Due to the shuttle effect and low conductivity of sulfur (S), it has been challenging to realize the application of lithium-sulfur (Li-S) batteries with high performance and long cyclability. In this study, a high catalytic active CNTs@FeOOH composite is introduced as a functional interlayer for Li-S batteries. Interestingly, the existence of oxygen vacancy in FeOOH functions electrocatalyst and promotes the catalytic conversion of intercepted lithium polysulfides (LiPS). As a result, the optimized CNTs@FeOOH interlayer contributed to a high reversible capacity of 556 mAh g\textsuperscript{-1} at 3,200 mA g\textsuperscript{-1} over 350 cycles. This study demonstrates that enhanced catalytic effect can accelerate conversion efficiency of polysulfides, which is beneficial of boosting high performance Li-S batteries.

Keywords: CNTs@FeOOH, oxygen vacancy, catalytic effect, polysulfides, Li-S batteries

INTRODUCTION

Currently, Li-S batteries have received extensive attention due to their high theoretical capacity (1,675 mAh g\textsuperscript{-1}), high energy density (2,600 Wh kg\textsuperscript{-1}) (Ji and Nazar, 2010; Manthiram et al., 2013), low cost and environmental friendliness (Nazar et al., 2014; Yang et al., 2018). Considering the low conductivity of S (σ = 5.0 × 10\textsuperscript{-30} S cm\textsuperscript{-1}) and the shuttle effect of LiPS, research on Li-S batteries is strongly delayed and thus hardly meets actual needs (Zhang, 2013; Rosenman et al., 2015). To solve these problems, many pioneering works are using a porous carbon-based host, a functional interlayer, absorbable polar composites [such as CoS\textsubscript{2}/C (Yuan et al., 2016), MnO\textsubscript{2}/GO/CNT (Kong et al., 2017), S@TiO\textsubscript{2} (Wei Seh et al., 2013) etc.] and a catalytic effect on LiPS conversion. Among them, carbon-based hybrid materials with absorbility to LiPS have always shown attractive characteristics (Tang and Hou, 2018). For instance, Pang and Nazar obtained C\textsubscript{3}N\textsubscript{4} by pyrolysis of melamine, which has rich pyridine nitrogen adsorption sites. This can lead to the fact that the sulfur electrode with ultra-low long-term capacity fades out by 0.04% for a cycle over 1,500 cycles at a practical rate of 0.5C (Pang and Nazar, 2016). However, it should be noted that absorbed LiPS is easy to release, because these materials are soluble in the electrolyte. Thus,
a more efficient strategy is urgently needed to meet this problem. Catalyst has been employed to accelerate the transformation of LiPS from liquid to solid, which corresponds to the transmission from long-chain Li$_2$S$_n$ to short-chain Li$_2$S. We strongly believe that this is an alternative option to modify Li-S batteries by introducing catalytic materials to catalyze the conversion of LiPS into insoluble products. Yang et al. reported the Fe$_3$C/Fe-N$_x@NPCN$ modified separator, due to the catalytic effect of Fe$_3$C to LiPS, the modified batteries yielded a high capacity of 1,517 mAh g$^{-1}$ at 0.1C and displayed a capacity decay rate of 0.034% per cycle after 500 cycles at 1C (Yang et al., 2019). Bian et al. took multi-functional porous carbon nanofibers (g-C$_3$N$_4@PCNF$) as the sulfur host, in which g-C$_3$N$_4$ contributed to rapid oxidation-remediation conversion of S species and slowed down LiPS yield. Consequently, the g-C$_3$N$_4@PCNF/S$ cathode achieves good flexibility and excellent cycling retention, e.g., long cycling with decay power of only 0.056% per cycle for 500 cycles at 1.0 A g$^{-1}$ (Bian et al., 2019). In addition, Lee and coworkers have demonstrated that catalytic activity in anoxic sites is higher in saturated sites as the oxygen vacancy can contribute to Fe$^{3+}\rightarrow$LiPS conversion. We propose using CNTs@FeOOH composite as the catalyst for LiPS conversion. We next explore how CNTs@FeOOH composites with various mass ratio can be employed to modify Li-S batteries by replacing the binder in the electrolyte. As reported in our previous work (Bian et al., 2019), the CNTs@FeOOH-I, II, and III, respectively). Compared to pure CNTs and FeOOH in Figure S1, the formation of rod-shaped FeOOH may induced by the reaction conditions. In addition, the X-ray diffraction (XRD) patterns of all samples are compared in Figure 1d. The peaks of pure FeOOH are located at 2$\theta$ = 11.925°, 16.901°, 26.874°, 34.185°, 35.311°, 39.386°, 46.656°, 52.349°, 56.158°, 61.278°, 64.718°, and 68.117°. These peaks are attributed to the (110), (130), (200), (211), (301), (411), (600), (251), (002), and (541) reflection planes of FeOOH (ICPDS 75-1594). The FeOOH peaks become more evident with increasing FeOOH content and no impurity phase is detected. Transmission electron microscope (TEM) images of CNTs@FeOOH-II, demonstrated that FeOOH particles are uniformly adhered on the surface of the CNTs, which consistent well with the results of SEM (see Figures S2a,b). The selected area electron diffraction pattern of the CNTs@FeOOH-II nanomaterials shows diffraction rings characteristic of FeOOH and CNTs (see Figure S2c), while the image of high resolution TEM in Figure S2d indicates lattice
fringes corresponding to (110), (200), (211), and (330) planes. The results are consistent with those of XRD spectrums. Besides, thermogravimetric analysis (TGA) was used to calculate the proportion of components in various composites (Zhang et al., 2017). As shown in Figure 1e, the FeOOH content is measured as 24.8, 45.7, and 71.5 wt% for the composites CNTs@FeOOH-I, II, and III, respectively.

X-ray photoelectron spectroscopy (XPS) was employed to investigate the composition of elements on the surface of CNTs@FeOOH nanocomposites and the chemical states of various bond elements. The result shown the presence of Fe, O, and C atoms at the CNTs@FeOOH sheet surface (Figure S3a). The binding energy peak observed in the high-resolution C 1s profile at 284.9 eV (Figure S3b) can be attributed to graphite carbon in carbon nanotubes. The peak at 285.4 eV belongs to epoxy and hydroxyl (Beamson et al., 1994). The other two peaks are caused by carbonyl (C=O, 288.7 eV) and the oxygenated carbons of carboxyl (O-C=O, 291.2 eV) (Gardella et al., 1986; Kokai, 1990). These peaks reveal the existence of oxygen-containing functional groups on the surface of CNTs (Zhang et al., 2017). Meanwhile, the finescanned Fe 2p XPS spectra of that sample was also shown in Figure S3c, and the Fe 2p3/2 and Fe 2p1/2 peaks located at 711.0 and 724.9 eV could be indexed to Fe3+ and Fe2+, respectively, and satellite peaks at 719.4 and 733.9 eV correspond well with FeOOH (Tan et al., 1990). In addition, the O 1s peaks (Figure 1f) can be assigned to Fe-O-Fe (529.5 eV), Fe-O-H (530.1 eV), and H-O-H bonds (531.2 eV). It is worthwhile to point out that the peak at 531.2 eV is attributed to defect sites with low oxygen coordination (Zhang et al., 2019). According to previous reports, surface oxygen vacancies are involved in the LiPS transformation reaction, which significantly improves the kinetics of the reaction, thus contributing to the fast LiPS transformation at high rate.

According to the previous paper, the shuttle effect of polysulfides can be diminished with an additional intermediate layer (Fan et al., 2019). In this work, various composites of CNTs@FeOOH were coated on the surface of the PP separator as a functional interlayer to study their effect on Li-S batteries. Figure S4a shows a schematic representation of conventional PP-separator Li-S structures and advanced Li-S batteries with functional CNTs@FeOOH layers. The surface morphology of the modified interlayer is shown in Figures 2a–c, the preserved porous structure will facilitate electrolyte penetration and lithium ions (Li+) transfer. Figure 2d and Figures S4b,c show the cross-sectional appearance, and the thickness of the interlayer is about 20 μm.

Coin-typed cells with different separators were assembled to evaluate electrochemical performances. Figure S4d exhibits the cycling performance of all Li-S cells with PP separator, CNTs, pure FeOOH and three proportional composites interlayer at 0.2C (1C = 1,675 mA g⁻¹) between 1.7 and 2.8 V. After 100 cycles, CNTs@FeOOH-II revealed the best cyclability of 662.1 mAh g⁻¹, which indicates a good synergistic effect between CNTs and FeOOH.

In order to explore the lithium diffusion properties and investigate the role of composite materials in Li-S batteries, we performed cyclic voltammetry (CV) measurements under various scanning rates ranging from 0.1 to 0.5 mV s⁻¹ between 1.7 and 2.8 V (vs. Li/Li⁺). As shown in Figures 3A–D, all curves show typical reduction/oxidation reaction of S cathode, with two
distinct cathode peaks and one anode peak. The cathode peak at about 2.3 V corresponds to the transformation of sulfur bonding with Li$^+$ into soluble long-chain polysulfide [Li$_2$S$_x$ ($x = 4–8$)]. Furthermore, the cathode peak around 2.0 V corresponds to the transformation of long-chain polysulfide into insoluble Li$_2$S or Li$_2$S$_2$ (Chung et al., 2016). In subsequent anode scanning, the
oxidation peak at ~2.4 V corresponds to its reverse process. According to the relationship between CV scanning rate (v0.5) and peak current (Ip), the lithium diffusion performance can be estimated using the classical Randles Sevcik equation:

\[ Ip = (2.69 \times 10^5)n^{1.5}D_{Li^{+}}^{0.5}C_{Li^{+}}^{0.5} \]  

where, Ip-peak current (A), n-number of electrons per type of reaction (n = 1), S-electrode area (S = 1.13 cm²), D_{Li^{+}}-diffusion coefficient of lithium ion (cm² s⁻¹), C_{Li^{+}}-initial concentration of lithium ion in the cathode (mol cm⁻³), v-potential scanning rate (V s⁻¹) (Tao et al., 2016). The n, S, and C_{Li^{+}} are constant in our battery system. The slope of the curve in Figure S5a is positively correlated with the corresponding Li⁺ diffusion. The calculated results show that the modified CNTs@FeOOH-II had the greatest diffusion capacity of Li 4.40 \times 10^{-12}, better than intact (2.08 \times 10^{-12}). Typically, the PP separator has difficulty catching soluble LiPS, which tends to dissolve in electrolytes in large quantities. As a result increasing viscosity of electrolyte leads to slower diffusion of Li⁺. On the contrary, since CNTs@FeOOH-II material can accelerate the conversion of LiPS to Li₂S or Li₂S, it is easier to increase the diffusion rate of Li⁺ in modified cells.

The discharge process in Li-S batteries can be expressed as follows:

\[ \text{S}_8 + 2e^- \leftrightarrow \text{S}_4^{2-} \]  

\[ 3\text{S}_8 + 2e^- \leftrightarrow 4\text{S}_6^{2-} \]  

\[ 2\text{S}_6^{2-} + 2e^- \leftrightarrow 3\text{S}_4^{2-} \]  

\[ \text{S}_4^{2-} + 4\text{Li}^{+} + 2e^- \leftrightarrow 2\text{Li}_2\text{S}_2 \]  

\[ \text{Li}_2\text{S}_2 + 2\text{Li}^{+} + 2e^- \leftrightarrow 2\text{Li}_2\text{S} \]  

The theoretical discharge capacity of Li-S battery at different stages is calculated by referring the number of electrons transferred. The details are given in Table 1 and specific formulas are given below (Diao et al., 2013):

\[ q = nF/M \]  

Among them, q is the specific discharge capacity, mAh g⁻¹; n is the number of transfer electrons per mole mass, mol⁻¹; F is the amount of electricity owned by 1 M electrons, 26.8 Ah; M is the molar mass of elemental sulfur, 32 g mol⁻¹ (Diao et al., 2013). Here, the discharge capacity of S₈ \rightarrow S₄⁻ is recorded as S₁, and that of S₈ \rightarrow Li₂S as S₂. Accordingly, S₁ : S₂ is approach to 1:3. Figures 4A-D shows the discharge curve of the PP separator and various barrier interlayers circulating for 200 cycles at 2C. The calculated results show that the capacity ratio of CNTs@FeOOH-II (1:2.44) is closest to the theoretical value, higher than 1:2.26, 1:2.27, and 1:1.74 of PP separator, CNTs and FeOOH, respectively, which indicates the enhanced transformation ability of CNTs@FeOOH-II to polysulfide ions at high current density.

Figure 4E compares the cyclic characteristics of the batteries with PP separator, CNTs, pure phase FeOOH and CNTs@FeOOH-II separator in a voltage range of 1.7–2.8 V at 2C. The initial discharge capacities are 870.6, 1,093, 1326.4, and 1121.9 mAh g⁻¹, respectively, show that CNTs@FeOOH-II interlayer can strengthen the utilization of S upon cycling. After 350 cycles, CNTs@FeOOH-II maintained a high reversible capacity of 556 mAh g⁻¹. In detail, although CNTs have good electrical conductivity, the weak van der Waals interactions between polar LiPS and non-polar carbon materials results in slow release of S-active substances from carbon materials and obvious capacity decay during long cycle (Song et al., 2016). Simultaneously, it is remarkable that the highest reversible capacity of 1326.4 mAh g⁻¹ can be obtained for pure FeOOH group. This can be explained by the presence of oxygen vacancies in FeOOH, which makes it electrocatalytic and prompts the rapid conversion of long-chain LiPS to solid Li₂S₂ and Li₂S. Since 75% of the discharge capacity (1,254 mAh g⁻¹) occurs in this conversion process, an enhanced reaction kinetics is beneficial for increasing the reversible capacity (Lim et al., 2019). However, since previous studies have shown that the absorption of LiPS by insulating interlayer is considered to be a “death zone” without transferring electrons during cycling (Hao et al., 2017). The fading trend in the battery with FeOOH interlayer mainly result from its low conductivity (10⁻⁵ S cm⁻¹). Compared with relevant studies (Table S1), the introduction of this functional interlayer delivered better performance improvement for Li-S batteries.

The existence of functional interlayer can be used both as a conductive top current collector and as a physical barrier to polysulfide diffusion and lithium (Li) metal, debase the corrosion of Li metal. In case of rate performance (Figure S5b), CNTs@FeOOH-II composites exhibit high discharge capacity of 1292.6, 957.5, 802.3, and 630.8 mAh g⁻¹ at various rates from 0.2 to 2C, which is more satisfactory than CNTs and FeOOH. In particular, when the rate was restored to 0.2C, the specific capacity of the battery returned to 972.5 mAh g⁻¹. These results confirm that CNTs@FeOOH-II interlayer enhances the stability.

| Discharge products | Transfer electron number/n | Depth of discharge DOD | Discharge specific capacity/q (mAh g⁻¹) |
|--------------------|---------------------------|-----------------------|--------------------------------------|
| S₈ \rightarrow S₄⁻  | 0.25                      | 12.5%                 | 210                                  |
| S₈ \rightarrow S₄⁻  | 0.33                      | 16.7%                 | 280                                  |
| S₈ \rightarrow S₄⁻  | 0.5                       | 25.0%                 | 420                                  |
| S₈ \rightarrow Li₂S₂ | 1                         | 50.0%                 | 840                                  |
| S₈ \rightarrow Li₂S | 2                         | 100.0%                | 1,680                                |
The presence of FeOOH in CNTs@FeOOH-II composite can enhance the rapid transformation of polysulfide ions and the hysteretic conversion kinetics of Li-S batteries, thus improving the rate capability of Li-S batteries.

The electrocatalytic effects of the different interlayers on PP separator were studied by electrochemical impedance spectroscopy (EIS). In Figure 5 shows the Nyquist plots when discharges up to 2.1 V after 10, 30, 50 cycles, respectively. Each plot consists of one oblique line in a low frequency region and one or two compressed semicircles in a medium and high frequency region. The corresponding equivalent circuit model is shown in Figure 5A. In the equivalent circuit, R_s represents the ohmic resistance of the reaction system; R_1 is related to the resistance of the solid electrolyte interface (SEI), corresponding to a semicircle of the high frequency region; R_{ct} represents the charge transfer resistance, corresponding to a semicircle of the mid-frequency region, the diameter of the semicircle is the size of R_{ct}, the larger the diameter, the greater the impedance, the more unfavorable to the high performance; CPE-double layer electrode/electrolyte capacitance; W characterizes the Warburg diffusion impedance of the electrode, which corresponds to an oblique line in the low frequency band, it characterizes the diffusion rate of Li^+ in the

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**Figure 4** | The 200th cycle Discharge profiles of the battery with (A) PP separator, (B) CNTs, (C) FeOOH, and (D) CNTs@FeOOH-II separator, respectively, at 3,200 mAh g\(^{-1}\); (E) cycle performance at 3,200 mAh g\(^{-1}\).
FIGURE 5 | Nyquist plots of (A) PP separator, (B) CNTs, (C) FeOOH, and (D) CNTs@FeOOH-II.

TABLE 2 | The comparison of Rct values of different interlayer and PP separator.

| Samples             | 10th   | 30th   | 50th   |
|---------------------|--------|--------|--------|
| PP separator        | 24.07  | 22.55  | 34.15  |
| CNTs                | 22.46  | 18.2   | 37.12  |
| FeOOH               | 48.49  | 28.68  | 54.96  |
| CNTs@FeOOH-II       | 19.24  | 15.94  | 21.64  |

material, the larger the slope, the better the high performance (Hu et al., 2018).

The fitting values of $R_{ct}$ were exhibited in Table 2, the S electrode with CNTs@FeOOH-II interlayer endowed the lowest $R_{ct}$ value after several charge-discharge cycles. The results are in good accordance with the results of the electrochemical cycle test. The conductivity of CNTs@FeOOH-II was measured by four-point probe method, which is about 4.6 S cm$^{-1}$. High conductivity of CNTs@FeOOH-II may accelerate electron transfer and reduce electrochemical polarization. This further indicates that electrocatalytic materials with high conductivity and faster electron and ion transfer rates can improve the electrochemical performance of Li-S batteries.

CONCLUSION

In conclusion, FeOOH combined with CNTs with excellent catalytic ability is applied for high performance Li-S batteries. Compared to the pristine samples, the modified battery exhibited a good performance of 556 mAh g$^{-1}$ at 3,200 mA g$^{-1}$ for 350 cycles. CNTs@FeOOH-II plays the following main roles: (i) oxygen vacancies in FeOOH promote the rapid transformation of polysulfide ions, thus enhancing the reaction kinetics; (ii) the presence of FeOOH can effectively adsorb soluble polysulfides, which sluggish the further diffusion to the anode; (iii) CNTs@FeOOH-II with high electric conductivity can be used as a "vice-electrode" to accelerate electron transfer and thus improve the rate capability. Therefore, for a high performance of Li-S battery, it is necessary to consider the high conductivity, adsorption, and fast conversion of LiPS.

DATA AVAILABILITY STATEMENT

All datasets generated for this study are included in the article/Supplementary Material.

AUTHOR CONTRIBUTIONS

YL, XL, and YH contributed conception and design of the study. YL organized the database, performed the statistical analysis, and wrote the first draft of the manuscript. All authors contributed to manuscript revision, read, and approved the submitted version.

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SUPPLEMENTARY MATERIAL

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The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.