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Chicken feathers as an intrinsic source to develop ZnS/carbon composite for Li-ion battery anode material

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ABSTRACT

Nowadays, renewable biomass-wastes are considered as one of the green precursors for developing multifunctional nanomaterials. Herein, we successfully developed a zinc sulfide (ZnS) doped chicken feather carbon (ZnS-CFC) composite using bio-waste of chicken feathers (CFs) as the natural sulfur and carbon precursor via simple two-steps approach. Under a hydrothermal condition, Zn precursor in situ reacts with the CFs to form a ZnS doped hydrothermally treated feathers (ZnS-HTF) hybrid, and further pyrolysis yields a ZnS-CFC composite. Transmission electron microscopy (TEM) revealed that ZnS nanoparticles are existing in a stable sub 10 nm size within the carbon matrix without any capping agents. As–prepared ZnS-CFC composite was utilized as an anode material for the Li-ion battery and achieved a reversible capacity of 788 mAh g⁻¹ after 150 cycles at 100 mA g⁻¹ with a good rate performance. Interestingly, the present work provides an eco-friendly way of developing metal sulfide-carbon composites that have the potential for various practical applications.

Keywords: ZnS; carbon composite; chicken feathers; Li-ion battery; capacity
1. Introduction

ZnS nanostructured materials have received potential research interest in multiple applications, including energy storage, environmental remediation, photo electronics, catalysis, etc. [1, 2]. However, ZnS alone have limitations, such as weak structural stability and poor conductivity, mainly in energy storage applications [3, 4]. A substantial research effort has been dedicated to enhancing the structural stability and conductivity of the pristine ZnS nanomaterial. Numerous research reports revealed that the carbonaceous materials are the most suitable and stable matrices for the impregnation of ZnS, which significantly enhances the conductivity of the ZnS due to its excellent porous architecture and structural stability [5, 6]. Moreover, ZnS doped carbonaceous composites have also drawn significant attention towards energy storage applications including, lithium-ion (Li-ion) and sodium-ion (Na-ion) batteries, supercapacitors, etc.[7-9]. Notably, the sulfur and carbon precursors are playing a vital role in the production of transition metal sulfides (TMSs) doped carbon (TMSs/C) composites, including ZnS-carbon composites. In general, sodium sulfide [10], thiourea [10], thioacetamide [11], elemental sulfur [12], hydrogen sulfide [13], glucose [14] and resorcinol-formaldehyde (RF) resin [15, 16] are the typical sulfur and carbon sources, respectively, used for the preparation of TMSs/C composites. However, most of the sulfur precursors are toxic, carcinogenic, and might generate a hazardous effect during the reaction and also cause environmental issues [17]. Therefore, it is necessary to explore an alternative and suitable sulfur precursor to produce the TMSs based hybrid materials on a large scale using a cost-effective method. In recent days, renewable biomass or bio-wastes are employed as intrinsic precursors for the large scale production of TMSs/C composites. Especially, with a distinct biomass waste having a natural carbon, nitrogen, and sulfur source, it could be more pronounced for the in situ synthesis of TMSs/C composite materials.

CFs are the most abundant bio-waste from the poultry industry, which is disposed of as a waste in lands, causing environmental issues [18, 19]. Therefore, converting CFs into a value-added material is one of the excellent ways of minimizing ecological problems. Currently, several research reports devoted that the CFs are a useful source for the development of progressive materials, including bio-films and porous carbons, significantly utilized for packing, energy storage, and environmental applications [20-22]. Based on the view, we found that bio-waste of CFs could be an alternative for the synthetic sulfur precursor as it owns the high content of
cysteine, which is the significant intrinsic source of sulfur. Consequently, CFs can be a suitable and inherent single bio-precursor for the \textit{in situ} synthesis of ZnS doped chicken feather carbon (ZnS-CFC) composite through a straightforward method. Besides, there have been no endeavors made to prepare the ZnS-CFC composite using CFs-aided routes. Thus, the main novelty of the present work is to generate the ZnS- carbon composite using intrinsic biosource of chicken feathers as the single precursor instead of synthetic precursors of carbon (sucrose, block copolymers, etc.) and sulfur (sodium sulfide, thiourea, elemental sulfur, thioacetamide, etc). Efforts have been paved on understanding the structure and morphology of the as-prepared ZnS-CFC using XRD, XPS and TEM analysis and explaining the growth mechanism for the formation of ZnS-CFC. Besides, the storage capacity of the as-prepared ZnS-CFC has been studied so that it could be effectively used as an active anode material for the Li-ion battery. The present work provides a promising way to utilize the bio-waste of CFs for the development of other transition metal sulfide (TMSs)/carbon composites via low-cost and green chemistry-based synthesis method.

2. **Experimental Section**

2.1. **Materials**

CFs were collected from a local poultry company in Abu Dhabi, United Arab Emirates (UAE). Zinc acetate hexahydrate (99.999\%) was used as received from Aldrich.

2.2. **Synthesis of Chicken Feather Carbon (CFC)**

CFs were thoroughly washed using deionized (DI) water and dried at 80 °C for 24 h and cut into small pieces. 7.0 g of dried CFs was soaked with 50 ml of deionized water in a 100mL of Teflon autoclave for 12h. Then, the autoclave was placed in the hot air oven and raised the temperature to 180 °C with the ramping rate of 5 °C min\(^{-1}\) and kept it for 12 h. Subsequently, the autoclave was allowed to cool in room temperature, and the content was freeze-dried at -20 °C. The resultant product was designated as hydrothermally treated chicken feathers (HTF). Further, the HTF was carbonized at 600 °C with a heating rate of 5 °C min\(^{-1}\) for 2 h in the Argon medium, produced a chicken feather carbon (CFC).
2.3. Synthesis of ZnS doped chicken feather carbon (ZnS-CFC) composite

ZnS-CFC was derived through the above said experimental condition (sec.2.2) using 7.0 g of dried CFs, and 50 mL of 0.5 M of Zinc acetate hexahydrate solution.

2.4. Characterization methods

The structural behavior of Zn-HTF, CFC, ZnS-CFC composites was investigated using wide-angle X-ray diffraction (WAXRD) analysis through an analytical powder diffractometer (Panalytical) equipment with Cu Kα radiation (λ=1.5406 Å) in reflection mode, and a zero-background holder was employed to reduce the noise peaks. CFC and Zn-CFC were separately scanned in the 2θ range of 5 to 80° with the 2θ step size of 0.03° and a step time of 10 s. The morphological behavior of CFC and ZnS-CFC composite was investigated in the FEI electron microscope (TECNAI) at 200 kV at room temperature without marking. Each of these samples was properly dispersed in isopropanol medium, and the dispersed solution was dropped to nickel electron microscopy grids (400 mesh formvar Ni grid coated with holey carbon film). The TEM images of the samples were captured using Digital Micrograph software (Gatan, USA). X-ray photoelectron spectroscopy (XPS) analysis of CFC and ZnS-CFC was studied using the PHI 5000 VersaProbe (Ulvac-PHI) instrument at a pressure below 109 Torr. Wide scan survey, C1s, N1s, S2p, and the Zn core level spectra of the respective samples were recorded in a monochromatic Al Kα radiation (photon energy =1486.6 eV) through the pass energy of 117.4 eV and 58.7 eV and an electron take-off angle of 45°.

2.5. Electrochemical analysis

The working electrode was made up of ZnS-CFC (80 wt. %), carbon black (10 wt. %), and polyvinylidene difluoride (PVDF) (10 wt. %). To fabricate the electrode, these materials were slurried in a required amount of N-methyl-2-pyrrolidone (NMP) medium, and the thick slurry was coated on Cu foil and dried at 140 °C under vacuum for 12 h to remove the trace solvent. Further, the electrode sheet was stamped into a disk shape with a diameter of 14 mm and used as a working electrode. CR2032-type coin cells consisting of the working electrode of Zn-CFC, counter/reference electrode of lithium metal, were fabricated in a glovebox. In the coin-cell, the electrodes were separated through the Celgard 2300 separator. 1M of Lithium hexafluorophosphate (LiPF6) LiPF6 in ethylene carbonate (EC)/diethyl carbonate (DEC) (1:1 by
volume) was used as an electrolyte in the coin-cell. The electrochemical performances of the fabricated coin cells were investigated with an electrochemical cycler (BioLogic VMP3). Galvanostatic charge and discharge (GCD) were studied at different current densities within a potential range between 0.01 to 2.5 V. Electrochemical impedance spectrometry (EIS) measurement of the sample investigated in the frequency range between 1 mHz and 100 kHz at an amplitude of 5 mV.

3. Results and Discussion

CFs mainly consist of the structural protein, keratin (>90%) along with the high content of hydrophobic residues, and cysteine [23]. The presence of the thiol (-SH) group in the cysteine molecule gets readily crosslinked via inter and intramolecular hydrogen bonding and resulted in a disulfide (S-S) linkage in the CFs. Then, the crosslinked cysteine molecule turns into the keratin polymer, which consists of S-S bonds, thereby strengthening the CFs [23, 24]. Cysteine is the well-known amino acid and a suitable sulfur precursor for the synthesis of TMSs. To generate a cysteine molecule, it must break the disulfide bond from the CFs through physical or chemical treatments [25]. In the present work, we adopted the hydrothermal method to break the disulfide bonds in the CFs, which resulted in a hydrolyzed form of free amino acids and peptides [26]. It is noteworthy that the transition metal (TM) precursors readily react with the thiol group, lead to a metal-sulfide (M-S) linkage via coordination and chelation mechanism [27]. The hydrothermally treated chicken feathers (HTF) might consist of free thiols groups on its surface, and it can be easily bound with the metal precursor solution, yielding a hybrid M-S complex structure (equ.1) (Scheme1).

Keratin -Cys -S – S- Cys- Keratin + Zn precursor \[\xrightarrow{\text{Hydrothermal}}\] [M (Cysteine)\(_n\)]\(^{2+}\) … (eq.1)

\[\xrightarrow{\text{Pyrolysis}}\] [Zn (Cysteine)\(_n\)]\(^{2+}\) ZnS-doped carbon composites ……(eq.2)

The growth mechanism of ZnS initiated on the surface of the HTF under the hydrothermal condition with a period of 12 h. Under a hydrothermal condition, the Zn precursor readily reacted with the HTF and showed a complete growth of ZnS nanoparticle on HTF, evidenced by the XRD analysis (Figure S1). Moreover, as-formed ZnS nanoparticles are more stable with HTF, because
the HTF consisting of a hydrolyzed form of free amino acids and peptide molecules, which are acting as a capping agent, and provided the structural stability to ZnS nanoparticles. During the pyrolysis process, ZnS-HTF gets carbonized at 600 °C, leading to graphitic carbon nanostructure with stable ZnS nanoparticle. Besides, the unreacted sulfur precursors of free amino acids and peptides in the ZnS-HTF get decomposed during the pyrolysis process, resulting in the co-doping of N and S on the carbon matrix (eq. 2) (Scheme 1) [27].

Scheme 1. Schematic illustration of the formation of ZnS-CFC composite.

Fig. 1 shows the XRD spectra of CFC and ZnS-CFC composite. As shown in Fig.1, CFC showed two broad diffraction reflections around 25.4° and 44.1°, which correspond to (001) and (002) planes, respectively, indicating the presence of the graphite phase in the CFC. The XRD pattern of ZnS-CFC showed the diffraction reflection at 2θ =29.10°, 48.2°, and 57. 02° corresponds to (111), (220) and (311) planes, which are perfectly matching with the crystal planes of the cubic ZnS phase (JCPDS. 00-005-0566) [28]. However, the characteristic diffraction reflection of the graphitic plane at around 25° was not be observed in the XRD patterns of the ZnS-CFC composite, indicating a broad graphitic peak is masked over by the high degree of ZnS crystal phase.

Fig. 2a-c shows the TEM images of CFC and ZnS-CFC, respectively. CFC exhibits a sheet-like morphology with the graphitic lattice planes (Fig. 2a). The presence of graphitic carbon nanostructure, which possesses an interconnected network that facilitates the electron transport property and minimizes the nanoparticle aggregation, resulting in significant structural stability to ZnS nanoparticle [29]. As shown in Fig. 2b-c, ZnS exhibited a spherical morphology, and it was uniformly distributed on the base plane of carbon sheets with a particle size of around 5 nm. The corresponding SAED pattern shows a ring pattern, indicating a polycrystalline nature of ZnS with the respective crystal planes of (111), (311), and (220) (Fig. 2d).
Fig. 1. XRD pattern of CFC and ZnS-CFC composites.

Fig. 2. a) TEM images of a) CFC, b) ZnS-CFC (low magnification), c) ZnS-CFC (high magnification) and d) SAED pattern of ZnS-CFC.

The chemical state and molecular environment of the CFC, and ZnS-CFC were identified by X-ray photoelectron spectroscopy (XPS). XPSPEAK software (4.1) was used to fit with XPS data in
a background correction of C1s, N1s, S2p, and Zn2p of ZnS-CFC composite. Fig.3a shows the XPS survey of the CFC, confirming the presence of C, N, and S elements in the carbon matrix. A major peak was observed in the C1s spectrum at 284.5 eV, corresponding to the C–C bond of sp2 hybridized atomic structure of graphite, ensuing that the CFC consists of a high graphitic content (Fig. 3b) [29, 30]. The N1s spectrum of CFC deconvoluted into four peaks, such as 398.8 eV (pyridinic nitrogen), 400.0 eV (pyrrolic nitrogen), 400.8 eV (graphitic nitrogen) and 404.9 eV (pyridinic N-oxide)(Fig. 3c) [31].

Fig. 3. XPS spectra of CFC: a) survey, b) C1s, c) N1s, and d) S2p
The presence of pyridinic nitrogen and pyrrolic nitrogen were significantly improved the pseudocapacitive behavior of the CFC. Also, the graphitic nitrogen in the CFC aided in improving the electrochemical performance of the carbon matrix [31]. The S2p spectrum of CFC showed two binding energy peaks at 164.0 eV for the 2p3/2 splitting of the S2p spin-orbital (C-S-C), and 165.2 eV for the 2p1/2 splitting of the S2p spin-orbital (C=S), respectively (Fig. 3d) [32].

![Fig. 3a](image1.png) ![Fig. 3b](image2.png) ![Fig. 3c](image3.png)

**Fig. 3.** S2p spectra of CFC: a) Cls, b) C1s, c) N1s, d) S2p, and e) Zn2p

The XPS survey, C1s, N1s, S2p, and Zn2p spectra of the ZnS-CFC are displayed in Figure 4a-e. From the survey, it was observed that ZnS-CFC has elemental peaks of C, O, N, S, and Zn in the composites with an atomic percentage of 73, 11, 9, 3, and 4%, respectively (Fig. 4a). In the ZnS-CFC composite, the chemical environment of C1s was not changed significantly (Fig. 4b). However, the N1s spectrum exhibits only one peak of pyridinic nitrogen at 398.8 eV (Fig. 4c), and the S2p spectrum exhibits splitting of two peaks at 162.1 and 163.4 eV, corresponds to S2p3/2 and S2p1/2 of S in the ZnS phase (Fig. 4d) [33]. Besides, the Zn2p spectrum exhibits two peaks at around 1022 and 1045.3 eV, attributing to the Zn 2p3/2 and Zn 2p1/2 spin-orbit peaks of ZnS (Fig. 4e) [33].

![Fig. 4a](image4.png) ![Fig. 4b](image5.png) ![Fig. 4c](image6.png) ![Fig. 4d](image7.png) ![Fig. 4e](image8.png)

**Fig. 4.** XPS spectra of ZnS-CFC: a) survey, b) C1s, c) N1s, d) S2p and e) Zn2p
Fig. 5a exhibits the CV profile of the ZnS-CFC anode at a scan rate of 0.5 mVs⁻¹ through the potential window range between 0 and 2.5 V. In the first cycle, a weak broad cathodic peak appeared between 1.0 to 1.5 V, and it disappeared in the subsequent cycles, indicating the degradation of the electrolyte and solid electrolyte interphase (SEI) formation in the electrode, signifying their reversible capacity behavior [34]. A reduction peak was observed in the 0.6-0.9V in the first scan, attributing to the reduction of ZnS to metallic zinc and the subsequent formation of Zn–Li alloys via several steps [34, 35]. In the subsequent cycles, the oxidation peaks appeared in the range of 0.3-0.7 V, owing to the delithiation of Zn-Li alloy [34]. Fig. 5b represents the galvanostatic charge-discharge (GCD) curve of the Zn-CFC composite for 1 to 300 cycles in the potential range of 0.01 V and 2.5 V with a constant current density of 100 mA g⁻¹. From Fig. 5b, we observed that the ZnS-CFC exhibits the initial discharge capacity of 512 mAhg⁻¹. The high capacity of the Zn-CFC might be due to the high irreversible SEI film formation on the composite surface. Fig. 5c shows the long term cycling stability of the ZnS-CFC composite at a current density of 100 mAg⁻¹. In the first cycle, ZnS-CFC exhibits the charge and discharge capacities of 352 and 512 mAhg⁻¹, with the initial coulombic efficiency of 69%. The irreversible capacity loss occurs due to the SEI layer formation on the active material surface or unreacted Li, and it is common for the conversion type anode materials [36, 37]. The coulombic efficiency rapidly raised to 93% and 97% at the 20th and 50th cycles, respectively (Fig.5c). The composite possessed a large capacity of 788 and 658 mAhg⁻¹ even after 150 and 300 cycles, respectively. The capacity of the ZnS-CFC increased significantly up to the 150th cycle, owing to the polymeric gel-like film formation at the interface between the electrode and electrolyte, which could offer large interfacial storage via pseudocapacitive mechanism [38]. Besides, the formation and destruction of SEI films are stable even after multiple cycles of the electrode, corresponding to the large improvement in their cyclic performance [38]. As well, the carbon matrix in the composite protects the volume expansion of ZnS nanoparticles during the electrochemical reaction, which also contributes to the high stability and large irreversible cycling performance of the anode material [39]. A similar trend was also observed in the ZnS-carbon composite reported earlier [39].

Table 1 shows the Li-ion battery (LIB) performance of the anode materials of ZnS based carbonaceous composites. From Table 1, we inferred that the ZnS-CFC composite shows a relatively high magnitude order of reversible capacity compared to the other ZnS based carbon composites [40-46]. The rate performance of ZnS-CFC anode was also investigated through GCD
mode at a different current density from 100 to 1000 mAg$^{-1}$ (Fig. 5d). The ZnS-CFC anode provides rate ability with reversible capacities of 510, 337, and 151, and 60 mAh g$^{-1}$ at a current density of 100, 300, 500, and 1000 mAg$^{-1}$. Besides, the current density is reverse from the 1000 mAg$^{-1}$ to 100 mAg$^{-1}$, a high capacity of 605 mAhg$^{-1}$ was achieved. The high capacity and stable rate capacity of the ZnS-CFC might be due to the composite structure of ZnS with the carbon matrix. The N and S- co-doping showed a synergetic effect on the carbon matrix, which effectively reduced the charge-transfer resistance of the ZnS nanoparticle and improved the charging behavior of the electrode [47].

**Table 1.** Li-ion battery performance of ZnS based carbon composites

<table>
<thead>
<tr>
<th>Anode materials</th>
<th>Reversible capacity (mAhg$^{-1}$)</th>
<th>Current Density (mAg-1)</th>
<th>Cycles</th>
<th>References</th>
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</thead>
<tbody>
<tr>
<td>ZnS/reduced graphene oxide</td>
<td>780</td>
<td>100</td>
<td>100</td>
<td>40</td>
</tr>
<tr>
<td>ZnS/Porous carbon</td>
<td>438</td>
<td>100</td>
<td>300</td>
<td>41</td>
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<tr>
<td>Core-shell structured ZnS-C</td>
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<td>500</td>
<td>500</td>
<td>42</td>
</tr>
<tr>
<td>ZnS/CNT.</td>
<td>451</td>
<td>5000</td>
<td>1200</td>
<td>43</td>
</tr>
<tr>
<td>ZnS/porous carbon fibers</td>
<td>718</td>
<td>200</td>
<td>150</td>
<td>44</td>
</tr>
<tr>
<td>ZnS-nitrogen doped carbon</td>
<td>690</td>
<td>100</td>
<td>100</td>
<td>45</td>
</tr>
<tr>
<td>ZnS/carbon nanosphere</td>
<td>659</td>
<td>1000</td>
<td>1200</td>
<td>46</td>
</tr>
<tr>
<td>ZnS-CFC</td>
<td>788</td>
<td>100</td>
<td>150</td>
<td>This work</td>
</tr>
</tbody>
</table>

The charge resistance behavior of the electrode was investigated through the Electrochemical Impedance Spectroscopy (EIS) analysis using the samples of before and after (300 cycles) the cycling performance (Fig. 5e). As-obtained EIS results could support the interpretation of results at the electrochemical charge-discharge process. In the Nyquist plot, a semi-circle was observed in the mid-frequency region, indicating the charge-transfer resistance (Rct) at the electrode/electrolyte interface [40]. Besides, the slope at a lower frequency region representing the Li-ion diffusion behavior in the electrode [48]. Before cycling, the ZnS-CFC shows the Rct value
of 265 ohms, and it drops to 238 ohms after 5 cycles at the current density of 100 mA g\(^{-1}\), indicating that the charge-discharge process aids to increase the compatibility between the active electrode material and electrolyte \[49, 50\].

![Graphs](image)

**Fig. 5.** a) CV profile of Zn-CFC at 0.5 mVs\(^{-1}\); b) Potential profile of ZnS-CFC at current density of 100 mA g\(^{-1}\); c) Cycling performance of ZnS-CFC at current density of 100 mA g\(^{-1}\); d) Rate capabilities of ZnS-CFC at the current density of 100, 300, 500, and 1000 mA g\(^{-1}\); and e) Nyquist plot of ZnS-CFC: before and after cycling.

### 4. Conclusion

In summary, ZnS-CFC was successfully derived using the intrinsic precursor of CFs through a simple and low-cost bio-inspired strategy. Under the hydrothermal condition, the CFs yield a cysteine molecule, and the Zn\(^{2+}\) precursor could coordinate with cysteine leading to the formation of metal sulfide (M-S) nanoparticle on HTF. After the pyrolysis reaction, a complete growth of ZnS nanoparticle on the carbon matrix was achieved. The as-synthesized ZnS-CFC composite was used as anode material for Li-ion battery. The presence of carbon exhibiting an interweaved network structure, enhanced the electrical conductivity and Li-ion transport property of the ZnS-CFC composite, and also reduced the volume buffering change of ZnS during cycling. As a result, ZnS-CFC demonstrated good electrochemical performance with a reversible capacity of 788 mAh g\(^{-1}\).
after 150 cycles at 100 mA g\(^{-1}\). Thus, the bio-waste of CFs acts as a single intrinsic precursor for the facile and environmentally friendly synthesis of TMSs doped carbon composites, and it can be extended to generate a wide range of metal sulfide architecture for advanced multi-disciplinary applications.

**Supporting Information.**

XRD spectra of ZnS-HTF and ZnS-CFC (Figure S1)

**Author Contributions**

F.B. suggested the idea of the paper. V.M.R perceived the idea and designed the experiments and A.A performed the experiments. V.M.R and A.A. characterized the materials. P.K. tested the material for the Li-ion battery application. V.M.R wrote the original draft of the manuscript. All authors have approved the final version of the manuscript.

**Declaration of competing interest**

The authors declare no competing financial interest.

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