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Biomass-Derived Activated Carbon-Coated Cobalt Phosphide Nanocomposites for High-Performance Energy Storage

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***Abstract* - Biomass-derived energy storage devices are emerging as a sustainable strategy to reduce reliance on nonrenewable resources. Herein, wheat husk-derived active-carbon coated CoP nanostructures (CoP@NWHC) were synthesized for the first time via facile treatment followed by hydrothermal growth. In the CoP@NWHC composite, carbon nanostructures are conformally grown on both the interior and exterior surfaces of the CoP framework. This integrated carbon layer not only provides structural support for CoP but also establishes continuous conductive pathways that facilitate rapid electron transport throughout the electrode. Furthermore, the hollow helical carbon structure with inherent capillary effects provides rapid ion diffusion channels. Owing to this hierarchical architecture, the CoP@NWHC electrode delivers a high specific capacitance of 521 F g^{-1} at 0.5A g^{-1}, excellent cycling stability of 90.4% after 8000 cycles. Demonstrating its potential for practical energy storage applications.**

Keywords: supercapacitors, CoP@NWHC, nanostructure, composite, electrochemical performance, energy storage.

I. INTRODUCTION

With growing concerns over petroleum depletion and environmental pollution, clean and sustainable energy technologies are gaining increasing attention [1, 2]. Supercapacitors, as one of the most promising energy storage devices, have attracted wide interest due to their high power density, fast charge–discharge rate, and long cycle life [3, 4]. Supercapacitors can be classified into two categories based on charge storage mechanisms; Electric double-layer capacitors (EDLCs): Typically fabricated from carbon-based materials. They exhibit high power density and excellent cycling stability but suffer from low capacitance [5, 6]. Pseudocapacitors: Generally composed of conductive polymers and metal oxides/hydroxides. They deliver high specific capacitance and energy density but show lower stability [7, 8]. Among various pseudocapacitive materials, cobalt phosphide (CoP) is regarded as a promising candidate for supercapacitors because of its high theoretical specific capacitance, well-defined redox transitions, and cost effectiveness [9, 10]. However, practical application of CoP-based electrodes remains limited by poor dispersion and low electrical conductivity, leading to reduced capacitance, poor rate capability, and short cycle life [11-13]. To fully exploit the intrinsic properties of CoP, various carbon materials with large surface area and high electrical conductivity have been incorporated to construct metal phosphide/carbon composites [14], including graphene, activated carbon, carbon nanotubes (CNTs), and carbon fibers (CFs) [15]. Among them, one-dimensional (1D) carbon structures such as CNTs and CFs can provide long-range pathways for efficient electron and ion transport, thereby enhancing the power characteristics of the composite [16, 17]. Nevertheless, conventional synthesis routes for these carbon materials are complex, costly, and environmentally unfriendly [18]. Therefore, developing 1D carbon materials with well-designed structures via a facile, inexpensive, and green approach remains urgently needed for supercapacitor applications [19, 20]. Recently, biomass-derived materials have emerged as attractive precursors for carbonaceous materials owing to their renewability, low cost, and environmental friendliness [21-23]. A wide range of biomass sources such as watermelon [24], leaves, bamboo char [25], boat-fruited sterculia seed [26], and pomelo peel [27] have been widely used to fabricate carbon materials. These not only significantly reduce cost but also inherit the extraordinary microstructures of natural precursors [28]. Wheat husk-derived carbon, used without pretreatment, has demonstrated excellent surface properties and energy storage

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performance due to its inherent ability to facilitate moisture and nutrient transport [29]. Accordingly, wheat husk-derived carbon can serve as an active support for nanomaterials such as cobalt phosphide composites.Moreover, wheat husk is economically inexpensive, and the carbon prepared from it introduces no hazardous byproducts during synthesis [30]. In this work, we report for the first time a novel carbon composite grown on CoPnanorod arrays to form a CoP@NWHC composite with a unique helical structure, prepared via facile acid treatment followed by a hydrothermal process. The helical architecture not only provides a corrugated external carbon surface for charge storage, but also renders the internal surface accessible, creating sufficient active sites for redox reactions [31]. The carbon coating can function as a long-range conductive current collector for electron transfer, while its hollow helical structure, combined with capillary effects, provides rapid channels for ion diffusion [32]. By combining the high pseudocapacitance of CoP with the high power characteristics of the micro-carbon framework, the as-prepared CoP@NWHC composite electrode exhibits a high specific capacitance of 521 F g^{-1} at 0.5 A g^{-1}, and excellent cycling stability with 90.4% retention after 8000 cycles. In summary, this work provides a new structural model for biomass-derived electrode materials after coating onto cobalt phosphide, paving the way for high-performance energy storage.

II. EXPERIMENTAL SECTION

2.1 Materials

Cobalt(II) nitrate hexahydrate ($Co(NO_3)_2·6H_2O$, 99%), ammonium phosphate monobasic ($NH_4H_2PO_4$, 99%), urea (CH_4N_2O, 99%), sodium hypophosphite dihydrate ($NaH_2PO_2·2H_2O$, 99%), sodium dodecyl sulfate (SDS, 99%), ferric chloride hexahydrate ($FeCl_3·6H_2O$, 98%), and hydrochloric acid (HCl, 37%) were purchased from Aladdin Industrial Inc. and Sinopharm Chemical Reagent Co., Ltd, Shanghai, China. All reagents were used as received without further purification. Deionized water was used throughout the experiments. NWHC was prepared according to our previous work reported by Souad *et al.* [33]

2.2 Synthesis of CoP nanorods

CoP nanorods were synthesized via a hydrothermal-phosphorization two-step method. In a typical procedure, 448 mg of $Co(NO_3)_2·6H_2O$, 150 mg of $NH_4H_2PO_4$, and 50 mg of urea were dissolved in a mixed solvent of 50 mL deionized water and 25 mL ethanol under magnetic stirring for 30 min to form a homogeneous solution[34]. The solution was then transferred into a 100 mL Teflon-lined stainless-steel autoclave and maintained at 160 °C for 6 h. After cooling to room temperature, the obtained $Co_3(PO_4)_2$ precursor was collected by centrifugation, washed several times with deionized water and ethanol, and dried at 60 °C for 8 h.Subsequently, phosphorization was carried out in a tubular furnace under N_2 atmosphere. 0.02 g of the $Co_3(PO_4)_2$ precursor and 0.2 g of $NaH_2PO_2·2H_2O$ were placed separately at the upstream and downstream positions of a quartz tube with a mass ratio of 1:10. The furnace was heated to 350 °C at a ramp rate of 3 °C min^{-1} and held for 2 h. After natural cooling, black CoP nanorods were obtained [35].

2.3 Synthesis of the CoP@NWHC composite

The CoP@NWHC composite was prepared through in-situ polymerization of pyrrole on CoP nanorods using NWHC as a carbon scaffold. First, 0.2 g of CoP nanorods was dispersed in 250 mL deionized water containing 0.04 g SDS under ultrasonication for 6 min, followed by vigorous stirring for 2 h. After cooling at 4 °C for 10 min, 0.2 mL of pyrrole monomer and 0.2 g of NWHC were added sequentially. Then, 0.78 g of $FeCl_3·6H_2O$ dissolved in 10 mL deionized water was dropwise added as the oxidizing agent. The mixture was ultrasonicated for 10 min and stirred for another 2 h at room temperature. The resulting precipitate was collected by centrifugation, washed thoroughly with deionized water and ethanol, and dried at 50 °C overnight. Finally, the product was carbonized at 800 °C for 3 h under N_2 flow with a heating rate of 5 °C min^{-1} to obtain the CoP@NWHC composite [36].

2.4 Materials characterization

The crystalline phase of the samples was examined by X-ray diffraction (XRD, Shimadzu D-6000) using Cu Kα radiation (λ = 0.15418 nm) over a 2θ range of 10–80° at a scan rate of 5° min^{-1}. The morphology and microstructure were investigated by field-emission scanning electron microscopy (FE-SEM, JSM-6701F) and transmission electron microscopy (TEM, JEOL CM200 FEG)

III. RESULTS AND DISCUSSION

3.1 Mechanism of the HBFCs formation

The CoP@NWHC composite electrode was synthesized via a facile two-step strategy. First, wheat husk-derived carbon nanomaterials (NWHC) were prepared by $FeCl_3$ and Urea treatment followed by carbonization. Second, NWHC nanomaterials were uniformly grown on both the inner and outer surfaces of CoPnanorod arrays through a subsequent hydrothermal process and phosphorization. This approach ensures strong interfacial adhesion between CoP and the carbon matrix while preserving the 1D hollow structure of NWHC.

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3.2 Morphology and structure characterization

The crystal structure of NWHC, CoP, and CoP@NWHC composites was examined by X-ray diffraction (XRD), as shown in Figure 1. For CoP, the diffraction peaks at 2θ = 31.6°, 36.3°, 48.1°, and 56.3° correspond to the (011), (111), (211), and (301) crystal planes, respectively, which match well with the standard CoP phase (JCPDS No. 29-0497). The XRD pattern of NWHC shows a two broad peaks were detected at 23° and 43°, matching the (002) and (100) planes of graphitic carbon layerc [37]. In the CoP@NWHC composite, all characteristic peaks of CoP are retained without noticeable shift, and no impurity phases are detected. This confirms the successful formation of the CoP@NWHC heterostructure and indicates that the carbon coating does not alter the crystalline phase of CoP. The absence of peak broadening or shifting suggests that NWHC grow epitaxially on CoPnanorods with minimal lattice strain. The carbon matrix acts as a structural buffer, preventing aggregation of CoP nanorods during synthesis and maintaining high crystallinity. The surface area and pore structure of CoP@NWHC were further analyzed using N_2 adsorption–desorption isotherms, shown in Figure 2. The isotherm exhibits a type IV curve with an H3 hysteresis loop according to IUPAC classification, indicating the presence of mesoporesc [38]. CoP@NWHC possesses a specific surface area of 96.8 $m^2\ g^{-1}$ and a pore volume of 0.24 $cm^3\ g^{-1}$. The pore size distribution (PSD) curve, inset in Figure 2, reveals that pores are predominantly distributed in the range of 2.0 nm. This hierarchical mesoporous structure is critical for supercapacitor performance. The high surface area provides abundant active sites for redox reactions, while the mesopores facilitate rapid electrolyte ion diffusion. The hollow tubular structure of NWHC, combined with capillary effects, shortens the ion transport path and enhances rate capability. Figure 3 present the surface morphologies of NWHC, CoP, and CoP@NWHC. CoP, Figure 3a, b, forms aggregated nanorod clusters with poor dispersion. In contrast, Figure 3c, d reveal that CoP nanorods are uniformly and densely anchored on both the external and internal surfaces of NWHC, forming a hierarchical “carbon material-on-nanorod” architecture. The carbon serves as a 1D conductive scaffold that prevents CoP aggregation and maximizes exposure of active sites. The gaps between nanorods create interconnected channels, allowing sufficient contact between the electrolyte and active material for efficient Faradaic reactions. The stable 1D architecture of CoP@NWHC provides multiple pathways for ion and electron transport. The carbon acts as a long-range current collector, while the open structure of nanorod arrays minimizes ion diffusion resistance. This synergy explains the enhanced specific capacitance and cycling stability observed electrochemically.

3.3 Electrochemical Performance

The electrochemical performance of the as-prepared electrodes was evaluated in a three-electrode system using 3 M KOH as the electrolyte. Figure 4a compares the cyclic voltammetry (CV) curves of CoP, NWHC and CoP@NWHC electrodes at a scan rate of 20 mV s^{-1}. All CV curves exhibit a pair of well-defined redox peaks, indicating that the capacitance is dominated by reversible Faradaic redox reactions of Co^{2+}/Co^{3+} in CoP.Notably, the CoP@NWHC electrode shows a significantly larger integrated CV area than CoP, implying a much higher specific capacitance. Moreover, the potential separation between the anodic and cathodic peaks for CoP@NWHC is smaller than that of CoP, suggesting improved electrochemical reversibility and faster reaction kinetics [39]. The CV curves of the CoP@NWHC electrode at different scan rates are presented in Figure 4b. As the scan rate increases from 5 to 50 mV s^{-1}, the redox peak positions shift only slightly and the curve shape is well-maintained. This indicates excellent rate capability, which can be attributed to the long-range conductive NWHC scaffold and its intimate contact with CoP nanorods that ensure rapid electron transport. Galvanostatic charge–discharge (GCD) curves of the CoP@NWHC electrode at various current densities are shown in Figure 4c. The nonlinear GCD profiles reveal typical pseudocapacitive behavior, consistent with the CV results. The nearly symmetric charge–discharge curves also confirm the high reversibility of the redox reactions. As illustrated in Figure 4d, the CoP@NWHC electrode delivers a high specific capacitance of 521 F g^{-1} at a high current density of 0.5A g^{-1}. The optimized CoP@NWHC composite exhibits the highest capacitance, which can be ascribed to the uniform growth of carbon nanostructureson the surface of CoP nanorods. This architecture provides abundant electrode/electrolyte interfaces for redox reactions while the 1D CoP prevents aggregation of carbon matrix. Electrochemical impedance spectroscopy (EIS) was performed to evaluate the charge-transfer kinetics of the electrodes. As shown in Figure 4e, the CoP@NWHC nanocomposite exhibits an Rs of 0.55 Ω and an Rct of 0.06 Ω, substantially lower than those of pristine CoP (Rct = 0.62 Ω). The diminished semicircle in the high-frequency region and the steeper slope in the low-frequency region indicate reduced charge-transfer resistance and faster ion diffusion at the electrode/electrolyte interface. This enhancement is attributed to the NWHC nanosheets grown on CoP nanorods, which establish a conductive network that accelerates electron/ion migration and improves the overall conductivity of the composite. The cycle performance of the CoP@NWHC electrode with 90.4% capacitance retention after 8000 cycles, the fabricated three device demonstrates exceptional cycling stability, as seen in Figure 4f. It’s also important to note that, because CoP@NWHC is less expensive and simpler to prepare, using it as the negative electrode in our device is

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preferable to using a commercial anode material. The improved cycling stability of CoP@NWHC can be assigned to the mechanical buffering effect of the NWHC matrix, which accommodates volume changes of CoP during repeated charge–discharge processes. The superior device performance originates from three synergistic factors: 1) The 1D conductive NWHC network provides fast electron pathways and structural stability, 2) The uniform CoP nanorods offer abundant redox active sites, and 3) The hierarchical porous structure facilitates rapid ion diffusion and electrolyte penetration. These results demonstrate that CoP@NWHC//NWHC is a promising candidate for high-performance energy storage devices.

IV. CONCLUSION

In summary, carbon-coated CoP nanostructures (CoP@NWHC) were successfully fabricated via treatment and hydrothermal growth. The conformal carbon coating on both interior and exterior surfaces of CoP created a hierarchical architecture that enhanced structural stability and provided continuous pathways for electron and ion transport. As a result, the CoP@NWHC electrode exhibited a high specific capacitance of 521 F g^{-1} at 0.5 A g^{-1} and retained 90.4% capacitance after 8000 cycles. Demonstrating the great potential of biomass-derived CoP@carbon composites for practical energy storage applications.

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DECLARATIONS: Ethics, Consent to Participate, and Consent to publish declarations (**Not applicable**).

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APPENDIX

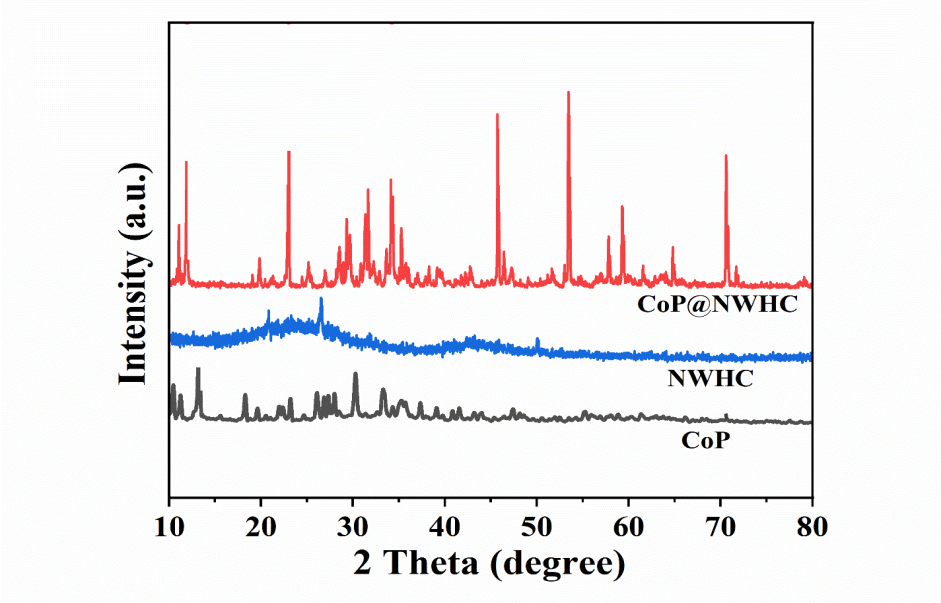

Figure 1: XRD patterns of CoP, NWHC and CoP@NWHC nanomaterials

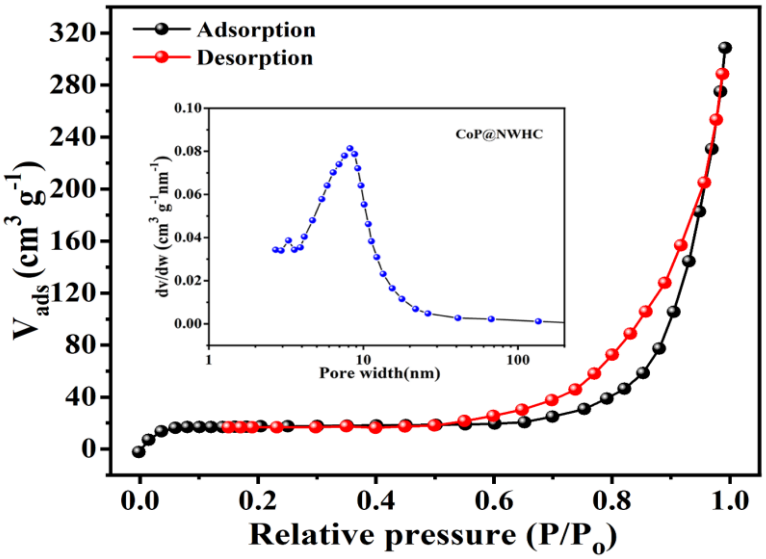

Figure 2: BET N_2 adsorption-desorption isotherms of CoP@NWHC composite and their corresponding BJH pore size distribution curves (the inset)

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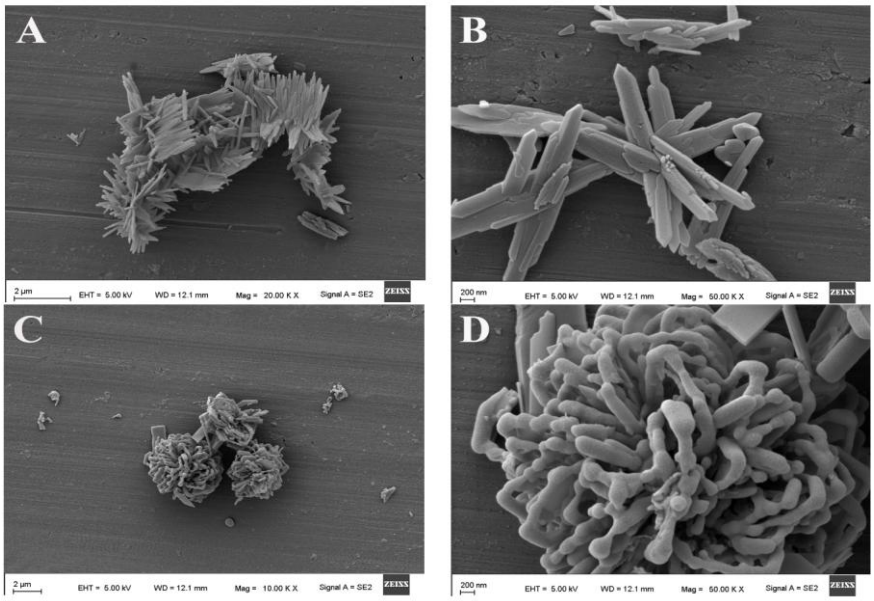

Figure 3: (A, B) SEM images of CoP nanorods; (C, D) SEM images of CoP@NWHC nanocomposites at different magnifications

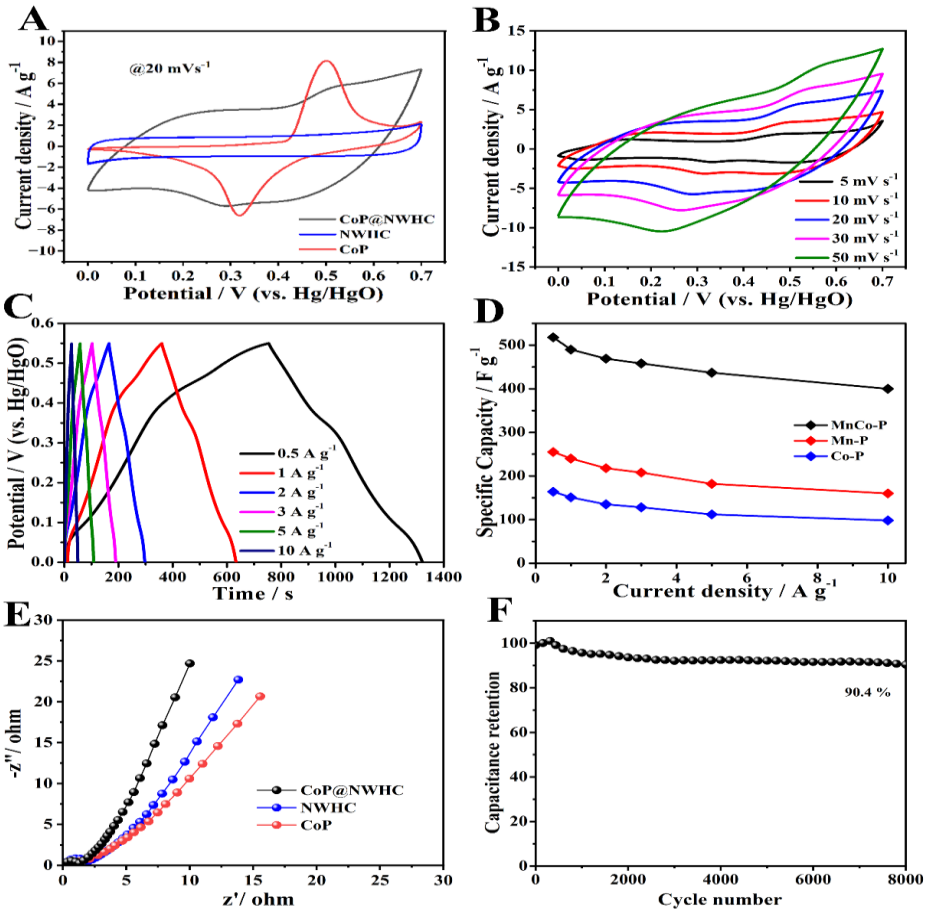

Figure 4: (A) CV curves of the CoP, NWHC and CoP@NWHC at 20 mV s^{-1}; (B) CV curves of CoP@NWHC; (C) GCD curves of CoP@NWHC at different current densities; and (D) Specific capacities of the CoP, NWHC and CoP@NWHC electrodes at different current densities; (E) The Nyquist plots of the CoP, NWHC and CoP@NWHC electrodes; (F) The cycling stability of the CoP@NWHC electrodeat 2 A g^{-1}in 0.5 M KOH electrolyte

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