SHORT COMMUNICATION

Discarded COVID-19 masks-derived-doped porous carbon for lithium-sulfur batteries

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Summary
Despite the high theoretical capacity and energy density of lithium-sulfur (Li-S) batteries, the development of Li-S batteries has been slow due to the poor electrical conductivity and the shuttle effect of the electrode materials, resulting in low sulfur utilization and fast long-term cycling capacity decay. The modified carbon materials are often used as sulfur hosts to significantly improve the cycling performance of the materials, but also bring high-cost issues. Here, the porous carbon materials are synthesized quickly and conveniently by the microwave cross-linking method using discarded medical masks as carbon sources and concentrated sulfuric acid as solvent. However, poor surface and structural properties limit the application of materials. The porous carbon material is modified with p-toluene disulfide and urea as the sulfur and nitrogen sources by the microwave cross-linking method, which not only improves the porosity and specific surface area of the porous carbon material, but also improved the electrical conductivity and interlayer spacing of the material. As synthesized SN-doped porous carbon is employed as the sulfur host, which exhibits a high discharge capacity (1349.3 mAh g\(^{-1}\)) at 0.1°C, the S-porous C/S, N-porous C/S, and SN-porous C/S can maintain 78.1, 43.9, and 59.5% of the initial capacity after 500 cycles. The results indicate that the doping of S and N atoms provides sufficient active sites for the chemisorbed lithium polysulfides (LiPSs) to improve the reaction kinetics of the materials.

KEYWORDS
discharge capacity, Li-S batteries, medical masks, porous carbon

1 INTRODUCTION

With the outbreak of COVID-19 and the arrival of work resumption, the global demand for masks has increased dramatically, the World Health Organization reports that the current global demand for masks is 100 times higher than the normal level, and the daily demand is about 2 billion pieces per day. In 2021, Danish researchers found that 3 million masks are discarded every minute globally, which means 129 billion masks are discarded every month globally.\(^1\) According to research statistics, 193 countries have generated about 8.4 million tons of medical plastic waste since the outbreak.\(^2\) Discarded masks and plastic gloves have either entered the ocean or clogged and fills, not only polluting the environment, but also threatening the lives and health of animals and humans. Then the disposal of a massive number of...
discarded masks has become a very tricky problem. The current chemical methods for recycling plastics are mainly through pyrolysis. However, the direct pyrolysis results in low added value due to the wide distribution of the product. In addition, the content of polyethylene terephthalate in discarded medical masks is close to 16 wt.%, which also does not meet requirements of companies for pyrolysis raw materials. Therefore, there is an urgent need for an appropriate method to solve the discarded medical masks pollution while achieving higher returns.

The percentage of polypropylene in medical masks is about 79.37%, and the carbon content in polypropylene is as high as 85.7%, which is a good carbon source. Carbon materials with low price, stable physical/chemical properties, high specific surface area, and rich pore structure can be used as space encapsulants, which can provide the volume space required for the conversion of sulfur into lithium sulfide. Therefore, porous carbon materials are considered an ideal sulfur host for physically trapping polysulfides. However, physical trapping mainly relies on weak van der Waals interactions between non-polar carbon and highly polar polysulfides, but the forces between pure carbon materials and polysulfides are relatively weak. Therefore, the interaction and immobilization ability of polysulfides can be enhanced by introducing heteroatoms into carbon materials or compounding them with metal oxides. Nitrogen is a typical heteroatom, and doping carbon materials with nitrogen atoms can increase their electrical conductivity and the adsorption capacity of polar substances on the carbon surface. In addition, the size of sulfur is larger than that of nitrogen, but its electronegativity is smaller, and its doping can effectively serve as a redox site for Li-S batteries reactions during cycling, thereby further enhancing its electrochemical performance. In these respects, the co-doping of nitrogen and sulfur is expected to contribute to improving the conductivity and enlarge the interlayer distance. It is reported that a large number of current works mainly use chemicals (dicyandiamide, sucrose, polyacrylonitrile, critic acid and urea) as carbon sources to study the effect of nitrogen and sulfur heteroatom doping on the electrochemical performance of electrode materials. In contrast, the study of electrode materials for Li-S batteries using nitrogen and sulfur heteroatoms co-doped with waste material as the carbon sources has rarely been reported.

Herein, we report an efficient and green microwave cross-linking method for the recovery of discarded medical masks to obtain S-doped porous carbon, N-doped porous carbon and SN-doped porous carbon (S-porous C, N-porous C, and SN-porous C). The energy storage performance of S-porous C, N-porous C, and SN-porous C in Li-S batteries are also investigated. The use of these materials as the host material significantly improves the charge-discharge capacity of Li-S batteries. The SN-porous C electrode exhibits a high discharge capacity (1349.3 mAh g⁻¹) at 0.1°C. The discharge capacity of SN-porous C is improved compared to S-porous C and N-porous C. This is mainly due to the fact that the introduction of S and N heteroatoms improves the electrical conductivity and interlayer distance of the material, which promotes the kinetics of the reaction of carbon materials with LiPSs.

2 MATERIALS AND METHODS

2.1 Synthesis of nitrogen-doped porous C

Medical mask-based porous carbon materials are synthesized by the microwave cross-linking method. A piece of discarded medical mask is sterilized and cut into rectangles of size 2 mm². Three gram of medical mask pieces are placed in a reaction bottle containing 60 mL of concentrated sulfuric acid, followed by urea of equal mass with the masks into the sulfuric acid solution. The reaction bottle is placed in a modified microwave reaction chamber and reacted at 500 W for 8 minutes. After cooling, it is washed to neutrality with deionized water and dried in an oven at 50°C for 12 hours. After the carbon material is completely infiltrated, the mixed material is sintered at 600°C for 2 hours in an N₂ atmosphere. When the sample is cooled and washed to neutrality to obtain N-porous C.

2.2 Synthesis of S-porous C and SN-porous C

The preparation process of S and SN co-doped porous C is the same as that of N-porous C. The urea is replaced with p-toluene disulfide and a mixture of p-toluene disulfide and urea in the above experimental procedure, named S-porous C and SN-porous C.

2.3 Synthesis of composite electrode material

The SN-porous C is mixed with sulfur in a mass ratio of 1:3. The mixture is ball-milled at 600 r/min for...
30 minutes, then mixed with CS₂ solution and stirred at room temperature until a gel is formed. The resulting mixture is kept at 155°C for 8 hours and then heated at 200°C for 2 hours in an inert atmosphere to completely remove the volatile sulfur on the surface of the porous carbon material. The products are collected to produce SN-porous C/S powder. The N-porous C/S and S-porous C/S are prepared according to the same procedures, with the difference that SN-porous C is substituted by N-porous C and S-porous C, respectively. The schematic diagram of the preparation of the SN-porous C/S electrode material is shown in Figure 1.

2.4 Structure and electrochemical characterization

The x-ray diffraction patterns of the sample are recorded on an x-ray diffractometer (Rigaku Ultima IV, Japan) with Cu Kα radiation (1.54 Å) in the range of 2θ = 10 to 80° and at 40 kV and 30 mA. The morphologies of the samples are analyzed by scanning electron microscopy (SEM, Phenom Pro X, Netherlands/FEI). The TG/DSC data analysis of samples is performed using a simultaneous thermogravimetric analyzer (STA449F3 Jupiter, Netzsch, Germany) in N₂ from 30 to 1000°C in 5°C/min steps. The chemical composition and chemical valence of the samples are determined by an XPS test system (Thermo Scientific K-Alpha, United States) with Al Kα radiation. The specific surface area and porosity analysis of the samples are calculated according to the BET method and the surface and porosity analyzer (BET, Mack 2460, United States). The electrode materials are assembled on a CR2025 coin cell for electrochemical performance testing. The preparation of positive electrode materials, the SN-porous C/S powder, carbon black, conductive agent (super-p), and polyvinylidene fluoride (PVDF) are mixed in a mass ratio of 80:10:5:5, and the mixture is uniformly mixed by ball milling at 600 r/min for 30 minutes, and the mixture is added with an appropriate amount of NMP and stirred for 4 hours to obtain a viscous slurry, which is coated on Al foil by a coating machine and hold for 12 hours at 80°C. The Celgard 2400 membrane is used as a separator to separate electrons, and the lithium foil is used as a counter and reference electrode. The electrolyte is a mixed solution of 1.0 M lithium bis(trifluoromethane)sulfonamide, 5.0% anhydrous LiNO₃ and 1,3-dioxolane and ethylene dimethyl alcohol (1:1 by volume). The batteries are assembled in a glove box where the water vapor and oxygen content are below 1.0 ppm. The charge/discharge tests are performed using a battery test system (Xinwei, CT-4000).
3 | RESULTS AND DISCUSSION

3.1 | Structure of SN-doped porous C

The microstructures of S-porous C, N-porous C, and SN-porous C are explored by XRD, TG, Raman, and BET method. As shown in Figure 2A, all samples show two broad peaks at about 21.24° and 43.42°, referring to the (002) and (100) lattice planes of disordered carbon, respectively. The peak on the (002) plane of S-porous C, N-porous C, and SN-porous C is slightly shifted to a lower angle compared to the porous C samples, implying an increase in the interlayer distance d_{002} (the d_{002} of porous C, S-porous C, N-porous C, and SN-porous C are 0.24, 0.40, 0.44, and 0.46 nm, respectively). From the TGA analysis, it can be seen that S-porous C, N-porous C, and SN-porous C possess good thermal stability (Figure 2B, Figure S1A,B). Figure 2C shows the Raman spectra of S-porous C, N-porous C, and SN-porous C, the two separate bands appear at 1336.2 cm⁻¹ (D-band) and 1574.9 cm⁻¹ (G-band), corresponding to the disorder (or defect) and tangential vibrations of the carbon atoms, respectively. The ratio of the intensity between the D and G bands (I_D/I_G) indicates the crystallinity of the carbon material. The I_D/I_G values of porous C, S-porous C, N-porous C, and SN-porous C are 0.94, 0.99, 0.97, and 1.12, respectively. The I_D/I_G values of S-porous C, N-porous C, and SN-porous C increase compared with pure porous carbon, indicating that the introduction of S and N heteroatoms increases the defects of carbon materials. The specific surface area and pore size distribution of SN-porous C are shown in Figure 2D, Figure S2B,D. The rapid increase in nitrogen adsorption at relatively low pressures indicates the presence of abundant mesopores in N-porous C, S-porous C, and SN-porous C, and the isotherm of three samples mainly presents IV type. The specific surface area of the three samples increased from 3, 5 and 8 m² g⁻¹ to 1165.39, 1772.51, and 2083.81 m² g⁻¹ before and after etching by KOH (Figure 2A,C,E). The pore sizes of the three samples also changed from a very dispersed distribution to a more concentrated distribution. The changes in specific surface area and pore volume can be attributed to etch with KOH solution.

XPS was used to analyze the chemical composition and bonding configuration of S-porous C, N-porous C, and SN-porous C (Figure 3 and Figures S3-S5). The XPS spectra show that S-porous C, N-porous C, and SN-porous C are mainly composed of C, N, S and O elements. Figure 3A-D shows the high-resolution XPS spectra of S 2p, N 1s, C 1s, and O 1s of SN-porous C, where sulfur is bonded to carbon in two ways, including thiophene S (C-S-C) and oxidized S (C-SOx-C). The peaks located at approximate 166.8 and 168.3 eV correspond to thiophene S 2p3 and S 2p1, respectively. The thiophene sulfur plays an important role in enhancing the catalytic activity. The content of thiophene S in SN-porous C is higher than in pure S-porous C.
increased from 0.77% to 0.98% compared to S-porous C, while the content of oxidized S in the two samples was almost 1.72% and 1.67%, respectively, suggesting that the co-introduction of urea and p-toluene disulfide in SN-porous C facilitates the doping of S atoms into carbon materials more easily. The N 1s high-resolution spectra of SN-porous C and N-porous C showed three N types (Figure 3B and Figure S5B), pyridine nitrogen (402.80 eV), pyrrole nitrogen (404.38 eV), and graphite nitrogen (406.58 eV), respectively. The content of pyrrolic nitrogen is the same for both samples. The pyridine nitrogen and graphitic nitrogen in the N-porous C increased from 0.26% and 0.21% to 0.38% and 0.40%, indicating that the introduction of p-toluene disulfide resulted in a decrease in the amount of N atoms compared to SN-porous C. The high-resolution spectra of the C 1s peaks of the three samples (Figure 3C, Figure S4D and Figure 5D) show four spectral peaks, corresponding to C-C/C=O (287.88 eV), C=O/C-N (289.68 eV), C=O (291.38 eV), and C-O-C (292.38 eV), indicating the presence of N/S/O doping in the samples. As shown in Figure 3D, three typical peaks appeared at 536.38, 534.88 and 533.98 eV in the high-resolution spectrogram of O 1s, corresponding to the C-O, S=O, and C=O groups, respectively,25 the existence of C=O can improve the surface wettability of carbon materials,26 which can take full advantage of the high specific surface area and promote the conversion of LiPSs. A detailed comparison of Figure 3 and Figures S4 and S5 reveals that the introduction of S and N heteroatoms leads to a positive shift in the binding energy of C and O, which further verifies the successful synthesis of S and N atom-doped C materials. The doping of heteroatoms in porous carbon can effectively improve the active sites of the material, which is beneficial to the adsorption of polysulfides and helps to improve the cycling stability of the battery.27

### 3.2 Morphology of SN-doped porous C

The morphologies of the as-prepared S-porous C, N-porous C and SN-porous C are characterized by SEM as shown in Figure 4 and Figures S6 and S7. The low magnification SEM image shows that the three samples mainly present some irregular blocks (Figure 4A, Figures S6A and S7A). After further magnification, it is found that the surface of the SN-porous carbon block shows a hollow structure, and the block is composed of nanoparticles (Figure 4D). The morphologies of S-porous C are diverse, with lumps, sheets, and rods (Figure S6). The N-porous C presents an irregular morphology. It is found that these irregular blocks are composed of small pieces after further magnification. After the three samples were etched by KOH, some irregular and fluffy pore structures appeared on the surface of the samples.
FIGURE 4  The SEM images at different magnifications of the SN-porous C

FIGURE 5  The electrochemical performance of the S-porous C/S, N-porous C/S, and SN-porous C/S as Li-S batteries. (A) Nyquist plots of the S-porous C/S, N-porous C/S, and SN-porous C/S. (B) The CV profiles of SN-porous C/S at different scan rates. (C) The rate capabilities of S-porous C/S, N-porous C/S, and SN-porous C/S range from 0.1 to 5.0°C. (D) The charge/discharge curves of SN-porous C/S in the 1th, 20th, 100th, 300th, and 500th at 0.1°C. (E) The long-term cycling stability of the S-porous C/S, N-porous C/S, and SN-porous C/S at 0.5°C
which facilitated the penetration of the electrolyte in the Li-S batteries.

### 3.3 The electrochemical performance of SN-doped porous carbon

In order to explore the electrochemical performance of S-porous C, N-porous C, and SN-porous C, the EIS, CV, rate capability, charge-discharge curve, and stability of samples are analyzed. By comparing TG curves of S-porous C, N-porous C, and SN-porous C before and after loading S powder (Figure 2 and Figures S1 and S8), the results show loadings of S in S-porous C/S, N-porous C/S, and SN-porous C/S are 60.96%, 58.28%, and 60.02%, respectively. Figure 5A shows that the resistance of SN-porous C/S (1.32 and 30.11 Ω) in both low frequency and high frequency regions is smaller than that of S-porous C/S (2.65 and 58.61 Ω) and N-porous C/S (2.81 and 62.43 Ω), indicating that the electrochemical reaction kinetics of porous carbons are improved by the codoping of SN. Figure 5B and Figure S9A,C demonstrate the CV curves of S-porous C/S, N-porous C/S, and SN-porous C/S at scan rates of 0.1 to 0.4 mV s⁻¹. The anode and cathode peaks shift toward positive and negative potentials as the scan rate increases, respectively, which is mainly due to the polarization effect of the electrode material. The CV curves of the three samples display a similar shape. The CV curves show two typical reduction peaks that can be attributed to the reduction of S₈ to higher polysulfides (Li₂S₈, Li₂S₆ and Li₂S₄), following the reduction to lower insoluble Li₂S₂ and Li₂S, and an oxidation peak due to the inverse process. The rate performance of the three electrodes at different current densities is shown in Figure 5C, where SN-porous C/S shows the best performance at 0.1 to 2.0°C. When the current density was restored to 0.5°C, the capacities of the S-porous C/S, N-porous C/S, and SN-porous C/S electrodes recovered rapidly and remained stable in subsequent cycles. The charge/discharge curves of S-porous C/S, N-porous C/S and SN-porous C/S for the 1st, 20th, 100th, 300th and 500th cycles are shown in Figure 5D and Figure S9B,D. The discharge capacities of the three samples are 1223.7, 1138.2 and 1349.3 mAh g⁻¹ at 0.1°C, respectively. And their Coulombic efficiencies are 95.6%, 97.6% and 91.8%, respectively. The irreversible capacity loss in the first cycle may be due to the decomposition of the electrolyte and the formation of a solid electrolyte interfacial layer on the electrode. But after the first cycle, the Coulombic efficiency returns to 100%, which may be due to the introduction of S and N to provide more active sites for carbon materials. Figure 5E shows the cycling stability of S-porous C/S, N-porous C/S and SN-porous C/S at 0.5°C. The three electrode materials can maintain 78.1, 43.9, and 59.5% of the initial capacity after 500 cycles, respectively. The electrochemical test analysis revealed that the introduction of heteroatoms markedly improved the discharge capacity and Coulombic efficiency of the porous carbon material, I₁ is mainly related to the excellent porous structure and synergistic effect of heteroatoms.

![Figure 6](image_url)
To further analyze the kinetics and electrochemical reaction mechanisms of S-porous C/S, N-porous C/S, and SN-porous C/S, the CVs curves of different electrode materials are measured at different scan rates from 0.1 to 0.4 mV s\(^{-1}\) (Figure 6). The current density (i) and scan rate (v) present the following relationship (Equation 1).

\[
i = av^b
\]

where a and b are variable parameters, \(b = 1.0\) means that the capacity contribution is derived from the capacitive-controlled process, and \(b = 0.5\) means that the capacity contribution is derived from the ideal diffusion-controlled process.\(^3\) In order to obtain the b value, the logarithm of the different scan rates and current peak values was taken to obtain the \(\log(v) \text{-} \log(\text{ip}eak)\) linear fitting curve (Figure 6A-D), where b is the slope of the linear fitting curves. The b values for SN-porous C/S and S-porous C/S are 0.81 and 0.69, and the capacity of the surface material mainly originates from the capacitive-controlled process and ideal diffusion-controlled process. The slope of the linear fitting curve for N-porous C/S is 0.40 and the charge storage mainly originates from the ideal diffusion-controlled process. This may be mainly attributed to the relatively small specific surface area and interlayer distance, which are not conducive to the diffusion process of capacitance. This explains the higher discharge capacity of SN-porous C/S and S-porous C/S than N-porous C/S, which is in agreement with the experimental results.

4 | CONCLUSION

In summary, the porous carbon materials derived from disposable medical masks were obtained by microwave cross-linking and alkali etching using various heteroatoms sources as doping sources, which not only realized in situ doping of heteroatoms, but also gave the materials high porosity and a large specific surface area. And then electrochemical performance test results show that the N-porous C, S-porous C, and SN-porous C electrode materials provide high discharge capacities of 1138.2, 1223.7, and 1349.3 mAh g\(^{-1}\) at 0.1°C. The discharge capacity of the three samples decayed by 43.9%, 78.1%, and 59.5% at 0.5°C for 500 cycles compared to the initial capacity, respectively. The co-doping of S and N atoms can provide sufficient active sites for the chemisorbed LiPSs and thus increase the discharge capacity of SN-porous C compared to S or N atom doping. This work verifies the feasibility of adding different heterogeneous atomic sources during the microwave crosslinking process to improve the electrochemical performance of the recovered carbon materials, which can serve as a sulfur host to reduce the cost of Li-S batteries and can also be extended to other fields. But considering the complexity of real-world recycling masks, this work needs further research.

AUTHOR CONTRIBUTIONS

Qian Rong: Writing and data analysis. Chao Yuwen: data analysis. Peng Liu: Data representation. Feixiang Chen: Modification of the article. Shubiao Xia: Project management.

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CONFLICT OF INTEREST

There are no conflict of interest to declare.

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**SUPPORTING INFORMATION**

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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