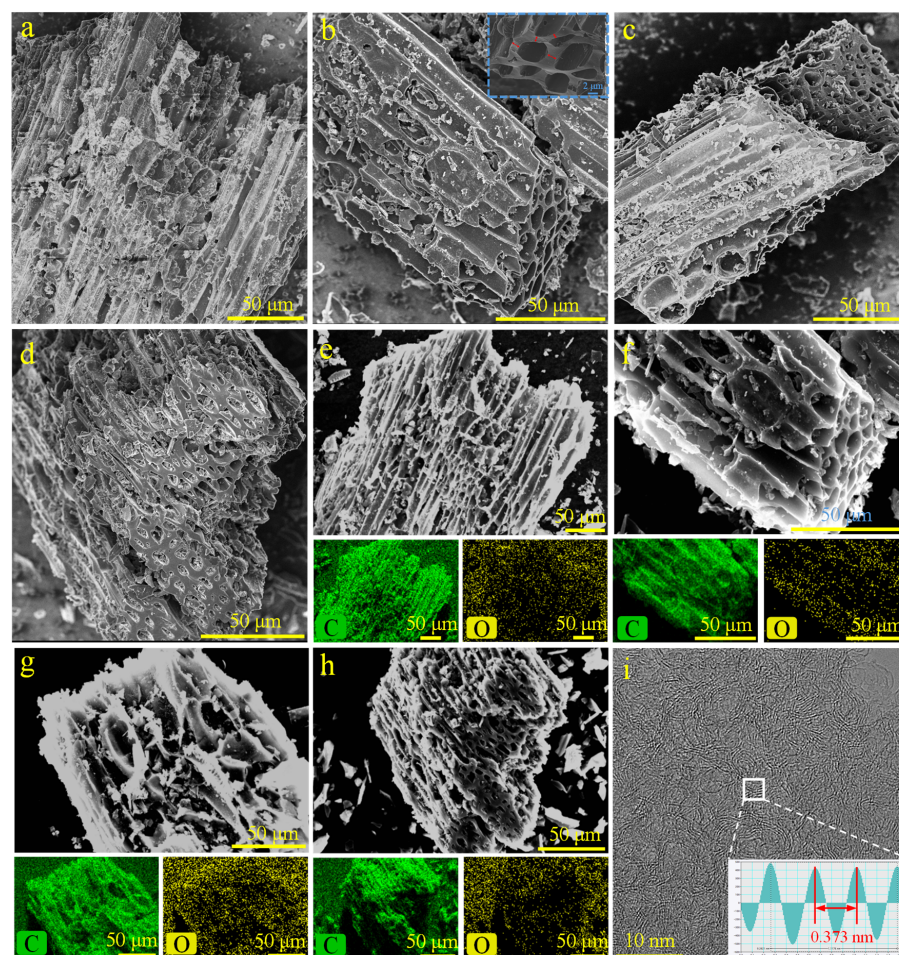


Figure 2. (a–d) SEM images and (e–h) corresponding EDS elemental mappings of (a,e) HC, (b,f) HC_{NaClO} , (c,g) $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$, and (d,h) $\text{HC}_{\text{H}_2\text{SO}_4}$. (i) HRTEM image of HC_{NaClO} . The insets in (b,i) are the high-magnification SEM image and the measurement of the interlayer spacing of HC_{NaClO} , respectively.

Nitrogen adsorption–desorption isotherms (Figure 3d) and the corresponding pore size distribution curves (Figure 3e) demonstrate that all samples exhibit hybrid type I/IV isotherms, indicative of the coexistence of micropores and mesopores. The Brunauer–Emmett–Teller (BET) specific surface areas and total pore volumes follow the sequence $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ ($99.7 \text{ m}^2 \text{ g}^{-1}$ and $0.097 \text{ cm}^3 \text{ g}^{-1}$) $\approx$ HC_{NaClO} ($97.6 \text{ m}^2 \text{ g}^{-1}$ and $0.069 \text{ cm}^3 \text{ g}^{-1}$) $\gg$ HC ($37.3 \text{ m}^2 \text{ g}^{-1}$ and $0.019 \text{ cm}^3 \text{ g}^{-1}$) $\approx$ $\text{HC}_{\text{H}_2\text{SO}_4}$ ($37.2 \text{ m}^2 \text{ g}^{-1}$ and $0.024 \text{ cm}^3 \text{ g}^{-1}$), with the former pair exhibiting approximately 2.7 and 5.1-fold enhancements relative to pristine HC in surface area and pore volume, respectively. The average pore sizes decrease in the order of $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ (3.9 nm) > HC_{NaClO} (2.8 nm) = HC (2.8 nm) > $\text{HC}_{\text{H}_2\text{SO}_4}$ (2.6 nm). The pore size distribution (Figure 3e), derived from the BJH desorption branch and strictly limited to the mesopore range (2–50 nm), further reveals

that $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ displays the most pronounced mesopore structure, characterized by a distinct peak centered at 3.5–4.0 nm. HC_{NaClO} shows a similar but markedly weaker mesopore signature. In contrast, HC and $\text{HC}_{\text{H}_2\text{SO}_4}$ exhibit severely limited pore development, with the latter displaying the lowest pore content owing to structural collapse. In contrast, HC and $\text{HC}_{\text{H}_2\text{SO}_4}$ exhibit severely limited pore development, with the latter displaying the lowest pore content owing to structural collapse.

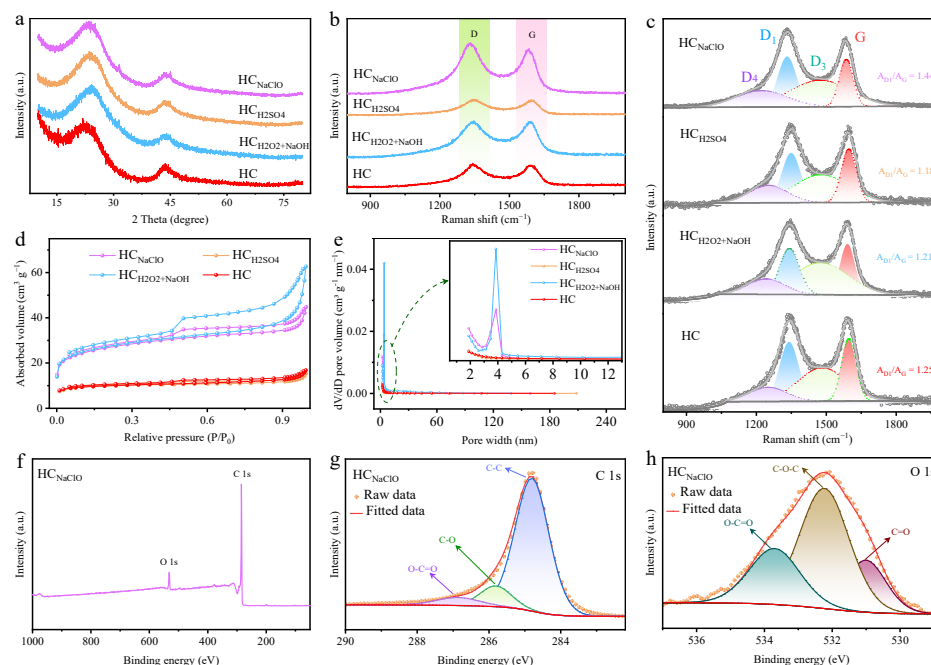


Figure 3. (a) XRD patterns, (b) Raman spectra and (c) peak fitting of Raman spectra, (d) N_2 adsorption/desorption isotherms and (e) pore size distribution. (f) Full-survey XPS spectra of HC_{NaClO} and its high-resolution XPS spectra: (g) C 1s and (h) O 1s.

XPS was employed to further elucidate the surface chemical states of HC_{NaClO} . The full-survey XPS spectrum (Figure 3f) reveals a surface composition dominated by C and O elements, and no impurities are detected. High-resolution C 1s (Figure 3g) and O 1s (Figure 3h) spectra, deconvoluted into constituent peaks, confirm the presence of abundant oxygen-containing functional groups on the HC_{NaClO} surface. The C 1s spectrum can be resolved into three components of C–C (284.8 eV), C–O (285.8 eV) and O–C=O (286.9 eV). The O 1s spectrum distinguishes three oxygen species of O–C=O (533.7 eV), C=O (531.0 eV) and C–O–C (532.4 eV).

To evaluate the electrochemical sodium storage performance of HC and HC_x as anodes for SIBs, systematic electrochemical measurements were performed. The initial charge–discharge profiles (Figure 4a) reveal that all electrodes exhibit characteristic HC sodium storage behavior, comprising a high-potential sloping region (>0.1 V, associated with Na^+ adsorption on carbon layer surfaces and defect sites) and a low-potential plateau region (<0.1 V, corresponding to Na^+ filling into closed pores and sodium cluster formation) [12,38]. All modified HC_x demonstrate a higher ICE than pristine HC (68.0%), with HC_{NaClO} attaining the highest value (81.7%), followed by $\text{HC}_{\text{H}_2\text{SO}_4}$ (75.3%) and $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ (74.2%). The superior ICE of HC_{NaClO} is attributed to fewer oxygen groups and more defects, which stabilize the solid-state electrolyte interface (SEI) film and create additional active sites for Na^+ storage. $\text{HC}_{\text{H}_2\text{SO}_4}$ and $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ exhibit lower ICE. This is most likely because $\text{HC}_{\text{H}_2\text{SO}_4}$ undergoes structural collapse that increases irreversible decomposition, while $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ possesses higher oxygen groups and a lower degree of structural order compared to HC_{NaClO} .

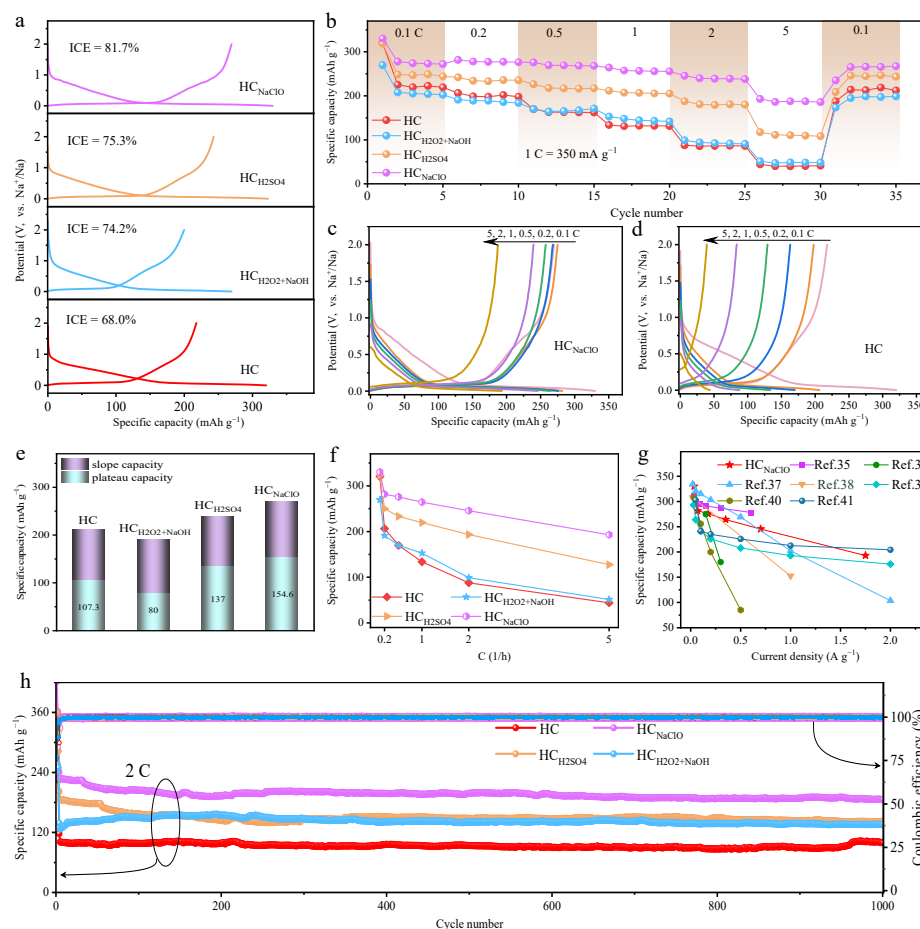


Figure 4. (a) Initial GCD profiles at 0.1 C, and (b) rate capabilities of all electrodes. GCD profiles of (c) HC_{NaClO} and (d) HC at various rates. (e) Slope-to-plateau capacity ratio of all electrodes and (f) their capacity retention versus C-rate. (g) Electrochemical performance comparison of HC_{NaClO} with previously reported typical HC anodes for SIBs. (h) Long-term cycling performance of all electrodes measured at 2 C.

Rate performance (Figure 4b) shows that HC_{NaClO} delivers discharge capacities of 330.2, 281.7, 275.9, 264.3, 245.6 and 193.0 mAh g^{-1} at 0.1, 0.2, 0.5, 1, 2 and 5 C, respectively, outperforming $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ (269.8 mAh g^{-1} at 0.1 C), $\text{HC}_{\text{H}_2\text{SO}_4}$ (323.2 mAh g^{-1} at 0.1 C), and HC (320.3 mAh g^{-1} at 0.1 C) across all rates. At 5 C, HC_{NaClO} retains 58.4% of its 0.1 C capacity, versus 39.5% ($\text{HC}_{\text{H}_2\text{SO}_4}$), 19.0% ($\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$), and 13.8% (HC), confirming the benefit of its porous architecture and oxygen-containing functional groups for rate capability. The GCD profiles (Figure 4c,d) reveal that, unlike HC, whose low-voltage plateau collapses at higher rates, HC_{NaClO} preserves a well-defined plateau even at 5 C (Figure 4c). Plateau capacity analysis (Figure 4e) further shows that HC_{NaClO} achieves the highest plateau contribution (154.6 mAh g^{-1} , 57.3% of total) and $\text{HC}_{\text{H}_2\text{SO}_4}$ (137 mAh g^{-1} , 57.4%), exceeding HC (107.3 mAh g^{-1} , 50.5%) and $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ (80 mAh g^{-1} , 41.7%). Moreover, HC_{NaClO} exhibits superior rate capability (Figure 4g) compared with typical hard carbon anodes reported previously [13,19,39–43].

Figure 4h shows the cycling performance of HC and HC_x measured at 2 C. Before cycling, all electrodes were activated at 0.1 C for a few cycles. HC_{NaClO} delivers an initial charge capacity of 226.0 mAh g^{-1} and retains 81.9% after 1000 cycles. $\text{HC}_{\text{H}_2\text{SO}_4}$ gives 183.5 mAh g^{-1} with 77.1% retention. $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ exhibits an obvious continuous activation behaviour, with its capacity rising from 122.6 to a peak of 156.6 mAh g^{-1} at the 191st cycle, then gradually declining to 136.3 mAh g^{-1} after 1000 cycles, corresponding to 87.0% retention relative to the peak. This activation and subsequent slow decay are likely

associated with its high specific surface area and abundant porosity (Figure 2d,e), which facilitate electrolyte infiltration but may also induce gradual structural rearrangement during prolonged cycling. Pristine HC shows only 100.2 mAh g^{-1} but retains 90.2% after 1000 cycles. Despite the differences in cycling stability, all electrodes achieve high average Coulombic efficiencies approaching 100%, confirming the formation of a stable SEI and good structural stability.

Figure 5 presents the kinetics of sodium storage through CV, EIS, and GITT analysis [44,45]. The CV profile of HC_{NaClO} (Figure 5a) exhibits a pair of broad redox peaks at 0.1 mV s^{-1} with a reduction peak at $\sim 0.02 \text{ V}$ and an oxidation peak at $\sim 0.11 \text{ V}$, indicative of reversible Na^+ intercalation/deintercalation and consistent with the combined sloping and plateau regions of the charge–discharge curves. Across the scan rate range of $0.1\text{--}1.0 \text{ mV s}^{-1}$ (Figure 5b), the fitted b-values for each redox peak all approximate 0.5, signifying that the electrochemical reaction is predominantly governed by semi-infinite diffusion kinetics. Quantitative assessment of the pseudocapacitive contribution (Figure 5c,d) reveals that the pseudocapacitive fractions for HC_{NaClO} at scan rates of 0.1, 0.2, 0.4, 0.6, 0.8, and 1.0 mV s^{-1} are 48%, 59%, 70%, 79%, 85% and 89%, respectively. This indicates a transition from diffusion-controlled behavior at low rates to capacitive-controlled behavior at high rates. Notably, the higher diffusion contribution at low rates (e.g., 52% at 0.1 mV s^{-1}) enables substantial bulk-phase sodium storage, which is consistent with the high reversible capacity and plateau capacity. In this process, Na^+ diffuses into interlayer galleries and closed pores rather than being restricted to surface adsorption.

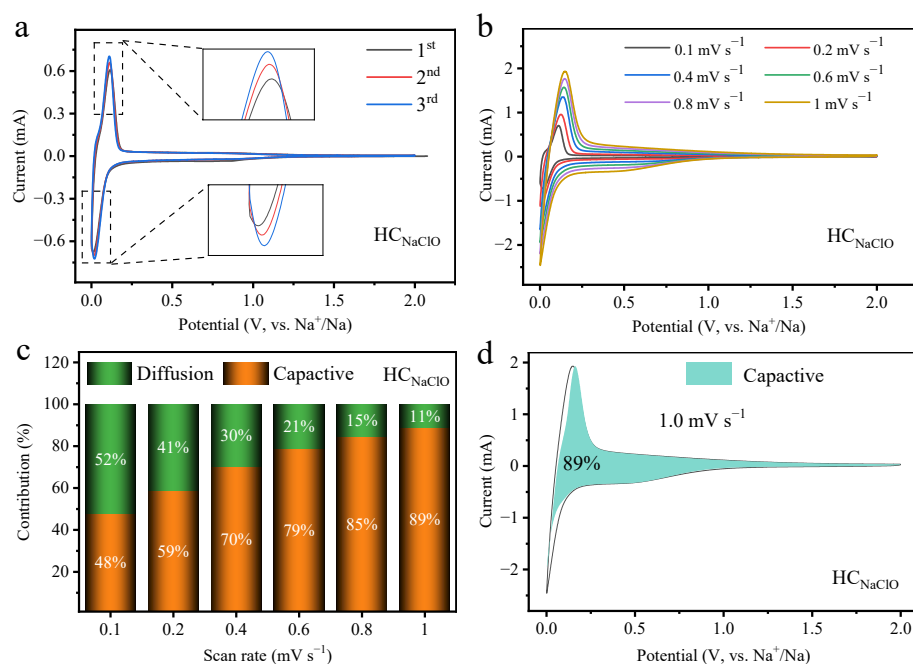


Figure 5. (a) Initial CV profiles for the first three cycles at 0.1 mV s^{-1} , and (b) CV curves at various scan rates for the HC_{NaClO} electrode. Insets of (a) are the corresponding enlarged areas. (c) Evolution of the capacitive- and diffusion-controlled capacity contributions for the HC_{NaClO} electrode as a function of scan rate. (d) Deconvolution of the capacitive contribution for HC_{NaClO} at 1.0 mV s^{-1} .

EIS analysis compares HC_{NaClO} , $\text{HC}_{\text{H}_2\text{SO}_4}$ and $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ measured at 2 C before cycling (Figure 6a) and after 100 cycles (Figure 6b). The equivalent circuit model (Inset of Figure 6a) used for fitting includes R_o (ohmic resistance from the electrolyte, separator, and current collector), R_e (SEI resistance for the electrode after cycling), R_{ct} (interfacial charge-transfer resistance, a key indicator of electrode kinetics), CPE (non-ideal double-layer capacitance), and W_o (semi-infinite Na^+ diffusion). The fitted R_o values are 12.9,

12.2, and 10.3 Ω , respectively, showing only slight variation and indicating consistent electrode/electrolyte contact across the samples. Notably, before cycling (Figure 6a), the pristine electrodes show R_{ct} values in the order of HC_{NaClO} (10.3 Ω) < $HC_{H_2SO_4}$ (31.4 Ω) < HC (34.7 Ω) < $HC_{H_2O_2+NaOH}$ (48.0 Ω). After 100 cycles at 2 C, all electrodes show decreased R_{ct} (Figure 6b), yielding 1.7 Ω for HC_{NaClO} , 5.2 Ω for $HC_{H_2SO_4}$, 17.6 Ω for HC , and 44.9 Ω for $HC_{H_2O_2+NaOH}$. This general decline arises from electrode activation during the initial cycling process. Nevertheless, the extent of reduction varies markedly. $HC_{H_2SO_4}$ exhibits the largest absolute drop (from 31.4 to 5.2 Ω), while HC_{NaClO} reaches the lowest value (1.7 Ω), both benefiting from the formation of robust and highly conductive SEI layers that effectively facilitate Na^+ transport. The untreated HC shows a moderate decrease but retains a higher impedance. In contrast, the H_2O_2+NaOH treated electrode experiences only a marginal reduction, indicating that excessive oxygen-containing groups and structural damage lead to a thick and poorly ion-conducting SEI, which severely blocks charge transfer.

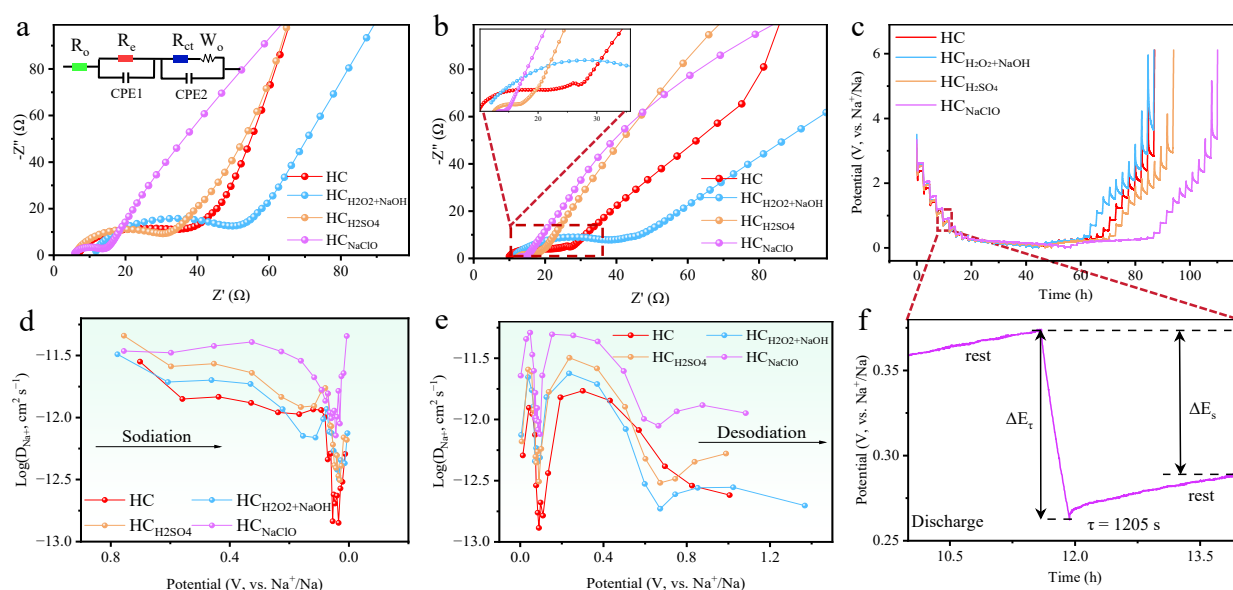


Figure 6. EIS spectra of all electrodes measured at 2 C (a) before cycling and after 100 cycles. Insets in (a,b) are the corresponding equivalent circuit model and enlarged areas, respectively. (c) GITT profiles for all electrodes during charge and discharge, and (d,e) the associated evolution of D_{Na^+} throughout the cycle. (f) The enlarged view of the HC_{NaClO} GITT profile from (c).

GITT measurements (Figure 6c–f) track the evolution of the Na^+ diffusion coefficient (D_{Na^+}) during cycling. HC_{NaClO} exhibits D_{Na^+} values in the range of 10^{-11} – 10^{-13} $cm^2 s^{-1}$, with an average of $\sim 2.35 \times 10^{-12}$ $cm^2 s^{-1}$, which is superior to HC ($\sim 7.47 \times 10^{-13}$ $cm^2 s^{-1}$), $HC_{H_2O_2+NaOH}$ ($\sim 4.0 \times 10^{-13}$ $cm^2 s^{-1}$), and $HC_{H_2SO_4}$ ($\sim 3.0 \times 10^{-13}$ $cm^2 s^{-1}$). This is in agreement with the rate performance and R_{ct} trends. The D_{Na^+} of all electrodes initially remains stable and then declines, reflecting the three-stage sodium storage mechanism with high-voltage adsorption (2.0–0.3 V, fast diffusion), mid-voltage intercalation (0.3–0.1 V, gradually decreasing diffusion), and low-voltage pore filling (<0.1 V, sharp drop due to sluggish transport through dense carbon layers into closed micropores).

Combining the morphological evolution and surface chemistry analysis, the mechanism underlying the different electrochemical performances of electrodes measured at 2 C before cycling and after 1000 cycles can be further revealed in Figures 7 and 8. As shown in Figure 7b, after 1000 cycles at 2 C, HC_{NaClO} preserves the most intact microstructure with negligible pulverization, whereas other electrodes all suffer varying degrees of structural degradation. Notably, $HC_{H_2O_2+NaOH}$ shows the most pronounced pulverization and

microcracks (Figure 7h). The corresponding EDX elemental mappings further confirm that the Na and O elements are remarkably uniformly distributed on the HC_{NaClO} surface (Figure 7c), once again supporting the formation of a homogeneous SEI layer, which effectively suppresses excessive electrolyte erosion.

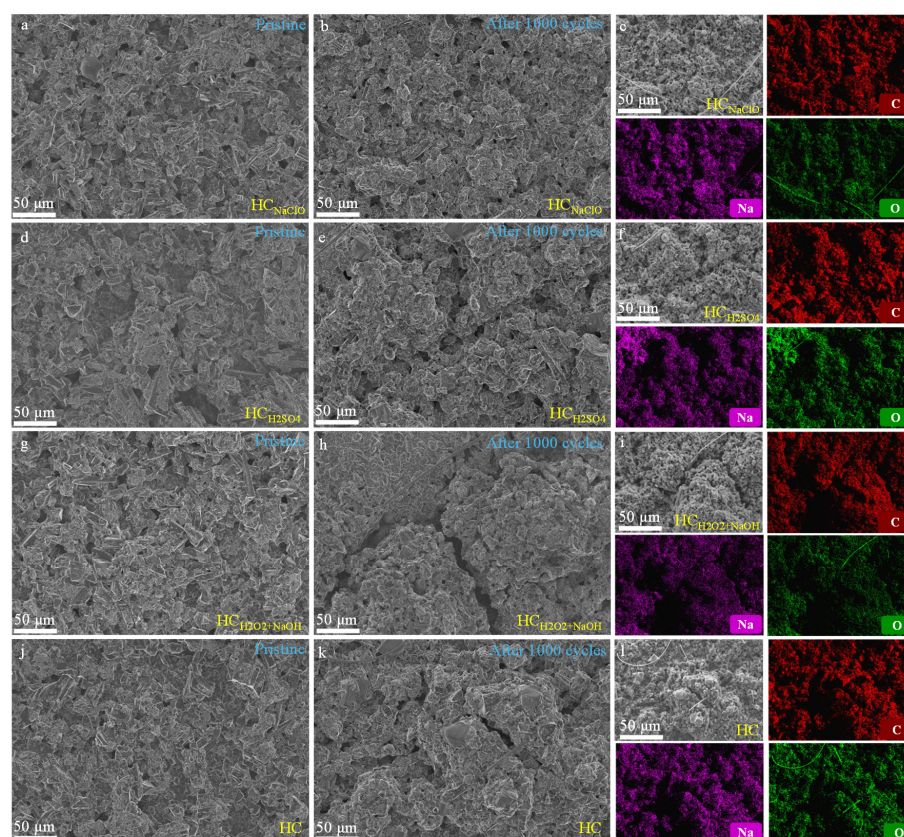


Figure 7. (a,b,d,e,g,h,j,k) SEM images and (c,f,i,l) corresponding EDX elemental mappings of Na, O, and C for (a–c) $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$, (d–f) $\text{HC}_{\text{H}_2\text{SO}_4}$, (g–i) HC and (j–l) HC_{NaClO} electrodes (a,d,g,j) before and (b,c,e,f,h,i,k,l) after 1000 cycles.

The XPS spectra (Figure 8) further elucidate the interfacial chemistry of the electrodes measured at 2 C after 1000 cycles. As can be seen, the O 1s spectra reveal that $\text{HC}_{\text{H}_2\text{SO}_4}$ exhibits the strongest O–C=O (carboxyl) signals (Figure 8e), while both HC (Figure 8h) and $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ (Figure 8k) show comparable moderate intensities, both of which are noticeably higher than those of HC_{NaClO} (Figure 8b). Quantified results show that HC_{NaClO} exhibits the lowest O–C=O content (13.3%), followed by HC (17.6%) and $\text{HC}_{\text{H}_2\text{SO}_4}$ (24.1%). The H₂O/–OH fraction is also minimal for HC_{NaClO} (18.8%) compared with both HC and $\text{HC}_{\text{H}_2\text{SO}_4}$ (>21%), while the C=O signal is strongest for HC_{NaClO} (55.3%) and weaker for the other two. It is generally suggested that moderate C=O/C–O groups can promote reversible sodium storage by providing active adsorption sites, whereas excessive O–C=O groups act as catalytic sites for electrolyte decomposition that generate a thick SEI [46,47]. Meanwhile, NaF is widely recognized as a crucial component for constructing stable SEI layers. HC exhibits the lowest NaF content (vs. C–F) in the F 1s spectrum (Figure 8l), compared to the other three. Overall, HC_{NaClO} features suppressed O–C=O signals with a balanced C=O/C–O distribution in the O 1s spectrum (Figure 8c), which combines with its moderate NaF content (Figure 8c) to promote the formation of a thin, robust, and inorganic-rich SEI. Consequently, HC_{NaClO} achieves the lowest charge-transfer resistance and superior cycling stability.

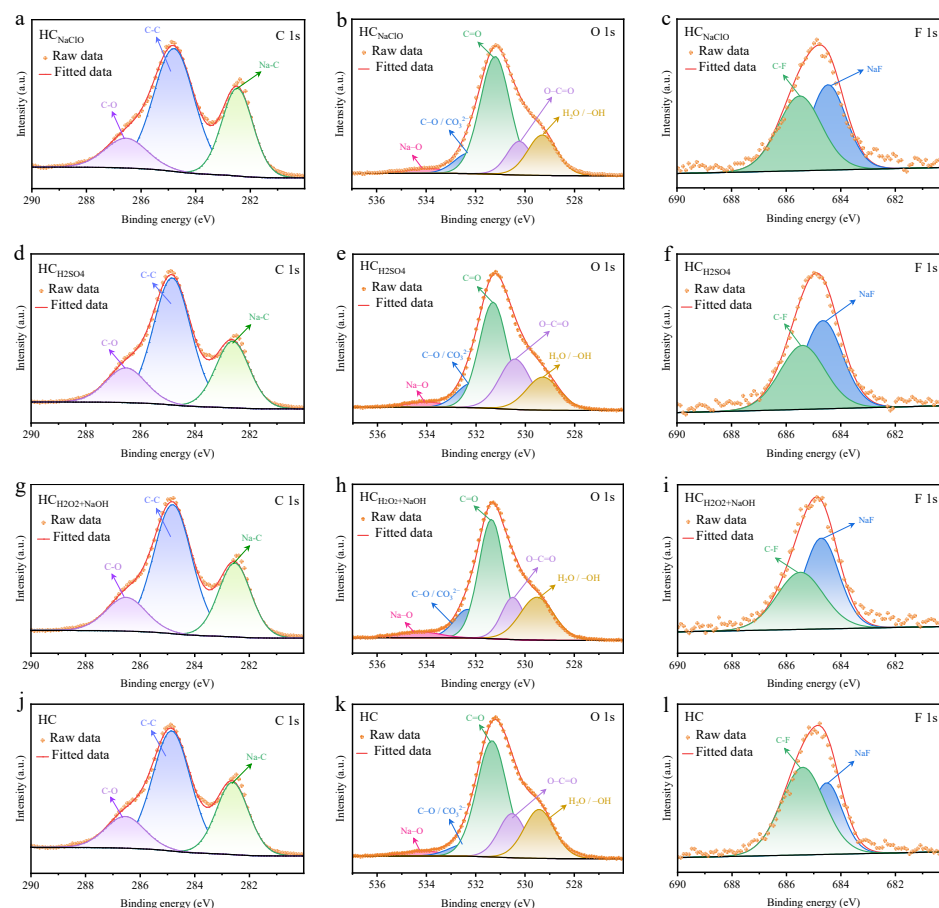


Figure 8. High resolution XPS spectra of C 1s, O 1s and F 1s of (a–c) HC_{NaClO} , (d–f) $\text{HC}_{\text{H}_2\text{SO}_4}$, (g–i) $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ and (j–l) HC measured at 2 C after 1000 cycles: (a,d,g,j) C 1s, (b,e,h,k) O 1s and (c,f,i,l) F 1s.

4. Conclusions

In summary, this work applies three chemical oxidation treatments (NaClO , H_2SO_4 , and $\text{H}_2\text{O}_2+\text{NaOH}$) to cotton stalk-derived HC carbonized at $1300\text{ }^\circ\text{C}$. All methods preserve the carbon skeleton and improve performance. The best results are from HC_{NaClO} , with a high ICE of 81.7% and a high discharge capacity of 330.2 mAh g^{-1} , followed by $\text{HC}_{\text{H}_2\text{SO}_4}$ (75.3% and 323.2 mAh g^{-1}) and $\text{HC}_{\text{H}_2\text{O}_2+\text{NaOH}}$ (74.2% and 269.8 mAh g^{-1}), all superior to pristine HC (68.0% and 320.3 mAh g^{-1}). At 2 C, the capacity retention after 1000 cycles is 81.9% for HC_{NaClO} (from 226.0 to 185.1 mAh g^{-1}) and 77.1% for $\text{HC}_{\text{H}_2\text{SO}_4}$ (from 183.5 to 141.5 mAh g^{-1}), while pristine HC maintains 90.4 mAh g^{-1} after 1000 cycles. Each treatment has its own feature, with NaClO building hierarchical pores and oxygen groups to boost capacity and ICE; H_2SO_4 causing densification that limits capacity; and $\text{H}_2\text{O}_2+\text{NaOH}$ thinning pore walls and opening mesopores to aid ion transport. Overall, chemical oxidation via combined pore and surface engineering proves an effective and versatile strategy for enhancing sodium storage in cotton stalk-derived HC.

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