Hierarchical Carbon Nanosheet Assembly with SiO$_x$ Incorporation and Nitrogen Doping Achieves Enhanced Lithium Ion Storage Performance

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Lithium ion batteries (LIBs) have dominated the markets of portable electronics due to the merits of a low self-discharge rate, high voltage platform, environmental friendliness, and portability. However, the limited theoretical capacity of the current commercial anode material causes unsatisfied energy density of LIBs, which falls behind the ever-increasing demands of society. Herein, a hierarchical porous carbon nanosheet assembly is successfully constructed with simultaneous SiO$_x$ incorporation and nitrogen doping (denoted as HPCNA-(N, Si)) through a supramolecular assembly–based one-pot strategy followed by a calcination process. Benefitting from the unique morphology, highly porous feature, and the synergy of SiO$_x$ incorporation and nitrogen doping, the HPCNA-(N, Si) exhibits largely enhanced Li$^+$ storage performance when evaluated as anode material for LIBs. Specifically, it can deliver a high specific capacity of 583.0 mA h g$^{-1}$ at 500 mA g$^{-1}$ with a stable cycling capability (700 cycles with an average attenuation rate of 0.32% at 1000 mA g$^{-1}$). The possible origins of the promising Li$^+$ storage behavior for HPCNA-(N, Si) are unraveled based on the cyclic voltammetry (CV) curves, where a fair capacitive contribution of 63.6% at 0.9 mV s$^{-1}$ could imply fast ion transfer kinetics for superior rate and cycling performance.

1. Introduction

Lithium ion batteries (LIBs) have been considered as the most successful energy storage devices that have already conquered the markets of portable electronics, such as cell phones, laptops, and digital cameras, due to the merits of a low self-discharge rate, high voltage platform, environmental friendliness, and portability.[1] Moreover, they have also been deemed as one of the most promising power source candidates for electric vehicles and stationary energy storage.[2] However, the ever-increasing demands for high capacity and high energy storage have raised urgent challenges for LIBs due to the limited theoretical capacity of the current commercial graphite anode (372 mA h g$^{-1}$).[3,4] Therefore, plenty of efforts have been dedicated to seek a new substitution with higher specific capacity and outstanding cycling performance as anode material for next-generation LIBs aiming to meet the fast increasing demands on power/energy density of the devices.

Among various promising anode materials proposed for LIBs, such as intercalation reaction–based Li$_x$Ti$_2$O$_12$ and graphite, alloying type Sn, Sb, and Ge, as well as the conversion-type transition metal oxides/compounds,[5–8] carbon materials have received considerable attention as one of the most promising candidates due to the inherent merits of abundant resources, favorable electronic/ionic conductivity, and superior physical/chemical stability.[9–13] However, the unfavorable specific capacity of carbon materials is tending to fall behind the increasing demand of high-energy LIBs. It has been well documented that advanced micro/nanostructure engineering can greatly enhance the Li$^+$ storage capability by shortening the ion diffusion path and providing extra exposed active sites, and heteroatom doping can improve the Li$^+$ storage performance by tuning the electronic structure and promoting the diffusion kinetics.[14,15] For example, Zhang and co-workers developed nitrogen-rich hollow carbon nanotubes via a template method using polypyrrole as the high-nitrogen-containing carbon precursor, which exhibited long-term cycling and a high rate property for Li$^+$ storage.[16] Tai and co-workers prepared nitrogen/oxygen codoped graphene-like carbon nanocages, which delivered favorable electrolyte penetration and improved the kinetics for ion and electron transport, resulting in extraordinary electrochemical performance.[17] Although these are encouraging achievements, there are still some remaining challenging tasks. First, the preparation of a carbon-based hierarchical nanostructure generally involves complicated conditions and is time consuming, which means a more convenient strategy is advantageous yet
challenging for practical low-cost production. Second, the synergy of facile incorporation of electrochemical active SiO\textsubscript{x} incorporation and nitrogen doping in a hierarchical porous carbon nanosheet assembly (denoted as HPCNA-(N, Si)) through a supramolecular assembly–based one-pot strategy followed by a calcination process, which exhibits enhanced Li\textsuperscript{+} storage performance as anode material. Specifically, the HPCNA-(N, Si) can deliver a specific capacity of 583.0 mAh g\textsuperscript{-1} at 500 mA g\textsuperscript{-1} with 100% capacity retention after 100 cycles. In addition, a high specific capacity of 524.2 mAh g\textsuperscript{-1} at 1000 mA g\textsuperscript{-1} can be achieved with a small capacity fading rate of 0.32% per cycle, showing superior cycling stability. The Li\textsuperscript{+} storage kinetics is also analyzed based on the cyclic voltammetry (CV) curves, which display a capacitive contribution of 63.6% at 0.9 mV s\textsuperscript{-1}, well proving the superior rate and cycling performance. This work not only provides a feasible strategy for an advanced carbon nanostructure, but also pushes forward the development of anode candidates for next-generation high-energy LIBs.

### 2. Results and Discussion

The synthesis route of HPCNA-(N, Si) is schematically shown in [Scheme 1](#), which can be briefly described as follows. Typically, 4 mmol of melamine (M) is dissolved in dimethyl sulfoxide (DMSO) to form solution A, and 4 mmol cyanuric acid (CA) and a certain amount of tetraethyl orthosilicate (TEOS) are dissolved in DMSO to form homogeneous solution B. After that, the as-prepared two solutions are mixed together under vigorous stirring to obtain the precursor (denoted as Si-MCA) through a supramolecular assembly strategy. Then, the HPCNA-(N, Si) hybrid nanostructures can be readily obtained by facile annealing treatment at 800 °C under a H\textsubscript{2}/Ar (10/90%) atmosphere.

The morphology and microstructure of the HPCNA-(N, Si) particle were investigated by field-emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM) characterizations. As displayed in [Figure 1A,B](#), the FESEM and TEM images reveal that the Si-MCA precursor exhibits a flower-like morphology with an average diameter of 1–2 μm, which is composed of numerous nanosheets assembly. The overall morphology can be well maintained after thermal annealing treatment except for the shrinkage in thickness of the nanosheets (<50 nm), as shown in [Figure 1C–E](#). The enlarged view of the TEM image ([Figure 1F](#)) confirms the reduction of the nanosheet thickness after calcination. Note, the highly porous structure can be also observed, which could be ascribed to the release of gaseous products by the decomposition of organic groups and the elimination of the generated small molecules during the calcination process. The porous feature of HPCNA-(N, Si) can be further proved by nitrogen absorption/desorption measurement ([Figure S1A](#), Supporting Information), where type IV isotherms can be observed and a high Brunauer–Emmett–Teller (BET) surface area of 706.6 m\textsuperscript{2} g\textsuperscript{-1} can be obtained.\textsuperscript{[18]} The corresponding pore size distribution ([Figure S1B](#), Supporting Information) confirms the porous feature of the product with sizes ranging from 2 to 50 nm. The high specific surface area and the highly porous structure are beneficial to contact between the electrolyte and electrode, and the mass transportation of Li\textsuperscript{+} during the charge/discharge process.\textsuperscript{[19]} Moreover, it is noteworthy to mention that the SiO\textsubscript{x} particle can be hardly distinguished, which is in line with the powder X-ray diffraction (XRD) results ([Figure S2](#), Supporting Information), implying the amorphous feature. As displayed in [Figure 1G](#), high-resolution TEM (HRTEM) analysis reveals that the HPCNA-(N, Si) is composed of twisted nanosheets that feature a defective characteristic with numerous amorphous microdomains. The defective characteristic is further demonstrated by the Raman spectrum ([Figure S3](#), Supporting Information), where both the defect-induced D band (1351.2 cm\textsuperscript{-1}) and graphitic-induced G band (1564.3 cm\textsuperscript{-1}) can be observed. The I\textsubscript{D}/I\textsubscript{G} ratio can be used as a descriptor to illustrate the degree of disorder for carbon materials, which is 1.05 for HPCNA-(N, Si).\textsuperscript{[20]} suggesting the defect-rich characteristic that is coincident with the HRTEM results. The I\textsubscript{D}/I\textsubscript{G} ratio further increases after SiO\textsubscript{x} incorporation, suggesting the generation of more defects. Note, the defects can not only function as active sites for Li\textsuperscript{+} storage, but also facilitate the mass transfer in the HPCNA-(N, Si).\textsuperscript{[21]} As displayed in [Figure 1H](#), the elemental mapping results manifest the uniform distribution of C, N, Si, and O elements throughout the particle. The weight ratio of SiO\textsubscript{x} in HPCNA-(N, Si) is calculated to be \approx4.0% ([Figure S4](#), Supporting Information), which is in line with the EDS results ([Figure S5](#), Supporting Information).

X-ray photoelectron spectroscopy (XPS) measurements were conducted to further characterize the surface composition and chemical state of HPCNA-(N, Si). [Figure S6](#), Supporting Information, shows the survey spectrum, in which the distinct peaks of C, N, Si, and O can be observed. [Figure 2A](#) displays the high-resolution XPS spectra of C 1s, which can be readily deconvoluted into C–Si (284.6 eV), C=C/C–C (284.7 eV), C=N/C–O (285.5 eV), and C=O (287.4 eV) bonds, respectively,\textsuperscript{[22,23]} indicating that the nitrogen has been successfully doped into the carbon skeleton. As shown in [Figure 2B](#), the

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**Scheme 1**. The illustration of the formation process of HPCNA-(N, Si).

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\[ \text{Si} \quad \text{TEOS} \quad \text{MCA} \quad \text{Calcination} \quad \text{HPCNA-(N, Si)} \]

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N 1s spectrum can be divided into pyridinic N (398.5 eV), pyrrolic N (400.9 eV) and graphitic N (403.3 eV). It has been well documented that the pyridinic N and pyrrolic N can function as electrochemical active sites for Li$^{+}$ storage, and the electron–donor characteristic of graphitic N is helpful for the enhancement of electronic conductivity of HPCNA-(N, Si). The O 1s spectrum was divided into four peaks located at 530.5, 531.6, 533.0, and 533.0 eV (Figure 2C), corresponding to the COOH bond, C─OH bond, C=O/O─Si bond, and Si─O─Si bond, respectively. The presence of oxygen can not only enhance the wettability to reduce the inert surface area, but also serve as electrochemical active sites. As shown in Figure 2D, the Si 2p spectrum can be deconvoluted into SiO$_2$C (101.8 eV), SiO$_2$C (102.7 eV) and SiO$_4$ (103.5 eV), respectively, suggesting that the Si could be coupled with carbon matrix through oxygen atoms.

The lithium ion storage performance of the HPCNA-(N, Si) anode was then investigated in a potential window of 0.01–3 V versus Li$^+$/Li in a half-cell configuration using lithium metal as the counter and reference electrode. As displayed in Figure 3A, the CV curves were collected at a scan rate of 0.1 mV s$^{-1}$. During the initial cathodic scan, a distinct irreversible peak can be observed at 0.50 V, which could be ascribed to the decomposition of the fluoroethylene carbonate (FEC) additive and electrolyte, further arousing the formation of a solid electrolyte interface (SEI) on the interface between the electrode and electrolyte. An evident peak emerged at 0.20 V extending to the cutoff voltage (0.01 V) is aroused by the intercalation of Li$^+$ into the carbon matrix and the reaction between SiO$_x$ and Li$^+$. During the first anodic scan, two recognizable peaks at 0.48 and 1.10 V can be assigned to the reversible stepwise delithiation processes. The overlapped following CV curves demonstrate good reversibility of the electrochemical processes for the HPCNA-(N, Si) anode. Figure 3B shows the charge/discharge profiles of HPCNA-(N, Si) at a rate of 100 mA g$^{-1}$. As shown, the HPCNA-(N, Si) electrode displayed a plateau centered at 0.62 V during the initial discharge process, which is in accordance with the CV results. With the end of the discharge process, the HPCNA-(N, Si) electrode delivered a discharge specific capacity of 853.2 mAh g$^{-1}$, obtaining an initial Coulombic efficiency (ICE) of 42.2%. The low initial CE issue could be caused by the formation of the SEI, which needs to be solved by prelithiation treatment if applied in full-cell devices. The CE gradually increased to 93.0% after 10 cycles, indicating the good reversibility of the HPCNA-(N, Si) electrode. In addition, the following profiles delivered a pseudo-linear voltage response, suggesting the possible fast ion storage kinetics. As shown in Figure 3C,
Figure 2. The XPS analysis of surface chemical state of HPCNA-(N, Si). High-resolution spectra of A) C 1s, B) N 1s, C) O 1s, and D) Si 2P, respectively.

Figure 3. Lithium ion storage performance of HPCNA-(N, Si) anode. A) CV curves at 0.1 mV s⁻¹, B) charge/discharge profiles at 100 mA g⁻¹, C) cycling stability at 500 mA g⁻¹, D) rate capability, and E) cycling stability at 1000 mA g⁻¹.
the cycling performance of the HPCNA-(N, Si) with different amounts of TEOS was compared. As indicated, the HPCNA-(N, Si) with 180 μL of TEOS (denoted as HPCNA-(N, Si)180) delivers a stable cycling performance and exhibits 100% capacity retention rate after 100 cycles at 100 mA g\(^{-1}\) (625.2 mAh g\(^{-1}\) at 100th cycle). As for the control sample of NC without SiO\(_x\) incorporation, although the cycling performance is quite stable, the reversible specific capacity is only \(\approx 291.9\) mAh g\(^{-1}\), much lower than that of HPCNA-(N, Si), revealing the significance of the SiO\(_x\) incorporation for storage capacity. Figure 3D displays the rate performance of NC and HPCNA-(N, Si) with different amounts of TEOS, where the HPCNA-(N, Si)180 shows enhanced performance with a capacity retention of 30.7% upon the current density rising from 100 to 5000 mA g\(^{-1}\) (277.8 mAh g\(^{-1}\) at 500 mA g\(^{-1}\) vs 904.6 mAh g\(^{-1}\) at 100 mA g\(^{-1}\) ), whereas the NC can only deliver a reversible specific capacity of 184.8 mAh g\(^{-1}\) at 5000 mA g\(^{-1}\). As shown in Figure 3E, the long-term cycling performance of HPCNA-(N, Si) with different amounts of TEOS at 1000 mA g\(^{-1}\) was also compared. Remarkably, the HPCNA-(N, Si)180 showed a reversible specific capacity of 524.2 mAh g\(^{-1}\) and stable cycling performance with a small fading rate of 0.32% per cycle (404.9 mAh g\(^{-1}\) at 700th cycle vs 524.2 mAh g\(^{-1}\) at 10th cycle), which was outperforming all the samples with various amounts of TEOS. As indicated earlier, the incorporation of SiO\(_x\) can efficiently improve the Li\(^+\) storage performance of HPCNA-(N, Si). However, as displayed in Figure S7, Supporting Information, the morphology becomes more degraded when the amount of TEOS is increased to 240 μL, which is in line with the deteriorated Li\(^+\) storage performance of HPCNA-(N, Si)1240.

The Li\(^+\) storage kinetics was then analyzed based on the CV curves at various scan rates, as displayed in Figure 4A, to reveal the internal relationship between the capacitive contribution and superior rate capability of the HPCNA-(N, Si)\(_{180}\) electrode. Based on the power relationship between current response \(i\) and scan rates \(v\) raised by Dunn and co-workers,\(^{[34]}\) as listed in Equation (1), the total stored charge of the electrode can be readily separated into a diffusion-controlled region and pseudo-capacitive contribution.

\[
i = av^b
\]  

(1)

where \(a\) and \(b\) are defined as adjustable constants. The \(b\) value can be easily calculated from the slope of the linear relationship of \(\log(i)\) versus \(\log(v)\), which is a parameter to describe the Li\(^+\) storage behavior qualitatively. Generally, it is a diffusion-controlled process when the \(b\) value reaches 0.5, whereas a capacitive-controlled process can be obtained when the \(b\) value is near 1.0.\(^{[15]}\) As shown in Figure 4B, the \(b\) values are calculated to be 0.87/0.62 for cathodic/anodic peaks, suggesting that the total capacity is composed of both a capacitive-controlled process and a diffusion-controlled process at the same time. After that, the capacitive contribution of the total capacity at various scan rates can be readily obtained based on Equation (2), which is deformed from Equation (1).

\[
i = k_1v + k_2v^{1/2}
\]  

(2)

where \(k_1\) and \(k_2\) are constants; \(k_1v\) and \(k_2v^{1/2}\) represent the capacitive-controlled process induced current and diffusion-controlled process induced current, respectively. Remarkably, the HPCNA-(N, Si)\(_{180}\) electrode delivered a capacitive contribution of 63.6% to the total stored charge at the scan rate of 0.9 mV s\(^{-1}\) (Figure 4C). The capacitive contributions at various scan rates are shown in Figure 4D.
scan rates are also calculated in Figure 4D, where the value gradually increases upon the increment of scan rates.

The enhanced electrochemical performance of HPCNA-(N, Si) hybrid nanostructures could be explained by the hierarchical morphology and the synergy of SiO𝑥 incorporation and nitrogen doping. First, the high specific surface area and the hierarchical porous structure originated from the hierarchical morphology are conductive to contact between the electrolyte and electrode, and the mass transportation of Li⁺ during the charge/discharge process, which is beneficial for the enhancement of the Li⁺ storage performance. Second, the incorporation of SiO𝑥 can not only generate extra defects, which is advantageous for Li⁺ storage and mass transfer,[21] but also function as active sites to store Li⁻. As for the nitrogen species doped in HPCNA-(N, Si)₁₈₀, it is also conductive to Li⁺ storage.[2₂⁻₂₆] Thus, the HPCNA-(N, Si)₁₈₀ delivers boosted reversible specific capacity and cycling performance after a small amount SiO𝑥 incorporation when compared with NC (625.2 mAh g⁻¹ for HPCNA-(N, Si)₁₈₀ vs 291.9 mAh g⁻¹ for NC after 100 cycles at 0.5 A g⁻¹), signifying the importance of synergy of SiO𝑥 incorporation and nitrogen doping. Moreover, the structural robustness evidenced by the SEM images of the cycled sample (Figure S8, Supporting Information) could be further beneficial to the superior rate and cycling performance.

3. Conclusion

In summary, we have successfully developed a supramolecular assembly protocol based one-pot strategy followed by a calcination process for the fabrication of a hierarchical porous carbon nanosheet assembly with simultaneous SiO𝑥 incorporation and nitrogen doping. When evaluated as an anode, it shows enhanced Li⁺ storage capability with high specific capacity, outstanding cycling stability, and rate performance, which is aroused by the hierarchical morphology and the synergy of SiO𝑥 incorporation and nitrogen doping. In addition, CV measurements were also conducted to explore the underlying Li⁺ storage mechanism and the possible origins of the superior rate and cycling performance. This work could not only contribute to the advancement of the carbon-based anode candidates for next-generation LIBs, but also provide a scalable yet low-cost synthesis protocol for the construction of hybrid nanostructures.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

anodes, carbon, lithium ion batteries, nitrogen doping, porous structure

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