



# Facile synthesis of MoS<sub>2</sub>/N-doped carbon as an anode for enhanced sodium-ion storage performance

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## Abstract

The scalable synthesis of MoS<sub>2</sub>/N-doped carbon (MoS<sub>2</sub>/NC) nanosheets was accomplished using a facile salt template-assisted synthesis method. The MoS<sub>2</sub> nanosheets were uniformly encapsulated in nitrogen-doped carbon frameworks. As an anode for sodium-ion batteries (SIBs), the electrochemical performance of the MoS<sub>2</sub>/NC electrode was significantly improved compared to pure MoS<sub>2</sub>, which demonstrated a reversible capacity of 334.6 mAh g<sup>-1</sup> after 100 cycles at 0.2 A g<sup>-1</sup>; even after 250 cycles of 1 A g<sup>-1</sup>, the capacity could still be maintained at 252.9 mAh g<sup>-1</sup>. This excellent sodium storage performance was mainly due to the coupling of nitrogen-doped carbon with MoS<sub>2</sub> and the uniform distribution of the nanosheets, which improved the reaction kinetics. Besides, the electrochemical reconstruction ensured the integrity of the electrode. This work was of great significance for the large-scale synthesis of MoS<sub>2</sub> anode material with enhanced stability SIBs.

**Keywords** MoS<sub>2</sub>/NC · Sodium-ion batteries · Anode material · Enhanced stability · Electrochemical reconstruction

## Introduction

Lithium-ion batteries (LIBs), as an excellent energy storage material, have been developed rapidly in the past few decades [1]. However, the limited reserves cannot satisfy increasing demand [2]. By contrast, sodium element is abundant and low-cost. Besides this, compared with LIBs, sodium-ion batteries (SIBs) are safer and compatible with existing lithium-ion devices because of similar working principles, gradually becoming a research hotspot [3–5]. Unfortunately, the development of SIBs still has some limitations. For anode materials, traditional materials such as graphite are not enough to support the free penetration of sodium ions on account of the larger radius of Na. Some anode materials suitable for LIBs are insufficient for SIBs simultaneously [6, 7]. Therefore, it is one of the main tasks to design new anode materials appropriate for SIBs.

According to the sodium storage mechanism, SIB anode can be categorized into intercalation-, conversion-, and

alloying-type materials. Transition metal oxides [8, 9] and sulfides [10, 11] show excellent electrochemical performance in various anode materials. Especially, MoS<sub>2</sub>, with its unique layer-by-layer structure and high sodium-ion storage capacity, has tremendous development prospects in SIBs. Nevertheless, poor electrical conductivity and agglomeration tendency from the van der Waals force between MoS<sub>2</sub> layers lead to a long diffusion path and slow kinetics [12–14]. Furthermore, sodium-ion insertion/extraction repeatedly leads to significant volume expansion of MoS<sub>2</sub> and severe capacity decay in the cycle process, which results in poor cyclic capacity retention and rate capability [15–17].

To solve these intractable problems, a series of efforts such as doping [18, 19], multicomponent integration [20, 21], and morphology engineering [22, 23] have been made to improve the performance of MoS<sub>2</sub> for SIB anode. Among them, combining carbon with high conductivity is a very effective measure. The addition of carbon material can not only enhance the conductivity of MoS<sub>2</sub> but also buffer the volume variation during sodium insertion and extraction. For instance, Li et al. [24] grew MoS<sub>2</sub> nanosheets vertically on cotton-derived carbon fiber through hydrothermal and subsequent high-temperature carbonization processes. The material remained 323.1 mAh g<sup>-1</sup> after 150 cycles at 0.5 A g<sup>-1</sup>, showing good cycling performance. The predecessors introduced nitrogen doping on this basis to further improve

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the cyclic properties of composites. N-doped carbon@MoS<sub>2</sub> nanosheet composite was obtained through the combination of hydrothermal synthesis and high-temperature annealing process using g-C<sub>3</sub>N<sub>4</sub> as carbon and nitrogen source by Li et al. [25]. Owing to the great active sites for the storage of Na ion and enhancement of the diffusion rate and conductivity endowed by N-doped carbon, this material as an anode of SIBs had a profitable electrochemical capacity with a 246 mAh g<sup>-1</sup> of high reversible capacity after 1000 cycles at 1.5 A g<sup>-1</sup>. Although significant progress has been made, the synthesis methods are still relatively complex and unsuitable for large-scale synthesis. Hence, seeking a facile synthesis strategy with mild conditions and low cost to prepare MoS<sub>2</sub>/NC nanosheets is still essential.

Herein, we synthesized MoS<sub>2</sub>/NC nanosheets using a facile salt template-assisted synthesis method. The layered MoS<sub>2</sub> nanosheets were uniformly coated in nitrogen-doped carbon frameworks. As an anode for SIBs, the electrochemical performance of the electrode was significantly improved, such as high specific capacity and superior cycling behavior, due to the coupling of nitrogen-doped carbon with MoS<sub>2</sub> and electrochemical reconstruction, which provided improved electrical/ionic transporting capacity and architectural stability. This work provided a reference for studying and applying MoS<sub>2</sub> materials in SIBs.

## Experimental section

### Material

Ammonium molybdate ((NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·4H<sub>2</sub>O), thiourea (CS(NH<sub>2</sub>)<sub>2</sub>), glucose (C<sub>6</sub>H<sub>12</sub>O<sub>6</sub>), and sodium chloride (NaCl) were all analytical grade without extra purification requirement. All chemicals were purchased from Shanghai Aladdin Co., Ltd.

### Preparation of MoS<sub>2</sub>/NC

0.4 g (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·4H<sub>2</sub>O and 0.4 g CS(NH<sub>2</sub>)<sub>2</sub> were dispersed in 30 ml of deionized water, then 0.5 g C<sub>6</sub>H<sub>12</sub>O<sub>6</sub> and 0.5 g NaCl were added into the solution with stirring for 2 h to dissolve completely. The precursor was diverted to a porcelain boat and incinerated at 800°C for 2 h in a protective atmosphere (Ar) with a heating rate of 5°C per minute. The collected product was washed with distilled water and ethanol repeatedly after cooling to room temperature and then dried for 5 h in a vacuum oven at 60°C to acquire the MoS<sub>2</sub>/NC sample. As the control, the samples without adding NaCl (MoS<sub>2</sub>/NC (no NaCl)) and pure MoS<sub>2</sub> were prepared in the same approach.

## Electrochemical measurements

The coin-type battery was assembled according to the following process. Specifically, the active materials (70 wt%), acetylene black (20 wt%), and sodium alginate (10 wt%) were added to deionized water by thoroughly grinding to form a uniform slurry, and evenly spread on a Cu sheet, drying at 60°C for 6 h to remove water, then copper foil was cut into discs a diameter of 14 mm, and subjected to vacuum drying for 12 h at 120°C. The mass loading of the electrode prepared was about 0.6–1.0 mg cm<sup>-2</sup> and the amount of electrolyte in each battery was 40 μl. Using Na as the counter electrode and GF/D Whatman as the diaphragm, CR2032 coin batteries were fabricated in an expandable glove box for a series of tests. 1 M NaClO<sub>4</sub> was dissolved in ethylene carbonate/propylene carbonate (EC/PC, 1:1 vol %) and 5% fluoroethylene carbonate (FEC) was added as the electrolyte. The galvanostatic charge/discharge tests were carried out on the LAND Instrument (CT 3002A, Wuhan, China). The cyclic voltammograms (CV, 0.01–3 V, 0.2 mV s<sup>-1</sup>) and electrical impedance diagrams (EIS, 100 kHz–0.01 Hz, 5 mV s<sup>-1</sup>) of the samples were determined by an electrochemical workstation.

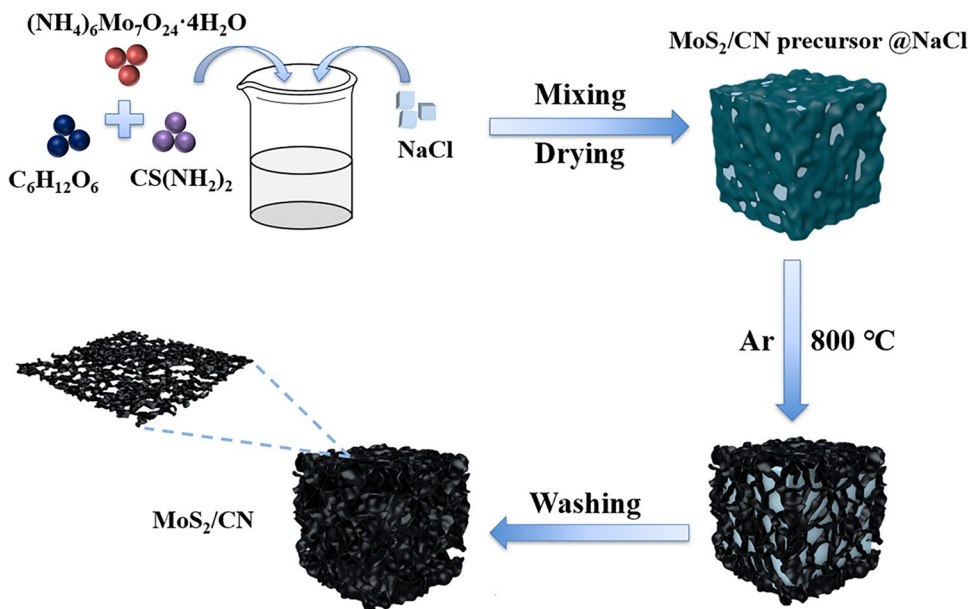
## Material characterization

The crystalline structure and phase composition of the samples were analyzed by X-ray powder diffraction (XRD, Rigaku D/max-2500). The thermogravimetric analysis was carried out at a heating rise of 20 °C min<sup>-1</sup> in the flowing air. The specific surface area and pore size distributions of the samples were obtained by analyzing the N<sub>2</sub> adsorption–desorption isotherms. The surface valence states of elements in the samples were characterized by X-ray photoelectron (XPS, Thermo Scientific ESCALAB XI<sup>+</sup>) with monochromatic Al Kα as the X-ray source. The Raman spectra were obtained on the InVia reflex Raman spectroscopy by using the laser excitation with a 532 nm. The microstructure was determined using a scanning electron microscope (SEM, Quanta FEG250) and a transmission electron microscope (TEM, Hitachi 800).

## Results and discussion

The preparation process of MoS<sub>2</sub>/NC nanosheets is shown in Fig. 1. First, the (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·4H<sub>2</sub>O, CS(NH<sub>2</sub>)<sub>2</sub>, C<sub>6</sub>H<sub>12</sub>O<sub>6</sub>, and NaCl were dissolved in deionized water and stirred for several hours. Secondly, the mixture was dried to remove water so the MoS<sub>2</sub>/NC precursor was well encased in the NaCl template. Subsequently, the obtained precursor was

**Fig. 1** Schematic illustration for the preparation of MoS<sub>2</sub>/NC nanosheets by a one-pot calcination method

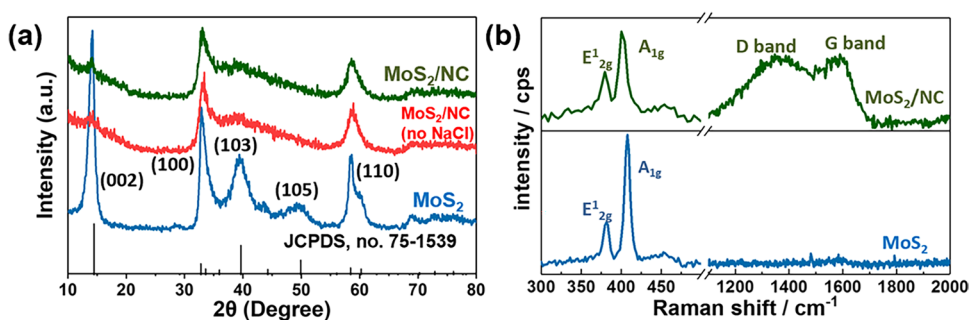


calcined in an Ar atmosphere. During the calcination process, Mo reacted with S ions, while glucose was carbonized at high temperatures and converted into an N-doped carbon framework. The interaction between the two made the MoS<sub>2</sub>/NC nanosheets grow uniformly on the surface of the NaCl template surface. Finally, MoS<sub>2</sub>/NC nanosheets were prepared by removing the NaCl template with deionized water.

XRD was conducted to characterize the crystalline of the pure MoS<sub>2</sub>, MoS<sub>2</sub>/NC (no NaCl), and MoS<sub>2</sub>/NC. As shown in Fig. 2a, the diffraction peaks at 14.1°, 32.8°, 39.8°, 49.3°, and 58.7° corresponded to the (002), (100), (105), (103), and (110) planes of hexagonal MoS<sub>2</sub> crystal (JCPDS No. 37–1492), respectively. It was noteworthy that the peaks located at (002) of the MoS<sub>2</sub>/NC and MoS<sub>2</sub>/NC (no NaCl) were weak, which could be the presence of carbon inhibiting the growth of (002) lattice plane [26–28]. In addition, according to the Debye–Scherrer equation, the crystallite sizes of the pure MoS<sub>2</sub>, MoS<sub>2</sub>/NC (no NaCl), and MoS<sub>2</sub>/NC samples were calculated using the peaks at (100) as 8.67 nm, 7.02 nm, and 7.08 nm, respectively. Compared with pure MoS<sub>2</sub>, the XRD peaks of MoS<sub>2</sub>/NC (no NaCl) and MoS<sub>2</sub>/NC samples were broader and the calculated grain sizes

were smaller, which might be caused by the reduction of crystallite size due to the addition of N-doped carbon [29]. Figure 2b presents the Raman spectra of the MoS<sub>2</sub>/NC and the MoS<sub>2</sub> materials. Two peaks at 379 and 404 cm<sup>-1</sup> were seen for the as-synthesized MoS<sub>2</sub> sample, representing the E<sup>1</sup><sub>2g</sub> and A<sub>1g</sub> vibration modes. Compared with pure MoS<sub>2</sub>, apart from the characteristic peaks of MoS<sub>2</sub>, the MoS<sub>2</sub>/NC showed two broad peaks at 1354 cm<sup>-1</sup> and 1578 cm<sup>-1</sup>, which could be well indexed to the D (disordered carbon) and G (graphitic carbon) bands of carbon, respectively [30]. The former represented the sp<sup>3</sup> hybrid, and the latter was related to the sp<sup>2</sup> hybrid. The calculated value of I<sub>D</sub>/I<sub>G</sub> was 1.22, indicating that the obtained MoS<sub>2</sub>/NC had a higher content of disordered carbon and more sp<sup>3</sup>-bond carbon. Raman’s results verified that pyrolyzed carbon and MoS<sub>2</sub> were successfully synthesized by this method. To estimate the relative contents of MoS<sub>2</sub> and C components in the MoS<sub>2</sub>/NC sample, thermogravimetric (TG) analysis was carried out in an air atmosphere. The weight loss of the MoS<sub>2</sub> sample at 350 °C–600 °C was 9.86%, mainly because of the oxidation of pure MoS<sub>2</sub> into MoO<sub>3</sub> (Fig. S1) [31–33]. For the MoS<sub>2</sub>/NC sample, the weight loss of the MoS<sub>2</sub>/NC sample had

**Fig. 2** a XRD patterns of MoS<sub>2</sub>/NC, MoS<sub>2</sub>/NC (no NaCl), and MoS<sub>2</sub>. b Raman spectra of MoS<sub>2</sub>/NC and MoS<sub>2</sub>

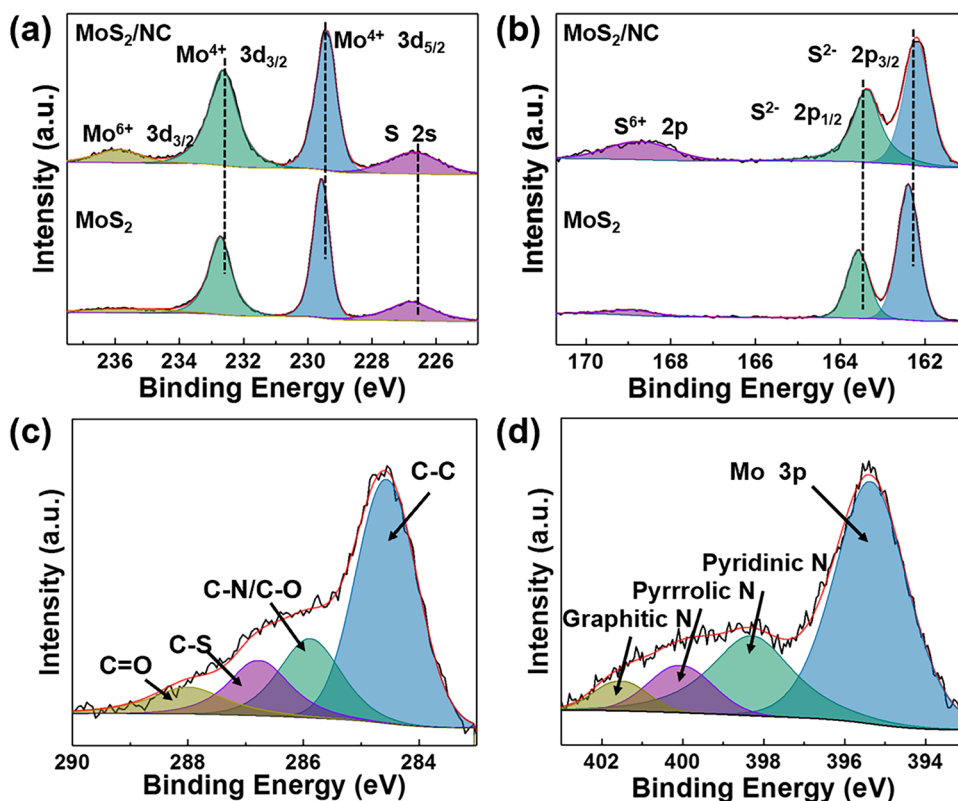


increased to 35.9% at the same temperature range because of the oxidation of  $\text{MoS}_2$  to  $\text{MoO}_3$  and the combustion of carbon. Therefore, the contents of  $\text{MoS}_2$  and carbon were 71.3% and 28.7%, respectively. Figure S2 shows the  $\text{N}_2$  adsorption/desorption isotherms of  $\text{MoS}_2/\text{NC}$  and  $\text{MoS}_2$  samples. The pure  $\text{MoS}_2$  sample showed a typical IV isotherm with a  $\text{H}_3$  hysteresis loop, while the  $\text{MoS}_2/\text{NC}$  sample displayed a  $\text{H}_2$  hysteresis loop. The specific surface areas of the two samples calculated by the Brunauer–Emmett–Teller (BET) method were  $5.95 \text{ m}^2 \text{ g}^{-1}$  and  $4.73 \text{ m}^2 \text{ g}^{-1}$ , respectively. The pore size distribution diagram showed that NaCl as a sacrificial template was crucial in reducing the pore size of the  $\text{MoS}_2/\text{NC}$ . Small pores could shorten diffusion pathways and facilitate sodium ions and electrons transfer during discharge and charge processes.

XPS was used to analyze the surface atomic composition of the samples of  $\text{MoS}_2/\text{NC}$  and  $\text{MoS}_2$ , as well as the results are displayed in Fig. 3. In the complete XPS spectra of the two samples, the signals of Mo, S, C, N, and O could be distinctly observed in  $\text{MoS}_2/\text{NC}$  sample, while the carbon content in pure  $\text{MoS}_2$  sample was significantly reduced, which was consistent with the expected results (Fig. S3). Figure 3a depicts the high-resolution XPS spectrum in the 3d region of Mo, where the two peaks at 229.4 and 232.6 eV represented  $3d_{5/2}$  and  $3d_{3/2}$  of  $\text{Mo}^{4+}$ , respectively, and the peak at 226.7 eV corresponded to the 2s signal of S. A small peak could also be observed at 236 eV, which was the

$3d_{3/2}$  signal of  $\text{Mo}^{6+}$  according to previous studies [34, 35]. The generation of this peak might result from the surface oxidation of the  $\text{MoS}_2/\text{NC}$  during the process of preparation and storage, which also explained the existence of the O element in the whole spectrum [36]. It can be seen from the XPS spectra of the S 2p region described in Fig. 3b that two peaks at 163.4 eV and 162.2 eV represented the  $2p_{1/2}$  and  $2p_{3/2}$  signals of  $\text{S}^{2-}$ , respectively. Similarly, a weak peak at 168.7 eV represented the  $\text{S}^{6+}$  signal, which was caused by the oxidation of  $\text{S}^{2-}$  in the atmosphere [37]. Compared with pure  $\text{MoS}_2$ , the peaks of  $\text{Mo}^{4+}$  3d and  $\text{S}^{2-}$  2p of the  $\text{MoS}_2/\text{NC}$  sample both shifted toward lower binding energy, which was caused by the increase of electron cloud density near  $\text{MoS}_2$  due to the addition of C [38]. Figure 3c is the fitted XPS spectrum of the 1s region of C, where 284.6, 285.9, 287.0, and 288.8 eV were categorized as C–C, C–N/C–O, C–S, and C=O bonds, respectively. This result indicated that N elements were successfully doped into carbon, which was further corroborated by the N 1s spectrum in Fig. 3d. Three peaks at 398.3, 400.2, and 401.9 eV could be identified as the N 1s region, representing pyridinic N, pyrrolic N, and graphitic N, respectively. The addition of N could improve the electrochemical performance of the materials mainly from the following aspects: on the one hand, N had a high electronegativity, and N doping could enhance the interaction between particles, thereby improving the electrochemical activity of the material. On the other hand, N

**Fig. 3** XPS spectra of  $\text{MoS}_2/\text{NC}$  and  $\text{MoS}_2$  a Mo 3d, b S 2p. XPS spectra of  $\text{MoS}_2/\text{NC}$ , c C 1s, d N 1s

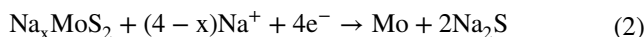


doping could also provide more defects, which improved the transport rate of sodium ions by interacting with them [39, 40].

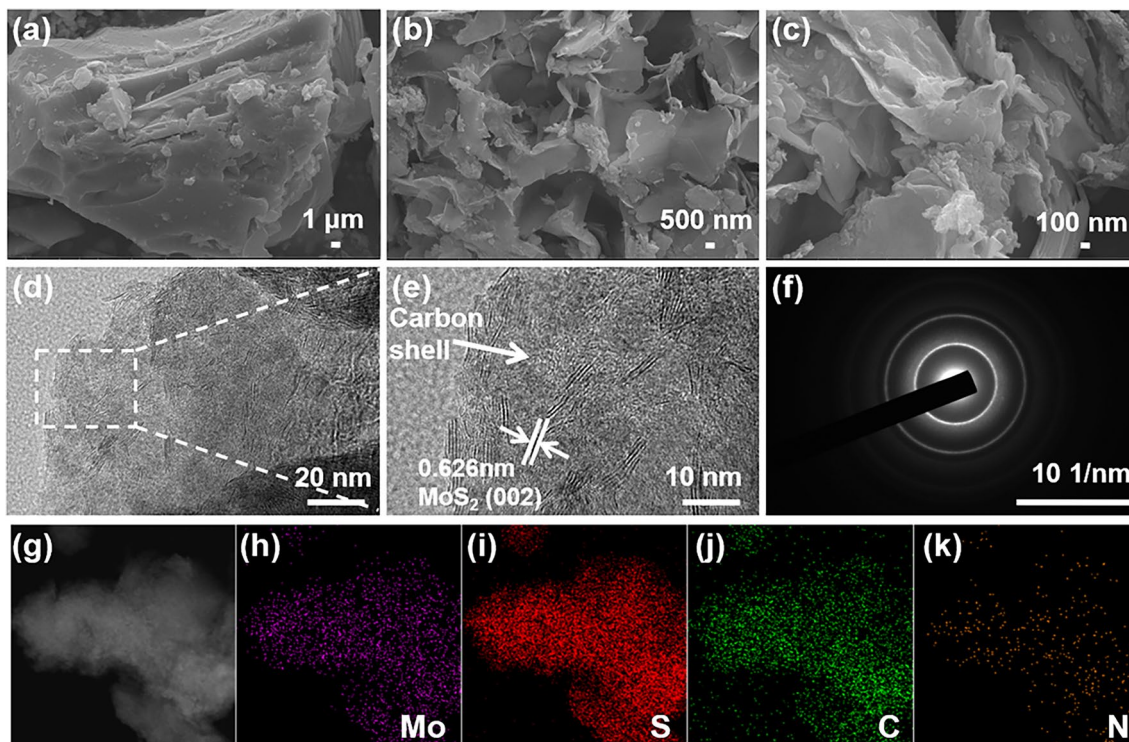
The morphological and microstructure characteristics were analyzed by SEM, TEM, and HRTEM. Figure 4a describes the SEM image of the MoS<sub>2</sub>/NC (no NaCl) sample, and as shown in the figure, the single MoS<sub>2</sub>/NC (no NaCl) sample presented an apparent massive structure. The MoS<sub>2</sub>/NC exhibited a typical nanosheet structure that the MoS<sub>2</sub> nanosheets were uniformly embedded in the nitrogen-doped carbon frameworks (Fig. 4b, c). The results indicated that the addition of water-removable template NaCl could effectively restrain the aggregation of MoS<sub>2</sub> and promote nanosheet formation. The average layer spacing of about 0.626 nm was represented to the (002) plane of MoS<sub>2</sub> in the TEM and HRTEM images (Fig. 4d, e). Specifically, we could observe that the MoS<sub>2</sub> nanosheets composed of 3–4 layers were evenly dispersed in the carbon shell. The cross-linked structure of these nanosheets was small and well combined with the carbon layer. This unique structure could availablely buffer the volume variation of MoS<sub>2</sub> during repeated charging-discharging processes, showing good cycling stability. The electron diffraction (SAED) pattern of the MoS<sub>2</sub>/NC is shown in Fig. 1f, illustrating the polycrystalline nature of MoS<sub>2</sub>. Figure 4g–k show the EDS mapping images of the MoS<sub>2</sub>/NC. It was obvious that Mo, S, C,

and N elements were uniformly distributed in the selected region. Based on the characterization mentioned above, the nanosheet-like MoS<sub>2</sub>/N-doped carbon was successfully synthesized.

To investigate the electrochemical behavior of sodium storage in the sample MoS<sub>2</sub>/NC, the electrode coated with 70% active substance was made into a button battery, and a series of electrochemical tests were carried out, such as the CV curves of MoS<sub>2</sub>/NC cycling three times (Fig. 5a). During the first discharge, two strong peaks could be observed near 0.72 V and 0.36 V while one weak peak near 0.65 V and one strong peak at ca. 1.82 V were seen during the first anodic sweep. The reduction peak at 0.72V corresponded to the formation of Na<sub>x</sub>MoS<sub>2</sub> by the reaction between Na<sup>+</sup> and MoS<sub>2</sub> (reaction 1). Another reduction peak at 0.36 V was caused by the conversion reaction of Na<sub>x</sub>MoS<sub>2</sub> and Na<sup>+</sup> into Na<sub>2</sub>S and Mo (reaction 2), accompanied by the formation of solid electrolyte interphase (SEI) film.

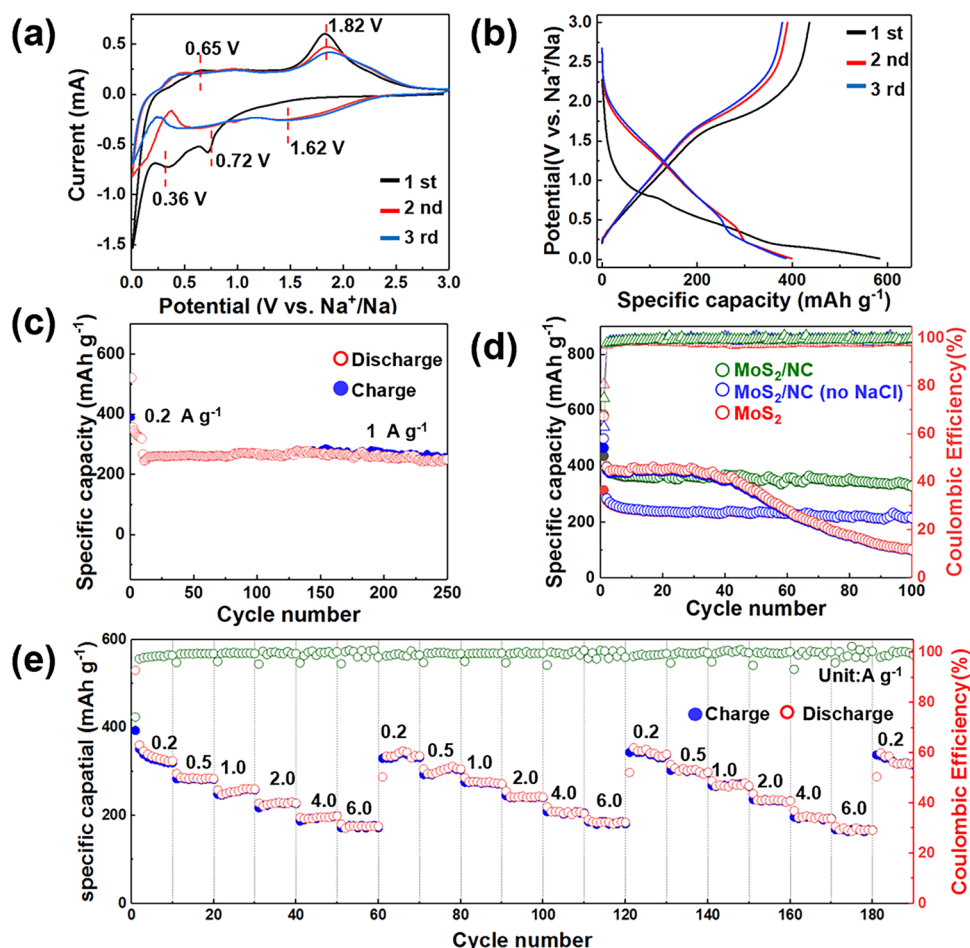


The weak oxidation peak at 0.65 V was determined by the recombination of Na<sub>2</sub>S with Mo into Na<sub>x</sub>MoS<sub>2</sub>, and the other oxidation peak at 1.82 V indicated the process of extracting



**Fig. 4** SEM images of **a** MoS<sub>2</sub> (no NaCl), **b–c** MoS<sub>2</sub>/CN. **d–e** HRTEM images of MoS<sub>2</sub>/CN composite at different magnifications. **f** SAED patterns of MoS<sub>2</sub>/CN. **g–k** EDS elemental mapping images of MoS<sub>2</sub>/CN

**Fig. 5** **a** CV curves of MoS<sub>2</sub>/NC electrode in the range of 0–3 V at a scanning rate of 0.2 mV s<sup>-1</sup>. **b** Charge–discharge curves of different cycles at 0.2 A g<sup>-1</sup>. **c** Cycling performance of MoS<sub>2</sub>/NC, MoS<sub>2</sub>/NC (no NaCl), and MoS<sub>2</sub> electrodes at 0.2 A g<sup>-1</sup>. **d** Long-term cycling performance at 1 A g<sup>-1</sup>. **e** Rate performance of MoS<sub>2</sub>/NC electrode



Na<sup>+</sup> from Na<sub>x</sub>MoS<sub>2</sub>. At the end of the first cycle, the original MoS<sub>2</sub> was partly transformed into a small amount of amorphous S and elemental Mo. In the second cathodic sweep, a new reduction peak appeared at 1.62 V, corresponding to the conversion of amorphous S into Na<sub>2</sub>S [41–44]. The reduction peak at 0.2–0.78 V remained unchanged but decreased intensity, indicating an irreversible phase transition occurred during the first cycle. In the subsequent few scans, the redox peaks overlapped highly, demonstrating that the MoS<sub>2</sub>/NC electrode had outstanding cycle capacity retention.

Figure 5b represents the galvanostatic charge–discharge curves of the MoS<sub>2</sub>/NC electrode at 0.2 A g<sup>-1</sup> in different cycles. The electrode's potential dropped sharply above 1.0 V and slowly below 1.0 V during the first cycling process, as seen from the figure, which might be due to the presence of carbon [45]. During discharge, two voltage platforms could be seen at 0–0.25 V and 0.5–0.75 V, while during charge, a voltage platform occurs at 1.75–2.0 V, which were primarily in accordance with the peak value observed in the CV curve [46]. Besides, the initial discharging and charging specific capacities of MoS<sub>2</sub>/NC electrode were 582.96 and 435.97 mAh g<sup>-1</sup>, respectively, giving a Coulombic efficiency of 74.75%. Other irreversible reactions, such as electrolyte

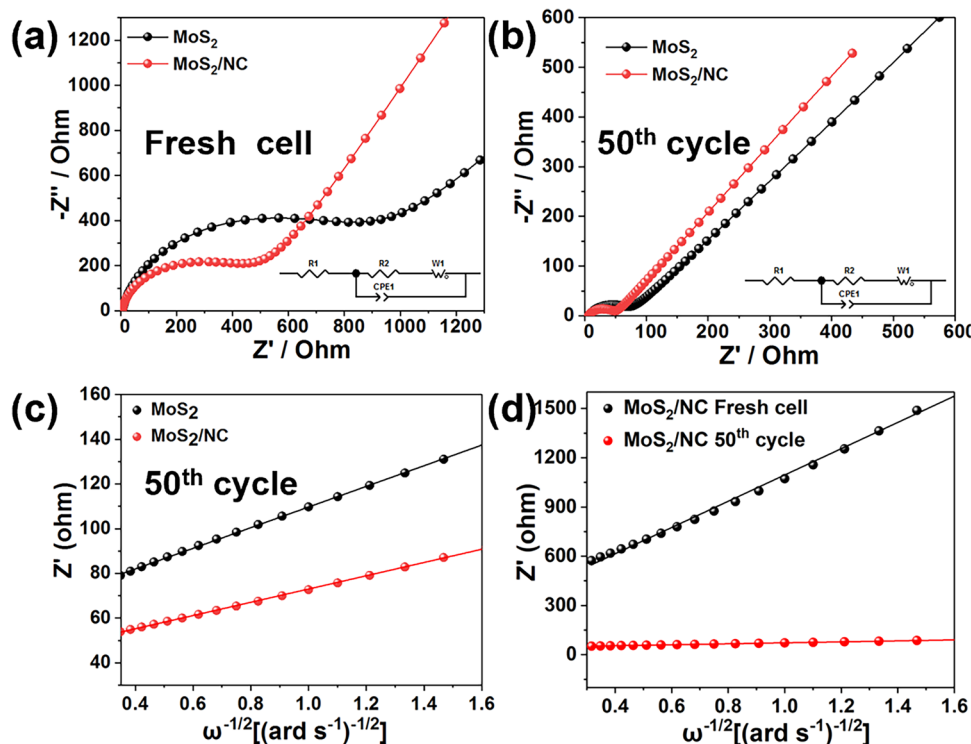
decomposition and SEI film formation, led to low initial Coulomb efficiency. The Coulomb efficiency of the electrode was above 98% in the subsequent cycles, indicating the formed SEI film was relatively stable [47–49]. Figure 5c describes the performance comparison of the electrodes of MoS<sub>2</sub>/NC, MoS<sub>2</sub>/NC (no NaCl) and MoS<sub>2</sub> for 100 cycles at 0.2 A g<sup>-1</sup>. The reversible specific capacities of the three samples after 100 cycles were 334.6, 213.2, and 100.8 mAh g<sup>-1</sup>, respectively, indicating that the specific capacity and cycling lifetime of MoS<sub>2</sub>/NC electrode was higher than MoS<sub>2</sub>/NC (no NaCl) and MoS<sub>2</sub>. Additionally, the capacity of the single MoS<sub>2</sub> electrode decreased rapidly after 50 cycles, showing poor electrochemical stability, mainly because of the poor conductivity of the material and the great volume variation during sodium-ion intercalation/de-intercalation. Compared with pure MoS<sub>2</sub>, the electrochemical performance of the MoS<sub>2</sub>/NC electrode had been improved, mainly due to the following two aspects. On the one hand, the introduction of N-doped carbon and the uniform distribution of nanosheets could enhance the conductivity of the material, alleviate the volume change during the cycle, increase the specific surface area, which improved the reaction kinetics, and thus further optimize the specific capacity [50, 51]. On

the other hand, the electrochemical reconstruction phenomenon could enhance the architecture of the electrode, showing more excellent cyclic stability [52]. Figure 5d describes the long-term cycling capability of the MoS<sub>2</sub>/NC electrode. The electrode could provide a reversible capacity of 252.9 mAh g<sup>-1</sup> at 1 A g<sup>-1</sup> after 250 cycles, demonstrating that the composite exhibits excellent long-term cycling behavior. Figure 5e describes the rate performance of the MoS<sub>2</sub>/NC electrode. At 0.2, 0.5, 1.0, 2.0, 4.0, and 6.0 A g<sup>-1</sup>, the reversible capacities were 351.3, 282.2, 258.6, 227.2, 199.67, and 172.31 mAh g<sup>-1</sup>, respectively. The reversible capacity could quickly recover to 338.2 mAh g<sup>-1</sup> after three large cycles when the current density returned to 0.2 A g<sup>-1</sup>, indicating that the MoS<sub>2</sub>/NC electrode had superior rate performance.

As one of the effective methods to study SIBs, electrochemical impedance spectroscopy has been conducted to investigate the electrochemical dynamics differences of MoS<sub>2</sub>/NC and MoS<sub>2</sub> electrodes. The Nyquist plots were fitted from the equivalent circuit in Fig. 6a and b. In the equivalent circuit, R<sub>1</sub> was the internal resistance between electrode and electrolyte, CEP was the abbreviation of constant phase element, R<sub>2</sub> was charge transfer resistance, and Z<sub>w</sub> was ascribed to the diffusion rate of sodium ions. As we all know, the Nyquist diagram consists of a semicircle and a diagonal line. The semicircle originated from the charge transfer resistance of the electrode, while the straight line was closely attributed to the impedance of the sodium-ion diffusion process. Table S1 lists the equivalent circuit parameters of different electrodes. The charge

transfer resistance of the MoS<sub>2</sub>/NC electrode was significantly lower than that of the MoS<sub>2</sub> electrode before cycling, showing faster reaction kinetics (Fig. 6a). After 50 cycles, the charge transfer resistance of the MoS<sub>2</sub>/NC electrode was still less than that of MoS<sub>2</sub> (Fig. 6b). Due to the coupling of nitrogen-doped carbon with MoS<sub>2</sub> and the uniform distribution of the nanosheets making the electrolyte to be more easily permeated into the electrode, which improved the reaction kinetics, finally leading to a lower R<sub>2</sub> on the macroscale. In the low-frequency region, Fig. 6c and d show the Nyquist plots of the real part (Z') of the two groups of samples versus ω<sup>-1/2</sup>. Among them, the Warburg coefficient was inversely proportional to the diffusion coefficient of sodium ions, which could be determined by the slope of the Z' and ω<sup>-1/2</sup> lines [53]. That was, the smaller the slope of the fitted line was, the larger the diffusion coefficient of sodium ion was, and the better the kinetic property of the electrode was. Calculated by fitting the slope of the curve, the diffusion coefficient of the MoS<sub>2</sub>/NC electrode was 5.31 × 10<sup>-15</sup> cm<sup>2</sup> s<sup>-1</sup> after 50 cycles, which was higher than that of the pure MoS<sub>2</sub> electrode (7.29 × 10<sup>-18</sup> cm<sup>2</sup> s<sup>-1</sup>), indicating that MoS<sub>2</sub>/NC could promote the transmission and diffusion of sodium ions, showing better electrochemical performance. The R<sub>2</sub> of both electrodes was decreased after 50 cycles, and compared with before cycling (2.19 × 10<sup>-15</sup> cm<sup>2</sup> s<sup>-1</sup>), the diffusion coefficient of the MoS<sub>2</sub>/NC electrode after cycling was still higher (Fig. 6d), which was because of the crystal structure transformation of MoS<sub>2</sub> during cycling, which was consistent with those reported previously [54].

**Fig. 6** Nyquist plots of the MoS<sub>2</sub> and MoS<sub>2</sub>/NC electrodes **a** before cycling, **b** after 50 cycles. **c** The real parts of the MoS<sub>2</sub> and MoS<sub>2</sub>/NC electrodes of the complex impedance versus ω<sup>-1/2</sup> before cycling. **d** The real parts of the MoS<sub>2</sub>/NC electrodes of the complex impedance versus ω<sup>-1/2</sup> before cycling and after 50 cycles



To further investigate the cyclic kinetics of the MoS<sub>2</sub>/NC electrode, CV tests were performed at the scanning rates of 0.2, 0.4, 0.6, 0.8, and 1.0 mV s<sup>-1</sup> (Fig. 7a). These curves were similar in shape as could be seen from the figure, but with the increase in scanning rate, the values of peak currents increase. The capacitive process and diffusion behavior of the sodium storage mechanism could be qualitatively analyzed by the following Eqs. (3, 4):

$$i = av^b (0.5 \leq b \leq 1) \quad (3)$$

$$\log(i) = b \log(v) + \log(a) \quad (4)$$

where  $a$  and  $b$  were constants,  $i$  was identified as the current, and  $v$  represents the sweep speed. As we all know, the  $b$  value represented the type of reaction kinetics. When the  $b$  value approached 0.5, the reaction was mainly controlled by the diffusion behavior. When closer to 1, the storage of sodium ions was mainly dominated by the capacitive behavior [55, 56]. Linear fitting was performed on oxidation peaks 1, 2 and reduction peaks 3, 4 in Fig. 7b.  $b$  values of peaks 1, 2, 3, and 4 were 0.762, 0.936, 0.776, and 0.756, respectively. The results indicated that the sodium-ion storage kinetics of the MoS<sub>2</sub>/NC composite was dominated by the capacitance behavior. According to the Eq. (5):

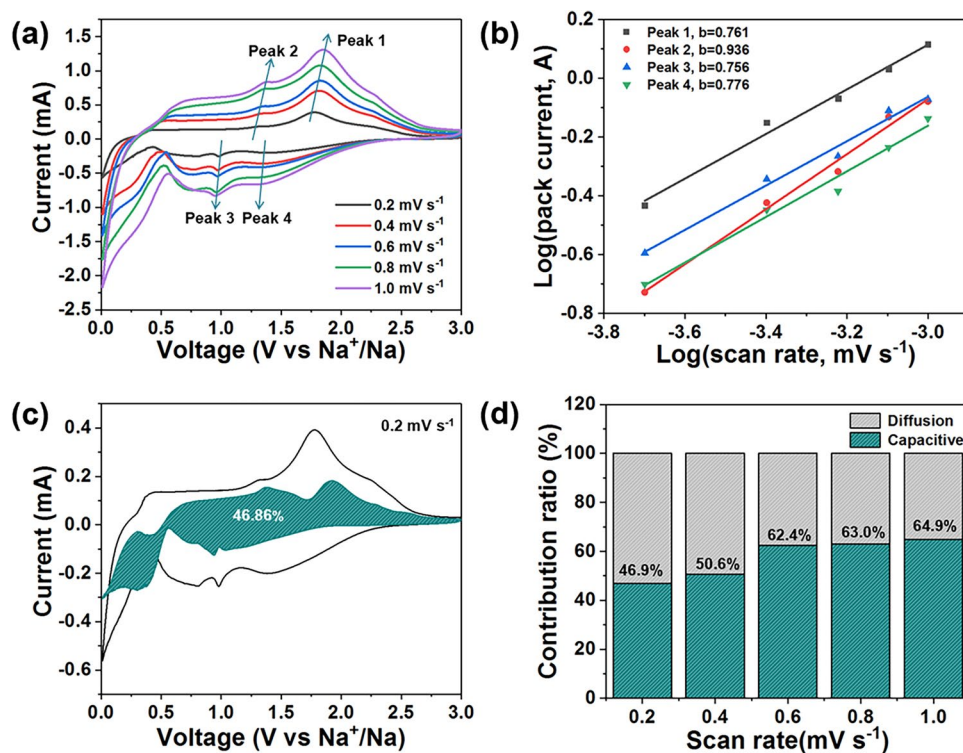
$$i = k_1 v + k_2 v^{1/2} \quad (5)$$

the capacitive contribution could be further calculated at different sweep rates, where  $k_1 v$  corresponded capacitive

process and  $k_2 v^{1/2}$  represented the diffusion process. Figure 7c and d describe the calculated results. When the scanning rate was 0.2 mV s<sup>-1</sup>, the contribution of the capacitive was 46.85%. It could be seen from the results that the contribution of capacitance improved with the increase in the sweep speed. When the scanning rate scaled up from 0.2 to 1.0 mV s<sup>-1</sup>, the ratios were 46.9%, 50.6%, 62.4%, 63.0%, and 64.9%, respectively. For comparison, the CV curves of pure MoS<sub>2</sub> and MoS<sub>2</sub>/NC (no NaCl) electrodes were measured at different scanning rates (Fig. S4). It was calculated that with the increase of scanning rate, the capacitance contributions of pure MoS<sub>2</sub> electrode were 48.7%, 52.9%, 55.1%, 57.2%, and 61.7%, respectively, and that of MoS<sub>2</sub>/NC (no NaCl) electrode were 57.8%, 59.8% and 61.7%, 62.2%, and 64.6%, respectively. The capacitive contributions of MoS<sub>2</sub>/NC (no NaCl) and MoS<sub>2</sub>/NC electrodes were relatively higher than that of pure MoS<sub>2</sub> electrodes, indicating that N-doped carbon could improve the capacitive contribution and promote sodium-ion transfer.

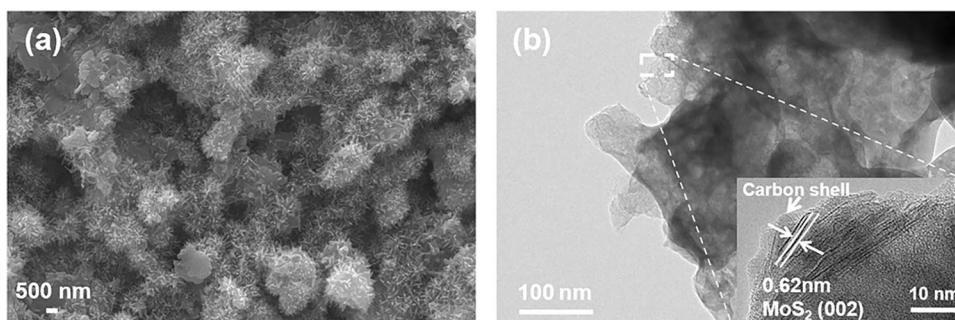
To further study the reasons for the excellent electrochemical performance of the MoS<sub>2</sub>/CN electrode, the morphological characteristics of the MoS<sub>2</sub>/CN electrode were analyzed after 100 cycles at a specific current density. It could be seen that the size of the MoS<sub>2</sub> nanosheets in the electrode after cycling was reduced, and the irregular rectangular bulges were attached to the surface of the nanosheets (Fig. 8a). This morphology could also be observed in TEM, where the size of the rectangular bulges was about 60–80 nm as shown in Fig. 8b. Additionally, clear lattice fringes

**Fig. 7** **a** CV curves of MoS<sub>2</sub>/CN electrode at different scanning rates. **b** The relationship between  $\log(i)$  and  $\log(v)$ . **c** Capacitive contribution in the CV curve at a scan rate of 0.2 mV s<sup>-1</sup> (shaded region). **d** Ratio of capacitive contributions at different scanning rates





**Fig. 8** **a** SEM, **b** HRTEM images of MoS<sub>2</sub>/CN electrode after 100 cycles



could be observed on the HRTEM image, where the 0.62 nm interplanar spacing corresponded to the (002) face of MoS<sub>2</sub>, which might be due to the electrochemical reconstruction phenomenon of the MoS<sub>2</sub>/CN electrode during the cycle, that was, the reaction kinetics changed because of the evolution of the microstructure [54, 57]. With the reduction of the size of MoS<sub>2</sub> nanosheets and the formation of irregular rectangular bulges, the contact between the electrolyte and the electrode enhanced, thus exhibiting better electrochemical reaction kinetics, which was consistent with measurements of the EIS. This extraordinary morphological change might be the reason for the good stability of the MoS<sub>2</sub>/CN electrode.

## Conclusion

In summary, the MoS<sub>2</sub>/CN nanosheets were prepared using a facile salt template–assisted synthesis method, in which the MoS<sub>2</sub> nanosheets were uniformly embedded in the nitrogen-doped carbon framework without severe agglomeration. The as-obtained MoS<sub>2</sub>/NC nanosheets exhibited outstanding electrochemical properties (334.6 mA h g<sup>-1</sup> at 0.2 A g<sup>-1</sup> after 100 cycles), indicating that the MoS<sub>2</sub>/CN nanosheets provided more excellent sodium storage properties, compared with pure MoS<sub>2</sub>. The improved electrochemical performance could be caused by the coupling effect of N-doped carbon with MoS<sub>2</sub>, which offered improved electrical/ionic transporting capability, and the electrochemical reconstruction, which guaranteed electrode stability. The electrode had the advantages of a simple synthesis method, low cost, and suitable for large-scale production, endowed MoS<sub>2</sub>/NC potential prospect in energy conversion and storage.

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**Data availability** The data presented in this paper are available on request from the corresponding author.

## Declarations

**Ethics approval** This work did not include any studies involving humans or animals.

**Competing interests** The authors declare no competing interests.

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